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Controlled Atmosphere and Fumigation in Grain Storages

B.E. Ripp et al. (editors)

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Developments in Agricultural Engineering 5

Controlled Atmosphere and Fumigation in Grain Storages

Proceedings of an International Symposium "Practical Aspects of Controlled Atmosphere and Fumigation in Grain Storages" held from 11 to 22 April 1983 in Perth, Western Australia

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PREFACE

The second International Symposium on Controlled Atmosphere Grain Storage held in Perth, Western Australia, was organised by Co-operative Bulk Handling Limited and the Australian Grain Institute Incorporated in collaboration with the Commonwealth Scientific and Industrial Research Organisation and Food and Agriculture Organisation of the United Nations. This followed the first Symposium held in Rome in May 1980 organised by Assoreni. During discussion in Rome, several participants expressed the view that the next meeting should cover more problem loaded aspects of controlled atmosphere storage and disinfestation of grains, gas tight facilities, sealing practices, inert gas production and high moisture grain storage. Dr Shejbal in his preface to the proceedings manual recalled these views and expressed the hope that his Symposium would be followed elsewhere in the world to permit a further diffusion of this environment - friendly, economical and safe preservation technique in modern storage facilities.

This was the call that Co-operative Bulk Handling Limited responded to when this Symposium was organised and leads to its title - "Practical Aspects of Controlled Atmosphere and Fumigation in Grain Storages". As our Chairman Mr H W Gayfer stated during his Keynote address, the reasons we believed we were in the most favourable position to conduct this Symposium were:-

1. Our year round climate is ideal for the development of grain insects.

2. As 95% of our produced grain is exported our control standards must be of high order.

3. C.S.I.R.O. officers have developed their research into C.A. Storage to the commercial application stage.

4. A National Committee of Bulk Handling Authorities and C.S.I.R.O. had combined their resources throughout Australia for the development of sealing technqiues.

5. C.B.H. Ltd had realised that unless the method could be applied to existing storages as well as new storages its potential was seriously diminished.

Considerable funds had been allocated by C.B.H. Ltd for the application of these techniques, sufficient to provide 1.5 million tonnes of sealed storages at the completion of the then current programme.

As this was to be a practical symposium a field trip to visit sites in various stages of the sealing exercise was a necessary part of the demonstration process. This was organised as part of the Controlled Atmosphere and Fumigation Week. Further, to provide coverage of the broad aspects of grain handling, storage, transport and quality control as an added attraction to visitors undertaking the long journey to Perth in a depressed economic climate, a second week was made available.

270 participants from 28 countries attended the Symposium and I have yet to see a more convivial, a more totally committed group of people interchanging freely, giving and receiving knowledge and experience.

The major gap yet to be covered in Controlled Atmosphere Grain Storage is its compatability with grain of very high moisture content. Such aspects could not be demonstrated in Australia by virtue of our climate and demonstration of theories <u>is</u> necessary. This to some extent limits the ideal geographic zone for the holding of the next C.A.F. Symposium. To this end our International Steering Committee have chosen to remain as a body for the purpose of aiding the promotion of the system and guiding the selection of the next venue.

My appreciation is extended to all those organising committees within C.B.H. Ltd, to our field staff whose efforts will be well known by all participants, to the Chairmen of sessions who actually controlled the running of the Symposium, and to the Rapporteurs. There would have been no Symposium on the practical aspects of the transfer of controlled atmosphere and fumigation technology into commercial use, especially on the scale we have seen, had the facilities not been made available and the costs underwritten. "The influence of this event will be felt in grain storage activities throughout the world for many years to come" is a statement made by a speaker at a Stored Grain Protectant Conference. Therefore I find it difficult to find words sufficient to express the degree of attribute deserved to our General Manager Mr Jim Green and our Board of Directors for their full support both during the Symposium and the years leading up to it. A special recognition is also well deserved for the efforts of Alan Grey and his willing assistance in the organising and the conduct of the Symposium and in the compilation of this manual of proceedings.

The International Steering Committee, who also formed the body of Editors, who assisted from all points of the Compass before, during and after the Symposium, were Ed Jay U.S.D.A., Ed Bond Canada Agriculture, David Calverley T.D.R.I., Geoff Corbett F.A.O., S. Navarro A.R.O., Jonathon Banks C.S.I.R.O.

A particular thank you to Jonathon Banks for his dedication to his work and for his support and guidance over a number of years. It was most unfortunate that JINDRICH SHEJBAL was unable to attend this follow up to his orginal work. I know he would have liked to have been with us.

I have maintained the order of events in this manual of proceedings as they occurred during the period from 11th to the 22nd April 1983.

Perth, Western Australia, April 1984. B.E. RIPP

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CURRENT USE OF CONTROLLED ATMOSPHERE STORAGE

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Edward Jay and Robert D'Orazio	-	United States of America
Lu Qian Yu	-	Peoples Republic of China
H. Akiyama	-	Japan

PROGRESS IN THE USE OF CONTROLLED ATMOSPHERES IN ACTUAL FIELD SITUATIONS IN THE UNITED STATES

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ABSTRACT

Field research on the use of carbon dioxide (CO_2) for stored product insect control in the United States in the last 3 years is described. Minor efforts were made in sealing the storage structures studied and in 7 tests in upright concrete silos or welded steel bins containing wheat, maize, rice or sorghum the amount of CO_2 used/1,000 metric tonnes (t) grain ranged from 3.1 to 4.5 t. However, treatment costs/t of grain ranged from 0.23 to 0.39 U.S. $ because of the availability and low costs associated with CO_2 marketing in this country.

Additional studies on the use of CO_2 in farm storage situations and in rail hopper cars containing flour are also described. The future of the use of CA in the U.S. is discussed.

INTRODUCTION

Since the last meeting in Rome (Shejbal, 1980) on the subject of controlled atmospheres (CA) there has been an increased interest on the use of this residue-free insect-control technique for pests of grain and oilseeds in storage in the United States (U.S.). This interest cannot be described as an all-out conversion from the use of conventional fumigants to the use of CA. Rather, it can be considered as an inquiry by large grain and oilseed handlers into the effectiveness of the technique with particular emphasis being placed on the economics of the treatment when compared to the costs of using conventional fumigants. This interest is possibly also motivated by the realization that the U.S. Environmental Protection Agency (EPA) may prohibit the use of liquid fumigants containing carbon tetrachloride and methyl bromide for grain treatment in the future in the U.S. This would leave the grain industry with only hydrogen phosphide (PH_3) produced by aluminium or magnesium phosphide formulations for the fumigation of grain or oilseeds in post-harvest situations and with CA for treatment of these commodities.

Laboratory and field research conducted by the U.S. Department of Agriculture has shown that carbon dioxide (CO_2) is the CA of choice for use in situations where little or no attempts are made to seal the storage structure prior to treatment. CO_2 is effective in controlling storage pests at concentrations of 35% or more (Jay, 1971, 1980; Jay and Pearman, 1973; Jay et al. 1970). Although CO_2 is more effective at concentrations of ca. 60% for most storage pests,it can be allowed to fluctuate in the range of from 35 to 100% and insect control can be achieved. The exposure time needed to obtain high levels of insect control is a function of CO_2 concentration, temperature, grain moisture content, and the species and life stages of the insects which are infesting the grain or oilseeds (Bailey and Banks, 1980; Jay, 1984a,b). Current recommendations state that a concentration of from 45 to 60% CO_2 should be attained and maintained for 5 to 6 days at temperatures above 27°C; for 10 to 14 days at a temperature at or above 21°C and for 21 to 28 days at temperatures at or above 16°C (Jay, 1984a). However, reluctance is encountered when these exposure times are suggested for use in cooperative studies with large grain processors since there is a general feeling that these exposure times are too long when the CO_2 treatment is compared with a PH_3 treatment. Therefore, most field studies conducted in this country have been for a 4-day period after a CO_2 concentration of ca 60% is attained in a storage structure. These treatments generally result in ca 95% control of natural or artificial insect infestations in the commodity being treated.

The U.S. EPA in 1980 granted an exemption from tolerance (approval for use) for the use of CO_2, nitrogen and combustion product gas (effluent from a CA generator) for all raw agricultural products (U.S. Federal Register 45, pp. 75663-64, Nov., 1980) and in 1981 for the use of these treatments on processed agricultural products (U.S. Federal Register 46, pp. 32865-66, June, 1981). This approval has stimulated some companies which produce CO_2 to attempt to introduce their product for use in stored-product insect control to the grain and oilseed industry. This paper describes some of the commercial scale tests that have been conducted in the last 3 years.

METHODS AND MATERIALS

Treatment of cylindrical grain storage bins

Silo and construction material, bin dimensions, commodity and amount of grain and its temperature are shown in Table 1. Trials 1 to 7 were conducted in terminal elevators or inland terminals located in Texas and trials 8 and 9 were conducted in Harvestore(R) bins located on a South Carolina farm. Harvestore bins are fiberglass lined steel bins generally used for the storage of silage.

CO_2 was supplied from pressurized tanks of 3.6 t, 5.4 t, or 10.9 t capacity and were equipped with suitable vaporization equipment. The CO_2 was introduced from the bottom of the bins in trials no. 2, 3, and 8 and from the top in the other trials (Table 1).

Table 1 - Bin type and dimensions and amount and temperature of the commodity in tests on the application of CO_2 to control insects.

Trial No.	Bin Type	Bin Dimensions (m) Height	: Diameter	Commodity	Amount Of Grain (t)	Temperature Range (C°)
1	Concrete	33.0	7.6	Wheat	1088	31 - 34
2	Welded Steel	12.8*	28.7	Wheat	5442	32 - 36
3	Welded Steel	12.8*	28.7	Wheat	5388	**
4	Concrete	37.0	7.4	Sorghum	1224	27 - 32
5	Concrete	37.0	7.4	Rice	1033	21 - 27
6	Concrete	**	**	Sorghum	726	**
7	Concrete	**	**	Maize	812	**
8	Harvestore			Wheat	381	30 - 35
9	Harvestore			Wheat	163	30 - 35

* Height of the wall to top cone.
** Data not available

Samples of grain were taken in trials no. 1, 8 and 9 to determine the percent reduction in emergence (% RIE) of adults which was caused by the treatments. In addition caged laboratory immature mixed-age cultures of Rhyzopertha dominica (F.) and Sitophilus oryzae (L.) were used for bioassay in trials no. 2, 4, 5, 6 and 7. The amount of CO_2 used in the purge and maintenance phases of the treatments was recorded from flow meters or from gauges installed in the supply tank. CO_2 concentrations were observed in gas samples taken from several locations in the silos.

Treatment of rail hopper-cars containing flour with dry ice

Four hopper-type rail cars equipped for the exclusive delivery of 77 t each of flour from the mill to bakeries located in the U.S. were modified for the tests. They were equipped with gas sampling lines and caged Tribolium confusum J. du Val. at depths of 0.6, 1.8 and 3.4 m (Ronai and Jay, 1982). The hopper cars were then filled with ca 77.1 t of flour. After filling, either 91 or 181 kg of dry ice pellets (small extruded pieces of solid CO_2) and 91 kg of dry ice blocks contained in cloth bags were pushed down into the bulk of flour in each car. The cars were shipped from the mill, located in Ohio, to a bakery located in Georgia. The length of the treatment (time from loading and applying the dry ice to unloading) was 10 to 11 days and the flour temperature was 33°C at loading and 24° to 28°C at the time they were unloaded.

RESULTS

CO_2 Application in Grain Storage Structures

Table 2 shows that the amount of CO_2 used during the purge phase in these tests varied from 32 to 158 kg. CO_2/h/1,000 t grain and during the maintenance phase from 13 to 52 kg. CO_2/h/1,000 t grain. This variation in the application rate during the purge phase was due to the duration of the CO_2 application, the CO_2 vaporizer delivery capacity and the size of the hose used to convey the CO_2 gas into the bin. The variation in the application rate during the maintenance phase was dependent on the gastightness of the experimental bins and the attempt to maintain the predetermined CO_2 concentration. The welded steel bins used in Trials 2 and 3 were more gastight than the concrete bins and consequently required lower maintenance rates. Trials conducted on Harvestores (Trials 8 and 9, Table 1) resulted in excessive usage of CO_2 (12.2 t CO_2/1,000 t of grain) due to equipment failure. Therefore, these results were not incorporated in Table 2.

CO_2 application time during the purge phase was dependent on the application rate chosen to attain the predetermined CO_2 concentration and was based on leak rates and the capacity of the vapourization equipment. However, the application time during the maintenance phase was kept in the range of 72 - 90 h (Table 2) to maintain an effective lethal CO_2 concentration for insect control.

Table 2 - CO_2 usage rate, application time, concentrations attained, and estimated cost of the CO_2 used to control insects (Trial numbers as identified in Table 1).

Trial No.	CO_2 application (kg CO_2/h/1000 t) Purge	CO_2 application (kg CO_2/h/1000 t) Maintenance	CO_2 application time (h) Purge	CO_2 application time (h) Maintenance	Concentration (% CO_2) attained and maintained	Delivery Cost \$US/t CO_2	Efficiency t CO_2/1000 t of grain	Cost \$US/t of grain
1	84-125	32-52	6	90	60±10	73	3.2	0.23
2	32	14	88	80	80±10	73	4.0	0.29
3	33	13	72	72	80±10	73	3.3	0.24
4	56-102	34-39	17	79	60±10	77	4.2	0.32
5	158	38	8	88	60±10	73	4.5	0.33
6	58	25	18	78	80±10	88	4.4	0.39
7	*	*	*	*	60±10	88	3.1	0.27
						Mean	3.8	0.30
						SE ±	0.23	0.021

* Data not available

The CO_2 concentrations maintained during the maintenance phase varied from 50 to 90%.

Costs of Treatment

Cost of CO_2 at delivery for application on site, varied from $US 73 to 88/t CO_2 in trial nos. 1-7 (Table 2). However, for trials 8 and 9 (Table 1) the required CO_2 was supplied at a delivery cost of $ US 221/t. This high delivery cost was due to the low projected yearly use rate on the farm.

The efficiency of CO_2 usage expressed as t CO_2/1,000 t of grain for the first 7 trials are shown in Table 2. Accordingly the mean CO_2 usage was 3.8 t CO_2/1,000 t of grain with a standard error representing the variation under the described trial conditions as $\pm$ 0.23. Based on the CO_2 cost at delivery and the amount of CO_2 used, the mean treatment cost was $ US 0.30/t of grain.

Efficacy of CO_2 Treatment to Control Insects

Results obtained from treatment of wheat in an upright concrete silo (Trial no. 1) are shown in Table 3. This table shows that after a comparison of the pre- and post-treatment samples there was a 99% RIE of the natural population after the samples were incubated at 26°C for 60 days. Carbon dioxide concentrations in the basement under the silo did not exceed 0.5% (TLV value for an 8-h day) at any time during the test. During outloading, which was conducted 24 h after completion of the treatment, CO_2 concentration reached 0.8% for 21 h and then dropped below 0.5% for the remainder of the outloading period.

Table 3 - Mean number of alive insects from naturally infested wheat samples (1 kg) and percent reduction in emergence (% RIE) of adults recorded on Trial no. 1.

Days After Treatment	Mean number of alive insects per sample		% RIE
	Pretreatment	Posttreatment	
3	22	0	100
15	63	20	68
30	215	10	95
60	1,227	6	99

Treatment of wheat in a welded steel bin (Trial no. 2) resulted in 98.2 % RIE of adults from the immature stages of S. oryzae and 100% of R. dominica. These insects were not exposed to high concentrations of CO_2 except during the last 80 h of the treatment since the CO_2 which was applied from the bottom did not reach the top of the bin in high concentrations until 88 h after the initiation of the purge.

Treatment of sorghum in an upright concrete silo was carried out in Trial no. 4. The bioassay conducted on the mixed aged immature S. oryzae and R. dominica resulted in 99% RIE and 97% RIE of adults, respectively at the end of the treatment. However, since these insects were in the headspace of the silo and were exposed to high CO_2 concentrations during the purge and subsequent maintenance phases of the test, these results are considered inconclusive.

Treatment of rough rice in an upright concrete silo (Trial no. 5) showed that mortality of the insects exposed to this treatment was 100% while tests with sorghum and maize (Trials no. 6 and 7) indicated that the % RIE of caged, mixed age immature S. oryzae after treatment was 98.5.

Despite the high treatment costs of farm stored wheat (Trials no. 8 and 9, Table 1) the results of the treatments were effective. Table 4 shows that a 95% RIE was obtained in samples taken from the top and a >99% RIE was obtained in samples (1 kg) taken from the bottom at the 60 days post-exposure observation of the grain taken from the 381-t bin (Trial no. 8).

Table 4 - Treatment of 381 t farm stored wheat (Trial no. 8, Table 1). Number of alive insects from naturally infested wheat samples and % RIE of adults resulting from the CO_2 treatment.

Days After Treatment	Mean No. Insects/Sample		% RIE
	Pretreatment	Posttreatment	
	Bottom Samples		
4	10	1	93
30	28	8	73
60	402	19	95
	Top Samples		
4	2	0	100
30	7	0.3	96
60	29	0.1	> 99

Table 5 shows that similar results were obtained in the smaller bin containing 163 t of wheat (Trial no. 9). In this bin the % RIE of adults was >99 at both the 30 and 60 day post-treatment examinations.

Table 5 - Treatment of 163 t farm stored wheat (Trial no. 9, Table 1). Number of alive insects from naturally infested wheat samples and % RIE of adults resulting from the CO_2 treatment.

Days After Treatment	Mean No. Insects/Sample		% RIE
	Pretreatment	Posttreatment	
4	48	22	54
30	150	1	>99
60	1,111	2	>99

Treatment of rail hopper-cars containing flour with dry ice

Table 6 presents observed CO_2 concentrations in one of the hopper cars before it left the mill (18 to 41-h readings) and observed CO_2 concentrations 10 days after the car was filled and shipped to the bakery. The dry ice changed to gas slowly in the first 41 h of treatment but an even distribution of from 31 to 40% was observed 10-days after filling. Readings for CO_2 concentration or distribution were not taken while the cars were in transit.

Table 6 - Carbon dioxide concentrations in hopper car S90143 containing 77 t flour and treated with 181 kg dry ice.*

Sample Depth (m)	% CO_2 at indicated time after treatment**			
	18 h	25 h	41 h	10 days
0.6	30	43	45	31
1.8	8	18	23	38
3.4	2	6	17	40

* From Ronai and Jay, 1982.

** Hopper car was in-transit between 41-h after application and arrival at destination 10-days later.

However, mortality of *T. confusum* larvae during the exposure period ranged from 95.2 to 99.1% indicating that the treatment was effective (Table 7).

Table 7 - Mortality of *T. confusum* larvae in flour contained hopper cars during a 10 or 11 day in-transit exposure.*

Hopper Car No.	CO_2 used (kg)	% Mortality
1	181	99.1
2	181	99.1
3	181	95.2
4	272	98.3

* From Ronai and Jay (1982)

This table also shows that mortality was not increased in hopper car number 4 when an additional 91 kg of dry ice pellets were added when compared with 2 of the other 3 cars which did not have the additional pellets and was only slightly higher than that observed in car no. 3.

Costs for dry ice for the three cars treated at the rate of 181 kg/car was $24 per car while the cost for a PH_3 treatment was estimated to be $17. However, labor costs involved in the 1.5 h aeration of a car treated with PH_3 prior to unloading was estimated to be $25 so the net savings per car when the dry ice was used was estimated to be $18. Also, cars treated with CO_2 could be unloaded immediately while cars treated with PH_3 would have to stand at the unloading point during aeration. Thus, the use of CO_2 would result in a much faster turnaround of the cars.

DISCUSSION

Carbon dioxide use per ton of grain in these studies was very high when compared to the amount used in well-sealed Australian storages (Banks, et al., 1980). The gastightness of the experimental bins was not determined, but they were suspected to be low. Despite the low level of gastightness of these storage structures, the CO_2 application costs/t of grain were low (Table 2). This is obviously because of the low delivery cost of CO_2 in most situations in the U.S. This low cost, which in many cases results in treatment costs for CO_2 being competitive with costs for the use of conventional fumigants, may cause large grain and oilseed companies to be reluctant to attempt any extensive sealing of their storage structures prior to the use of this treatment. However, studies on the economics of sealing the discharge areas of upright concrete silos, where the majority of the CO_2 is lost during treatment, are to be initiated so that gas loss can be partially reduced.

The fact that large grain companies in this country are interested in this technique is encouraging. The rice industry in particular, is adopting this technique and, by the end of 1983, five large rice processors located in the states of Texas, Arkansas and Louisiana are planning to have CO_2 vessels located on their premises for routinely treating rough and processed rice with CO_2. There is also considerable interest in the use of CO_2 for control of insects attacking stored tree nuts and groundnuts (peanuts) indicating that processors of agricultural commodities having a high value per ton, as compared to cereals, may be the first to use this technique on a large scale in the U.S.

The use of CO_2 in on-farm storage situations presents additional problems as far as treatment costs are concerned. Although ca. 60% of the wheat and feed grains are stored on-farm in the U.S., the logistics of transporting liquid CO_2 to these areas appear to be costly ($221/t CO_2) for delivery in the one on-farm test reported above. Obviously, the use of gaseous CO_2 in on-farm situations will involve either sealing to very rigorous gas-tight specifications or development of some other form of CA treatment before the use of this technique can be competitive with conventional fumigants.

The use of CO_2 and other CA treatments in the U.S. need not be restricted to stored grain. The effectiveness of CO_2 when used in rail hopper cars containing flour for insect control can be expanded to other processed agricultural products in both static and in-transit situations. Also, the technique could be expanded in the U.S. for use with many agricultural products in in-transit situations such as truck-ship type containers, barges and ocean going ships. More research in these areas is needed to provide the grain industry with techniques and information to determine situations where CA is advantageous over alternative insect control methods.

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AN OVERVIEW OF THE PRESENT STATE OF CONTROLLED ATMOSPHERE STORAGE OF GRAIN IN CHINA

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Abstract: Controlled atmosphere (CA) technology is widely used in China in various forms for the preservation of stored grain. The airtight enclosures required for the process are made in several ways: by covering the grain surface with polyethylene sheet which is then fixed to the walls previously treated with asphalt, by completely enclosing the grain bulk in PVC sheet or, for small quantities, by sealing the grain in polyethylene/polyester laminated film (skinpacking). Carbon dioxide and simple hermetic storage processes are commonly used to provide the controlled atmosphere. A mobile unit for producing suitable atmospheres has been developed. This unit, containing cultures of microorganisms, is connected to the sealed system and reduces the oxygen content through the respiration of the microorganisms. Nitrogen production using exothermic inert-atmosphere generators and molecular sieve air separation systems are under investigation.

Rates of oxygen reduction under hermetic conditions and the influence of controlled atmospheres on grain quality and mould growth have been investigated. CO_2 at 80% inhabits the growth of moulds and yeasts and can preserve quality of stored rice for six months through the summer. It is preferable to combine CA treatment with reduction of temperature below 20°C to obtain the best storage conditions.

1. INTRODUCTION

Controlled atmosphere (CA) techniques have been adopted as the main methods for grain storage in large stores in China. CA systems are particularly acceptable as they either minimise the quantity of fumigant used or eliminate its use completely.

Two main problems must be overcome during the development of CA technology: how to create airtight enclosures and how to create suitable atmospheres. It is also necessary to determine the effect of CA storage on the quality of the various commodities and the optimum concentration of the various gases involved, both against pests and to preserve quality and for safety reasons. These problems have been studied in various regions in China. Some of the results of these studies are described in this paper.

Generally, it has been found that CA technology is convenient and cheap and that it may be applied effectively to the storage of many kinds of grain. It may also be used during transportation of grain, its sale to the consumer and even for the storage of medicinal materials and various native products. It can also be used as an emergency storage for grain of excessive moisture content which cannot be dried at once.

2. DEVELOPMENT OF CA TECHNOLOGY

2.1 Sealing of grain storages for CA use

2.1.1 *Sealing of the grain surface with polyethylene sheet ('one-side sealing').* This method is suitable for the sealing of stores in which the grain is loaded in bulk. The walls and floor of the storage are rendered airtight by painting with asphalt. Polyethylene film is spread over the surface of the grain pile and sealed with wax into a trench in the top of the wall of the store (Fig. 1). The method has been widely used for the storage of bulk grain and is always used in large stores.

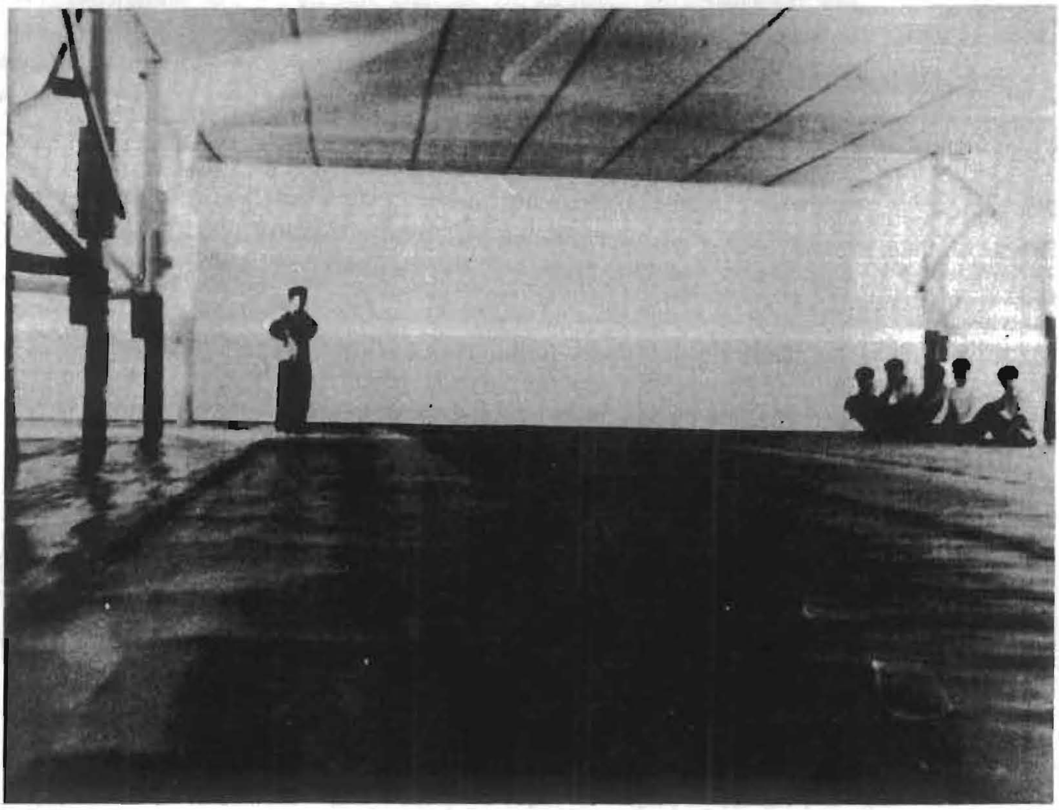

Fig. 1. Grain covered with PVC sheeting in a large store ('one-side sealing')

2.1.2 *Complete sealing in PVC ('six-side sealing').* In this method the grain bulk is completely enclosed in PVC membrane. The enclosure is made in the following steps:

(i) The cover is made of 0.23mm thickness PVC film. The cover should be 40 cm larger than the stack.

(ii) Any leaks in the cover are patched or mended using an adhesive made of dichloroethane and perchloroethane (4:1).

(iii) Straw bags are placed as dunnage at the bottom of the stack (above the PVC groundsheet) and either straw or jute bags are laid on the surface of the stack.

(iv) The enclosure is made around the stack using a portable high frequency welding machine to seal the cover and groundsheet together.

The method is primarily used for storing various types of grain in cities (Fig. 2).

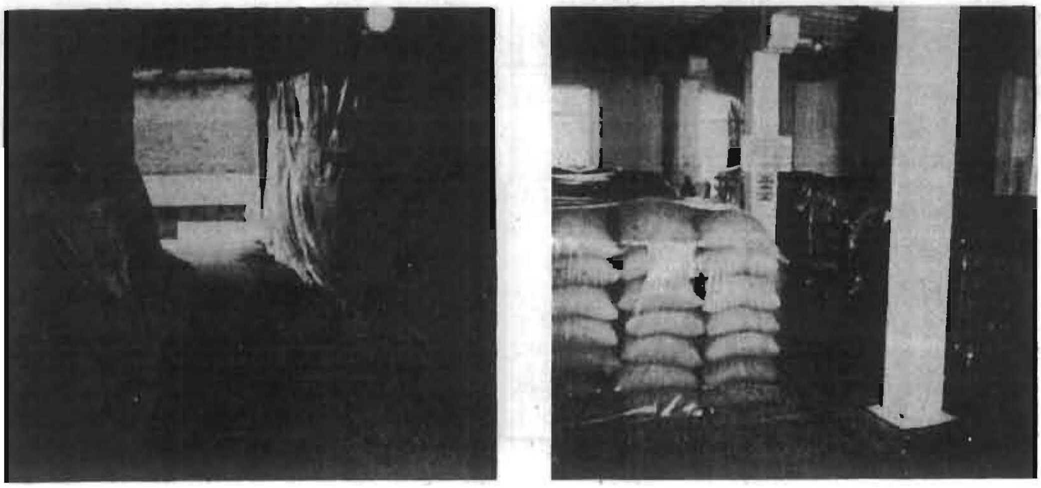

Fig. 2. Enclosing bag stacks of grain in PVC film ('six-side sealing').

2.1.3 *Skin-packing.* Packs of 2-20kg capacity are made for rice and other grains with polyethylene or polyethylene/polyester film using either natural reduction of oxygen, vacuum or the carbon dioxide exchange method (Fig. 3). When CO_2 is used with rice the pack becomes rigid in 3 hours. The method is now used for storing various products (e.g. peanuts, "green grain", red beans, sesame seed) in cities for direct supply to consumers. The stored materials can be kept in this way for a year.

2.2 Methods of generating and maintaining controlled atmospheres

2.2.1 *Natural reduction of oxygen in an airtight storage.* The grain grain bulk is enclosed in a plastic membrane. The grain respiration in the enclosure creates an oxygen-free environment, thus preserving the grain. The rate of reduction of oxygen is much slower for aged grain than for fresh grain. The moisture content of paddy or polished rice stored in this way should be 14.6 - 16%.

2.2.2 *Using CO_2 or N_2 from gas cylinders.* CO_2 or N_2 are supplied in steel gas cylinders. The gases are introduced at the base of the grain bulk, displacing the air in the bulk. The gas concentrations are maintained at 40-85% for CO_2 and 98-99% for N_2. This system provides excellent control of the activities of insects and preserves the grain in good condition for much longer than with storage in normal air atmospheres.

2.2.3 *Methods of nitrogen generation for CA use.*

(i) Combustion of hydrocarbons. Gas may be generated from combustion of hydrocarbons giving a 98.5% N_2, 1.5% O_2 mixture. Generators of this type are now used for CA storage.

(ii) Molecular sieve absorption. The system is based on the seperation of oxygen and nitrogen from air using multiple layers of Type 4A or 5A molecular sieve and calecite *(sic)* or natrolite. Seperation is achieved utilising the difference in diffusion rate of oxygen and nitrogen within the extremely small pores of the materials. The system (Fig. 4) gives 95-98% N_2 and can give a low oxygen atmosphere in 4-5 hours, with recirculation of the atmosphere through the machine.

2.2.4 *Utilisation of microorganisms to remove oxygen.* A 'culture box', containing microorganisms and culture medium, is connected to the grain bulk in an airtight enclosure (Fig. 5). The respiration of the microorganisms reduces the oxygen content of the bulk to a level which controls the growth of pest insects.

For use with large bulk storage bins, the culture box is installed in a mobile apparatus (Fig. 6).

3. RESEARCH STUDIES ON CA STORAGE OF GRAIN

3.1 Rate of natural reduction of oxygen.

In a grain bulk kept under airtight conditions, the oxygen content gradually decreases while the CO_2 content rises to a certain limit. The rate of change and the range of variation of the CO_2 and O_2 concentration is determined by grain type and the temperature

Fig. 3. Skin-packing of (a) rice under hermetic storage, (b) wheat flour in vacuum and (c) rice under CO_2.

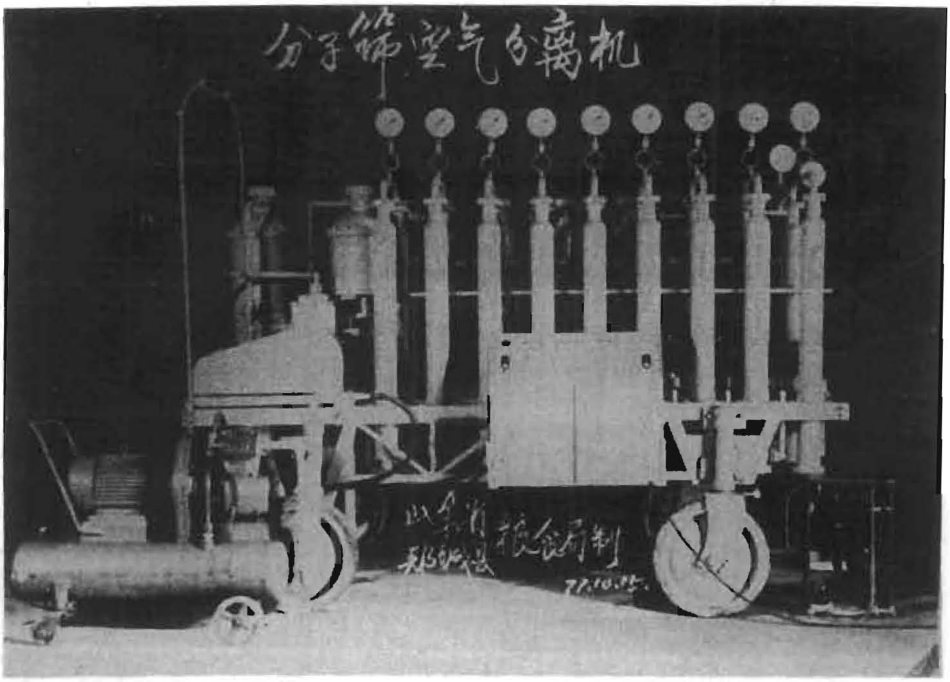

Fig. 4. A molecular sieve machine for separation of oxygen and nitrogen from air.

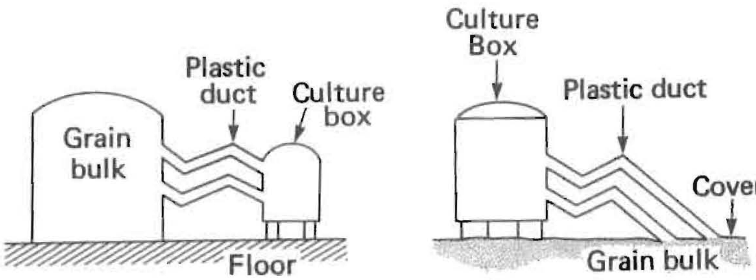

Fig. 5. Connection of a 'culture box', containing microorganisms, to grain bulks in airtight enclosures for a stack (left) or a plastic-covered grain bulk (right).

and moisture content of the bulk. The rate of reduction of oxygen content is greater at greater moisture contents (Figs. 7 and 8).

3.2 Studies on the growth of microorganisms.

CO_2 can control the growth of moulds. The growth of both anaerobic and aerobic microorganisms is substantially inhibited if the CO_2 content exceeds 80% (Table 1).

Under 80% CO_2 rice is in a dormant state and all species of insect are killed. Thus, in summer, grain bulks may be kept without moulding and heating and free of insects. If the grain moisture

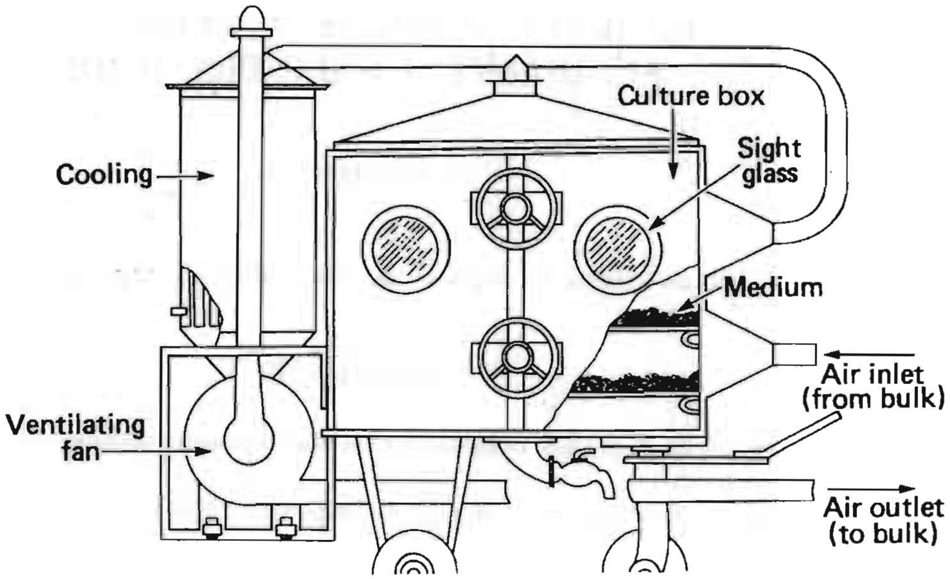

Fig. 6. A mobile apparatus, incorporating a 'culture box', for CA storage of grain in large bulk bins.

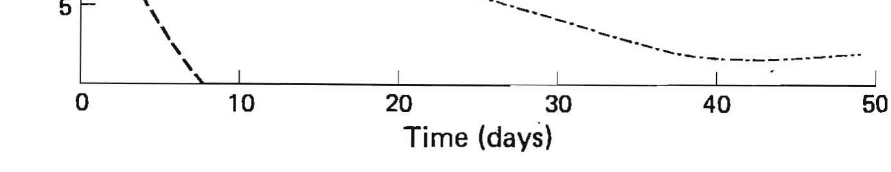

Fig. 7. Variation in the rate of change of oxygen content with moisture content for non-glutinous long grain rice under hermetic storage conditions.

content is >16% or there is condensation at the grain surface, moulding may occur under natural reduction of oxygen (hermetic storage). This may cause the plastic film enclosures or bags to swell up. The moulds found under these conditions are always either *Aspergillus* spp. or *Rizopus* spp. Thus, to ensure that rice may be stored safely in summer, it is recommended that the moisture content be kept below 16%. If the moisture content exceeded 16%,

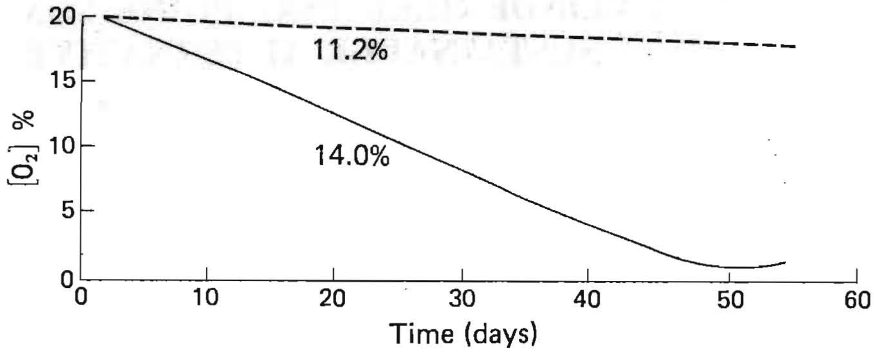

Fig. 8. Variation in rate of change of oxygen content with moisture content for wheat under hermetic storage conditions.

TABLE 1.

Areas of various colonies (cm^2) grown from a single spore under various CO_2 levels.

Species	% CO_2 90	80	60	40	20	0
Aspergillus candidus	0	0	0	1.0	1.2	3.2
A. flavus	0	0	0	2.6	2.4	7.8
A. niger	0	0	1.4	/	1.0	5.4
A. versicolor	0	0	0	0.16	0.16	1.44
Penicillium sp.	0	0	0	0	0.16	7.7
Saccharomyces sp.	0.49	2.6	10.2	5.3	9.0	32.4

dew may form at the surface of the bag stack and moulding may occur when the stacks are stored under the natural hermetic storage system. The plastic covers may balloon out (Fig. 9). Interestingly, it has been found that the mould occurrence varies with season under hermetic storage conditions. The quantity of mould rises as the grain temperature increases and falls again when the grain temperature falls (Fig. 10).

3.3 Studies on grain quality.

The quality of stored grain can be preserved fairly well for six months if the CO_2 content in the store, initially at 80%, can be maintained above 70%. This is particularly so if the temperature is kept below 20°C as well. After storage over a summer season, the cooking quality of rice is as good as fresh grain (Table 2).

Under hermetic storage conditions in summer, despite the high

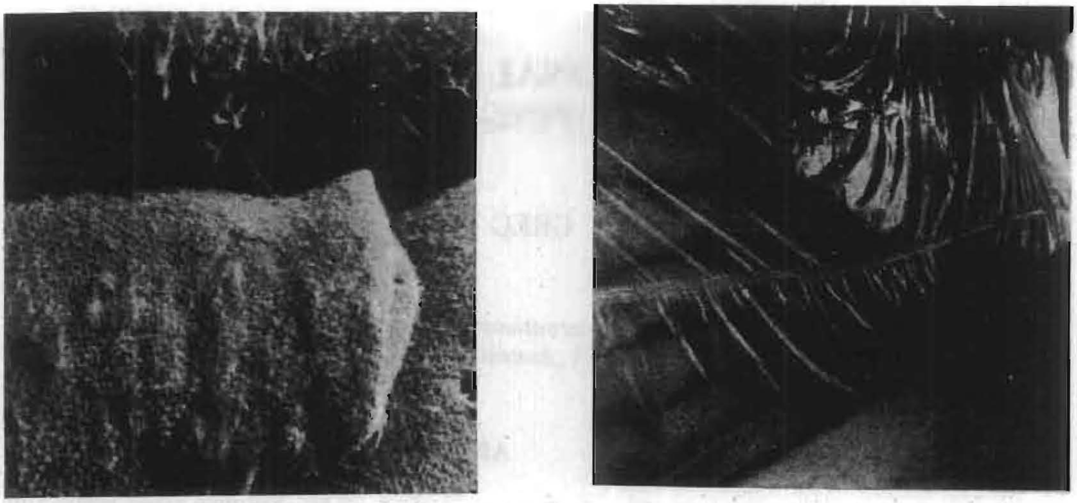

Fig. 9. Mould growing on the surface of bags of rice stored at > 16% m.c. (left). Ballooning of the stack cover with rice stored at > 16% m.c. (right).

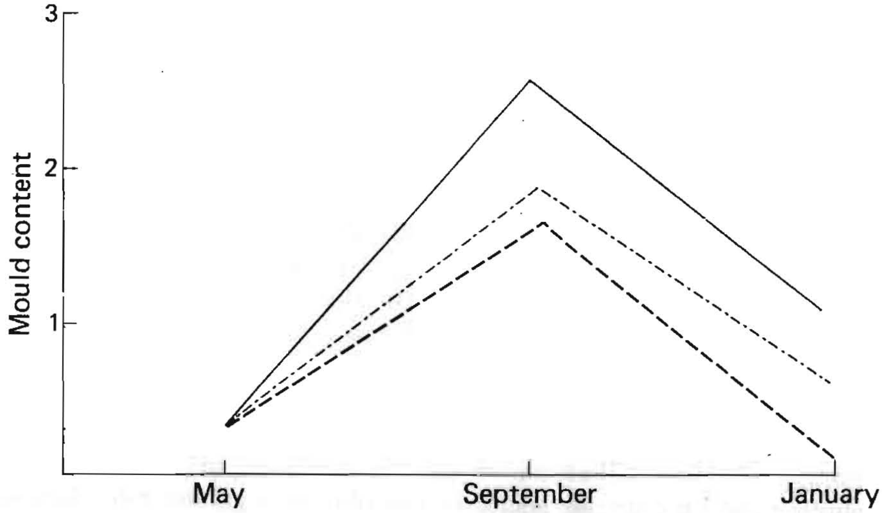

Fig. 10. The variation in mould content with season of rice stored in hermetic conditions.

prevailing temperatures, the grain quality does not deteriorate obviously, although various degrees of starch aging may take place.

The effect of the gas composition of the storage atmosphere on grain quality (Table 3) is much less than that of temperature. In experiments on rice stored under 98.5% CO_2, 1.5% O_2 at various temperatures it was found that temperature influenced the quality even when kept under CA. Aging of rice still occurs after storage

TABLE 2.

Variation in quality of rice stored under various CO_2 atmospheres for 6 months (temperature 13-20°C, moisture content 16.2%)

Storage atmosphere CO_2%	O_2%	Cooking qualities	Acidity of water extract (ml N ROH/ 10 g d.m.)	Fat acidity (mg KOH/ 100g d.m.)	Viscosity (c.st.)
0	21	50	3.17	29.4	-
75-85	3-5	93	0.7	81.9	14.4
85-95	1-3	95	0.7	82.8	15.1
Original value		100	0.7	57.4	16.4

in summer with temperatures reaching 35°C. If the temperature is kept below 25°C the quality is maintained better under CA conditions. It is evident that CA storage is superior to ordinary storage (Figs. 11, 12).

3.4 Studies on Skin-Packing

Polyethylene/polyester composite films made in China are used for sacks of high grade rice of ca. 15.5% moisture content. Rice stored in these sacks at the standard temperature, 20°C, can be held for one to two years without degradation of quality from insects, overheating and other deterioration. The ageing of the rice is retarded.

It is claimed that the best storage atmosphere is made by creating a vacuum in a sack and replacing this vacuum by CO_2. The second best system is to replace the air directly by CO_2. These methods give a skin-packed product. Filling with nitrogen only does not give the skin-packing effect and is inferior, but still better than simple airtight storage. With simple airtight storage the packing film shrinks onto the grain as the CO_2 produced in the pack is absorbed. This takes at least 24 hours. Simple airtight storage is less convenient to use than the vacuum packing method.

It is found that the lower the temperature, the better the quality of the stored grain.

The quantity of CO_2 taken up in the skin-packing method has been investigated. Over 50 bags of 16 x 24 cm polyethylene/polyester film were filled with Grade II rice of 15.1% moisture content. The air in the bags was evacuated with a vacuum packing machine. The packs were then sealed and held at 12°C for 4 to 5 days. After

TABLE 3

Variation of quality of rice under various atmospheres for 3 and 8 months.

	Natural oxygen-deficient atmosphere			CO_2			Air		
	initially	after 3 mths	after 8 mths	initially	after 3 mths	after 8 mths	Initially	after 3 mths	after 8 mths
Acidity of water extract (ml N KOH/ 10g.d.m)	0.7	1.1	0.62	0.7	1.8	0.37	0.7	0.65	0.40
Fat acidity KOH mg/ 100g.d.m.	7.4		73.1	7.4		58.4	7.4		45.2
Hardness (kg/kernel)	4-7		4-7	4-7		3-6	4-7		4-7
(%)	76		74	76		84	76		79
Iodine-Blue value (transmission, %)	53	77	71	57	78	68	53	67	64
Specific viscosity ($E^{50^\circ}_{20^\circ C}$)	1.71	2.06	1.80	1.82	2.06	2.22	1.71	1.85	1.94
Amylogram									
Gelatinization temperature (°C)	83	84	81	83	85	80	83	85	81
Temperature of peak viscosity (°C)	93	94	89	93	94	89	93	94	89
Peak viscosity (B.U.)	680	780	790	730	950	1000	680	860	860
Final temp. of viscosity (°C)	97	98	95	97	89	96	97	98	96
Final point of viscosity (B.U.)	540	590	630	560	720	660	540	660	660
Cooking qualities									
Lustre			5			5			5
Taste			5			5			5
Freshness			5			4			5
Cohesiveness			4			4			4

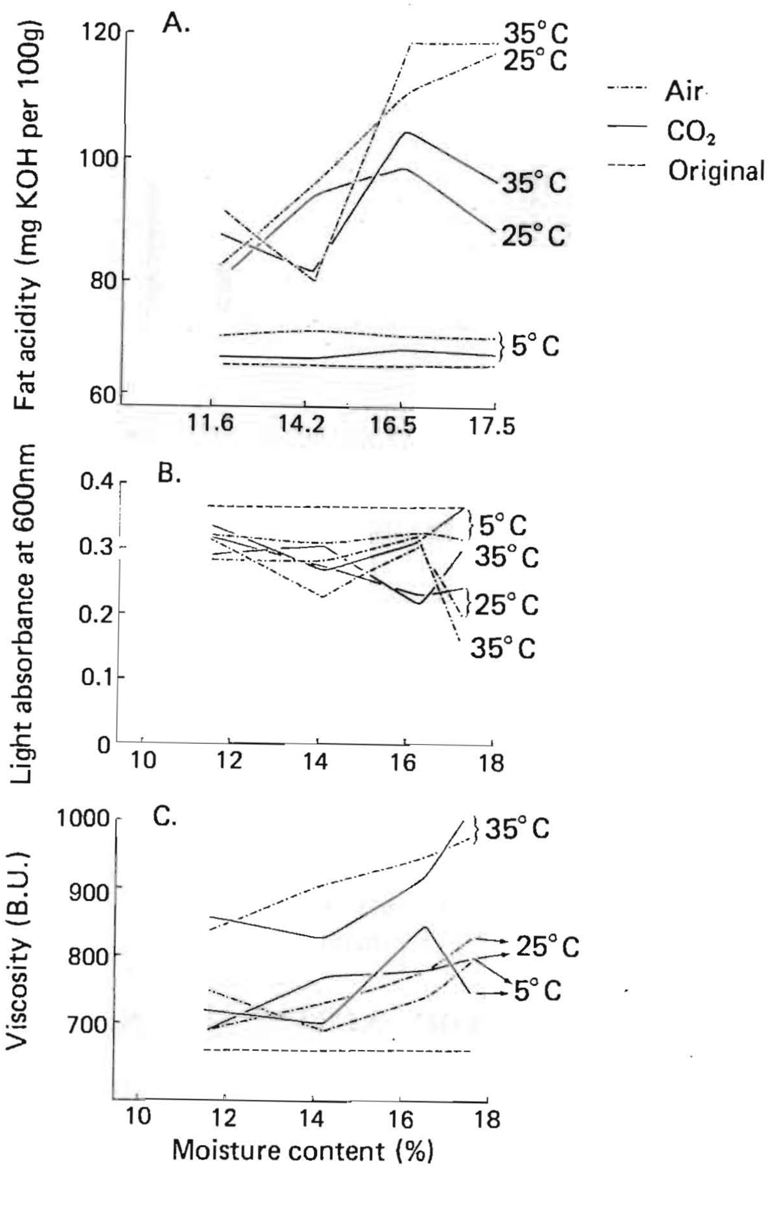

Fig. 11. (A) Variation in free fatty acid content in rice stored in air and CO_2 at three temperatures. (B) Variation in iodine-blue reaction of rice starch in rice stored in air and CO_2 at three temperatures. (C) Variation in paste viscosity from rice stored in air and CO_2 at three temperatures.

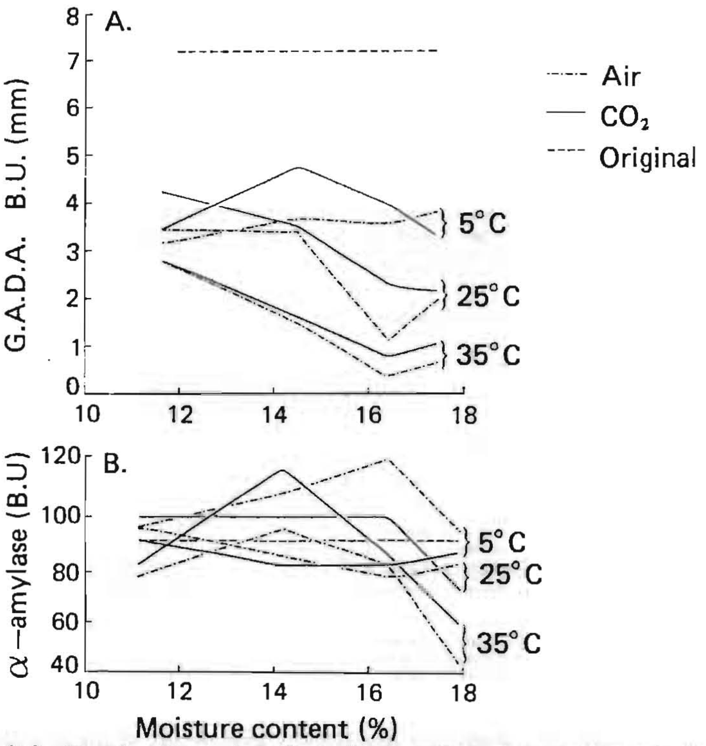

Fig. 12. (A) Variation in G.A.D.A. in rice stored in air and CO_2 at three temperatures. (B) Variation in α-amylase content of rice stored in air and CO_2 at three temperatures.

checking for any leaks in the film or its sealing, CO_2 was admitted to each pack through a flow meter. The quantity of CO_2 taken in was calculated from the time taken to refill the pack.

The quantity of CO_2 absorbed averaged 73 ml kg^{-1}. After allowance for diffusion through the packing film, this corresponds to an absorption of 69 ml CO_2 kg^{-1}. In Japan, where polyethylene/polyamide films are used, the absorption is reported to be 70 ml CO_2 kg^{-1}, a value close to that we observed.

4. OUTLOOK

CA techniques were initially applied to rice storage through the summer. This successful technique has been unanimously welcomed by personnel engaged in grain storage. Application of the hermetic storage technique using plastic films is inexpensive and is gradually being extended to the storage of maize and wheat. In state-owned or commercial storage in large cities, CA storage using either natural oxygen depletion or filling with CO_2 is widely adopted. Both the 'one-side' and 'six-side' sealing systems are used. The use of CO_2 seems preferable to filling with N_2. The use of multi-

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† Editor's Note: These references are not cited in the text. The editor is grateful to Dr R. Conroy for translating the references into English from Chinese.

PLANT QUARANTINE AND FUMIGATION OF IMPORTED CEREALS IN JAPAN

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ABSTRACT:
In 1982, more than 30 million tonnes of cereals were imported into Japan. As much as 60% of these imports were fumigated at the port of entry, either in the silo, warehouse, container or barge, in order to prevent the entry of injurious insect pests. Remarkable progress has been achieved since 1950 in techniques of cereal fumigation with methyl bromide and phosphine for "quarantine fumigation". Most silos and warehouses that are currently approved by the Plant Protection Station are sufficiently gas tight to be suitable for fumigation. The high level of sealing results from the development of suitable construction and sealing techniques.

Practical aspects of quarantine fumigation in Japan and Japanese research on factors affecting fumigation and fumigant residues in cereals are described. Japanese quarantine fumigation schedules are given. Dosage-response data for methyl bromide treatment of the most tolerant stages of several stored product pests is summarised.

HISTORY AND SYSTEM OF PLANT QUARANTINE SERVICE

Plant Quarantine Service in Japan was initiated in 1914 by the promulgation of the Plant Quarantine Law. At first only live plants were inspected. With the increase in international exchange of agricultural products, the objects of inspection have been expanded. Import quarantine of cereals was initiated in 1950 under the current Plant Protection Law.

At present, the Plant Quarantine Service in Japan is carried out by the Plant Protection Station under jurisdiction of Plant Protection Division, Agricultural Production Bureau, Ministry of Agriculture, Forestry and Fisheries. Japan is divided into five blocks under the headquarters which are located in Yokohama, Nagoya, Kobe, Moji and Naha respectively. Each division has branches and sub-branches at major sea and air ports, where plant inspectors are stationed. The total number of the stations including branches and sub-branches in 1983 is 96, and about 600 plant inspectors are engaged in import and export inspection.

INSPECTION AND TREATMENT OF IMPORTED CEREALS

The quantity of grains and beans imported has increased year after year. In 1982, 25 million tonnes of grains and 5 million tonnes of beans were imported, largely from Australia, Canada, U.S.A. and Argentina (Table 1). Together with timber, inspection of grains and beans occupies a great share in the total work load of import quarantine.

Table 1.
Amount of grains imported into Japan and proportion treated by plant quarantine fumigation in 1978-1982

	Grains		Beans	
Year	Imported (tonnes)	Fumigated (%)	Imported (tonnes)	Fumigated (%)
1978	24,150,000	67.6	4,850,000	76.5
1979	25,710,000	66.4	4,620,000	71.0
1980	25,550,000	60.7	5,070,000	70.2
1981	25,120,000	56.2	4,710,000	67.7
1982	25,480,000	62.7	4,730,000	77.2

In parallel with this large increase in imported grains and beans, interceptions of insects of quarantine concern have been also on the increase. More than 100 species of stored-product insects were intercepted by the inspection in past five years, including many species of economic importance not known in Japan. These include, *Sitophilus granarius* , *Trogoderma granarium*, *Callosbruchus rhodesianus* and *Zabrotes subfasciatus*.

When injurious pests are found upon inspection of grains and beans, their import is permitted only after complete disinfestation. As much as 60% of imported cereals were fumigated at the port of entry in the silo, warehouse, container or barge in order to prevent the entry of injurious insect pests.

METHODS OF QUARANTINE TREATMENT OF IMPORTED CEREALS

Since the first trial of fumigation of stored products with carbon disulphide performed in 1897 in Japan, a variety of fumigants such as chloropicrin, hydrocyanic acid, ethylene oxide, methyl bromide and aluminium phosphide have been used in Japan. Methyl bromide was introduced in Japan in 1948. Since it can be used at low temperatures, it has gained the status of a major fumigant for quarantine treatment since 1950 when import quarantine of cereals began. Aluminium phosphide was introduced as a quarantine fumigant in 1971. Its use is limited only to cereals which sorb much methyl bromide, since it requires a much longer exposure period than methyl bromide. Production and importation of these fumigants in Japan are shown in Table 2.

Table 2.
Quantities of some fumigants (tonnes) produced in or imported by Japan

Year	1977	1978	1979	1980	1981
Methyl Bromide	4791	6091	6223	6609	5630
Aluminum phosphide	25	37	51	44	47

Fumigation Facilities:

Warehouses and silos for plant quarantine fumigation must be approved by Plant Protection Station. Facilities for quarantine fumigation are divided into three classes according to their gastightness. To test gastightness, methyl bromide gas is introduced into an empty fumigation facility at a dose rate of 10 g m^{-3}. The facility is classified "A" when the concentration of methyl bromide averaged from three different points at upper, middle and lower of the facility is more than 7 g m^{-3} 48 hours after dosing; "B", 7 - 5.5 g m^{-3}; "C", less than 5.5 g m^{-3} respectively. Alternatively a pressure test is applied to test the gastightness of silos. The internal pressure of a silo is raised to 5,000 Pa. If the time taken to fall to 2,000 Pa. is longer than 20 minutes, the silo is classified "A".

During the early years of the quarantine fumigation of cereals, the gastightness of the warehouses and silos was not sufficient for fumigation. However, with the development of the economy and increase of international trade of agricultural products, the storage facilities have been completely modernized and many multi-storied ferroconcrete warehouses have been constructed. As much as 80% of the total number of warehouses that are currently approved by the Plant Protection Station are sufficiently gastight for fumigation.

In 1950, only a few silos were available for fumigation. Moreover, no silo was equipped with a gas circulation system. However, at present, most silos are sufficiently gastight for fumigation and are equipped with a vapourizer for methyl bromide and a modern recirculation system (Table 3). Most silos are of ferroconcrete or steel. The average capacity of a bin is about 500 tonnes but there are some bins of more than 4000 tonnes capacity.

Table 3.
Number of warehouses and silo bins in 1982 in Japan approved by the Plant Protection Station for fumigation

Classification	Warehouses	Silo Bins
A	2,121	8,994
B	454	7
C	123	22
Total	2,698	9,023

Fumigation Schedules

The current dosages and exposure periods set for quarantine fumigation with methyl bromide have been established on the basis of both basic and practical experiments. In plant quarantine work the object of fumigation is to obtain complete mortality of all stages of the insect pests against which treatment is directed. For this reason, the dosage based on the susceptibility to methyl bromide of *T. confusum* or *T. castaneum* pupae, relatively tolerant species and stages to methyl bromide, have been applied against almost all insect pests. In 1977, a lower dosage against *Sitophilus* spp., which are very sensitive to methyl bromide, was established. Factors that require to be taken into account in methyl bromide fumigation are as follows; a) fumigation facilities b) gastightness c) kinds of commodities d) load e) temperature f) susceptibility of insects to methyl bromide g) exposure period h) gas circulation.

The dosage of methyl bromide used and exposure period for quarantine fumigation of imported cereals in the facilities classified "A" are shown in Table 4. The dosage of aluminium phosphide and exposure period for quarantine fumigation of imported cereals in the facilities classified "A", "B" and "C" are shown in Table 5.

Apparatus for Measuring Gas Concentrations

In 1956, the Riken interferometer was introduced into quarantine fumigation for measuring gas concentrations. This indicator is now commonly used to check the validity of each quarantine fumigation by the plant inspector. Gas detector tubes, Kitagawa, Gastec and Drager, have been used for the detection of methyl bromide and phosphine in the TLV region. A halide leak detector is used for the detection of leaks of methyl bromide from warehouses. Gas chromatography is commonly used for laboratory fumigation experiments for estimation of both methyl bromide and phosphine.

Table 4.
Dosage and exposure period for methyl bromide for quarantine fumigation of imported cereals

Fumigation facility	Species of insect pest	Exposure period (hours)	Temperature (^{o}C)	Dosage ($g\ m^{-3}$) Cereal type (*a*) (1)	(2)	(3)	(4)
Warehouse with gas circulation system (Bagged cereals)	*S. zeamais* and *S. oryzae*	24	20 and above	15	18	20	23
			10 - 19	20	24	28	32
			below 9	35	42	49	56
	T. confusum and others (*b*)	24	20 and above	21	25	29	33
			10 - 19	29	34	40	45
			below 9	35	42	49	56
	"	48	20 and above	13	17	21	25
			10 - 19	17	23	29	34
			below 9	21	28	35	42
	"	72	20 and above	12	15	19	25
			10 - 19	16	21	27	34
			below 9	21	27	34	42
Silo bin with gas recirculation system	*S. zeamais* and	24	20 and above	20	25	30	-
			10 - 19	29	34	41	-
			below 9	49	59	59	-
	T. confusum (*b*)	24	20 and above	29	35	35	-
			10 - 19	41	48	48	-
			below 9	49	59	59	-
	"	48	20 and above	18	24	25	-
			10 - 19	24	32	34	-
			below 9	29	39	42	-
	"	72	20 and above	16	22	23	-
			10 - 19	22	30	31	-
			below 9	28	38	41	-

a) Cereals are divided into four groups according to their degree of methyl bromide sorption as follows; (1) Low : rice, wheat, barley, barley malt (2) Relatively low : maize, sorghum, millet (3) Relatively high : soybean, ground nut, rape seed (4) Very high : buckwheat, safflower seed, powder products, pellets.

b) Double the dosage for *T. confusum* and others is used against *Trogoderma granarium*.

Fumigation Practice

Fumigation is not carried out directly by the plant quarantine inspectors. Quarantine inspectors check the application of fumigants into the fumigation facilities and the effectiveness of the treatment by measuring gas concentrations and by bioassay. Usually *T. confusum* is used as a test insect.

Table 5.
Dosage of phosphine and exposure period used for quarantine fumigation of cereals (*a*)

Fumigation facility	Classification	Dosage (g m^{-3} as PH_3)	Temperature (°C)	Exposure period (days)
Warehouse (bagged cereals)	A	0.5	20 and above	5
	B	0.75	10 - 15	6
	C	1.0	5 - 9	7
Silo bin	A, B, C	2.0	20 and above	3 - 5
			15 - 19	4 - 6
			10 - 14	5 - 7
			7 - 9	6 - 8
			5 - 6	7 - 9

a) This schedule does not apply for cereals infested with *Sitophilus zeamais*, *S. granarius*, *S. oryzae* and *T. granarium*.

At present, quarantine fumigation of cereals is performed by commercial fumigation companies, equipped with such instruments and materials required for fumigation (eg. gas measuring equipment, gas masks, sealing materials and first-aid equipment). Employees in charge of fumigation operations must attend a special training course organized by the Plant Protection Station. The course includes the following subjects: techniques of warehouse fumigation, silo fumigation and safety arrangements related to fumigation. Persons working regularly with fumigants must have blood tests and physical examinations.

RECENT RESEARCH ON QUARANTINE FUMIGATION OF CEREALS

Some aspects of fumigation as a plant quarantine treatment still remain to be fully developed. Studies are required still on safe and efficient fumigation. Remarkable progress on fumigation techniques and gas measuring apparatus, such as gas chromatograph, have occurred in the past ten years. Also, many laboratory experiments have been conducted on the susceptibility of insect pests to methyl bromide, sorption of methyl bromide to cereals and residues in commodities in order to improve fumigation schedules. The research for quarantine in Japan are summarised below.

Susceptibility of Stored-Product Insects to Methyl Bromide

Methyl bromide has been used as a fumigant to control stored-product insects for more than forty years, but basic data on the susceptibility of major insect pests to this fumigant under long exposure periods was insufficient.

The effects of temperature and exposure period in tests with methyl bromide on the developmental stages of *Tribolium confusum*, *Sitophilus zeamais*, *Trogoderma granarium*, *Callosobruchus maculatus*, *C. rhodesianus* and *Ephestia kuehniella* were studied. These test insects were reared at 26°C, 70% R.H., except *T. granarium* which was reared at 30°C, 70% R.H. Laboratory experiments based on the recommended FAO Method No. 16 (FAO 1975) were conducted at various temperatures (5, 15 and 25°C) and exposure periods (5, 24 and 48 hours). Methyl bromide gas was injected into the specially designed 5-litre glass jar. The gas concentrations present were measured using a gas chromatograph equipped with a flame ionization detector and concentration-time products (*CT*-products) were calculated, based on measured gas concentrations. The mortality of immature stages of insects was determined by adult emergence and the mortality of adults was as sensed after a post-exposure interval of two weeks. Mortality data was analysed by probit analysis. LC_{50} and LC_{99}'s were estimated from dose-probit mortality curves.

LC_{50} and LC_{99}'s for the most tolerant stages of six species of stored-product insects at various temperatures and exposure periods are shown in Table 6. The results indicated that the *CT*-products required for 99% mortality increased with an increased exposure period at 25°C in the six species of insects tested and at 15°C in *T. granarium*, *C. maculatus* and *E. kuehniella*, respectively. Whereas, they were almost constant at any exposure period at 15°C in *T. confusum* and *S. zeamais* and at 5°C in *C. maculatus*, *C. rhodesianus* and *E. kuehniella*. In temperatures ranging from 5°C to 25°C, the greatest susceptibility was shown by *S. zeamais* pupae and *E. kuehniella* pupae when the exposure period was 48 hours. The susceptibility of *S. zeamais* was higher than that of *T. confusum* at any exposure period at 15°C and 25°C, but there were little differences between two species at 5°C. The susceptibility of *C. maculatus*, *C. rhodesianus* and *E. kuehniella* was higher

Table 6.
Susceptibility to methyl bromide of the most tolerant stages of some stored-product insect pests at various temperatures and exposure periods.

Species	Stage	Exposure period (hours)	Temp. (^{o}C)	LC_{50}		LC_{99}	
				g m^{-3}	g h m^{-3}	g m^{-3}	g h m^{-3}
Tribolium confusum	Pupae	5	25	12.9	64.5	15.3	76.5
		5	15	28.2	141.0	35.2	176.0
		5	5	39.6	198.0	63.1	315.5
		24	25	3.7	88.8	4.2	100.8
		24	15	5.7	136.8	7.6	182.4
		24	5	11.3	271.2	17.4	417.6
		48	25	2.7	129.6	3.2	153.6
		48	15	2.7	129.6	3.4	163.2
		48	5	3.3	158.4	5.5	264.0
Sitophilus zeamais	Pupae	5	25	5.2	26.0	10.9	54.5
		5	15	10.7	53.5	17.1	85.5
		5	5	19.1	95.5	80.7	403.5
		24	25	1.6	38.4	2.9	69.6
		24	15	1.3	31.2	4.2	100.8
		24	5	2.7	64.8	15.5	372.0
		48	25	1.2	57.6	2.9	139.2
		48	15	1.2	57.6	2.1	100.8
		48	5	1.8	86.4	6.8	326.4
Trogoderma granarium	Diapausing larvae held for 2 months at 30^{o}C	5	25	14.7	73.5	27.5	137.5
		5	15	32.9	164.5	73.2	366.0
		24	25	6.4	153.6	8.9	213.6
		24	15	8.5	204.0	25.7	616.8
		24	5	22.7	544.8	40.5	972.0
		48	25	4.0	192.0	6.5	312.0
		48	15	6.2	297.6	8.7	417.6
		48	5	10.6	508.8	18.6	892.8

Callosobruchus maculatus	Pupae	5	25	3.0	15.0	4.9	24.5
		5	15	6.0	30.0	9.4	47.0
		5	5	9.0	45.0	16.4	82.0
		24	25	1.4	33.6	2.2	52.8
		24	15	0.9	21.6	3.0	72.0
		24	5	2.0	48.0	3.5	84.0
Callosobruchus rhodesianus	Pupae	5	25	4.6	23.0	8.3	41.5
		5	15	5.3	26.5	12.6	63.0
		5	5	7.1	35.5	25.6	128.0
		24	25	1.5	36.0	2.5	60.0
		24	15	1.4	33.6	2.5	60.0
		24	5	3.0	72.0	5.6	134.4
Ephestia kuehniella	Pupae	5	25	5.8	29.0	7.5	37.5
		5	15	8.6	43.0	11.7	58.5
		5	5	23.2	116.0	32.1	160.5
		24	25	1.8	43.2	3.1	74.4
		24	15	1.9	45.6	2.7	64.8
		24	5	4.5	108.0	6.8	163.2
		48	25	0.9	43.2	2.0	96.0
		48	15	1.1	52.8	1.8	86.4
		48	5	2.2	105.6	3.3	158.4

than that of *T. confusum* at any temperatures and exposure period. Diapausing larvae of *T. granarium* showed exceptional tolerance to methyl bromide among six species tested.

Residues of Fumigants in Cereals after Quarantine Fumigation

Consumer's concern on fumigant residues in imported cereals after quarantine fumigation has increased in recent years. Work on the residue level of some fumigants in commodities resulting from quarantine treatment has been carried out in our laboratory in response to this concern.

Methyl Bromide

Effects of aeration and processing on methyl bromide and total bromide residues in wheat and soybean fumigated with methyl bromide under quarantine treatment conditions were investigated. Methyl bromide residues were analyzed by the method of Asaka and Seguchi (1974). Methyl bromide, liberated by a modified sweep codistillation method (Malone 1969) or acid reflux method, was converted to O,O-diethyl dithiophosphate in acetone. This derivative was determined by gas chromatography using a flame photometric detector. The maximum detectable level was 0.002 ppm with a 50 gram sample. Total bromide residues were analyzed according to modifications of the recommended FAO/WHO method. The minimum sensitivity of this method was 1 ppm in a 5 gram sample.

The residues of unchanged methyl bromide in wheat were over 1 ppm one day after treatment at 5°C and 15°C, and then decreased rapidly as shown in Table 7. However, the residues were only 0.08 ppm one day after treatment at 25°C, and then decreased gradually. Very little amount of methyl bromide retained in fumigated wheat over 1 to 3 months. The residue level of total bromide was almost constant at any storage period. After baking, no residual methyl bromide was detected. No residual methyl bromide was detected and only 1 ppm total bromide remained in tofu (bean curd) made from treated soybeans (Table 8).

Aluminium Phosphide

Effects of fumigation conditions and storage on the residues of phosphine in some raw cereals fumigated with phosphine generated from aluminium phosphide were studied. Phosphine residues were analyzed using a modification of the method of Bruce *et al.* (1962). The phosphine liberated by distillation was oxidized to phosphoric acid with bromide and determined colorimetrically. With a 500 gram sample and with the phosphate in 5 ml of solution, 0.002 ppm of phosphine could be determined. Recoveries of phosphine from untreated wheat by adding 0.1 and 0.05 ppm phosphine were 73.2% and

Table 7.

Effect of aeration on the residues of methyl bromide and total bromide in wheat fumigated with methyl bromide under various conditions (*a*).

Variety (Origin)	Treatment temperature	Dosage $g\ m^{-3}$	Days after (*b*) treatment	Residues (ppm w/w)	
				Methyl bromide	Total bromide
Red Spring (Canada)	9	Untreated	-	ND (*c*)	5
	5	29	1	1.48	13
			7	0.132	12
			14	0.030	14
			21	0.014	-
			28	0.011	14
Red Spring (Canada)	9	Untreated	-	ND (*c*)	7
	15	24	1	1.29	27
			7	0.040	21
			14	0.031	23
			91	0.031	22
Hard Winter (USA)	9	Untreated	-	0.004	7
	24	18	1	0.080	29
			7	0.051	30
			14	0.042	28
			28	0.045	29
			62	0.027	30

a) Load : 0.54 - 0.56 $t\ m^{-3}$, exposure period : 48 hours, data averaged from three replicates.

b) Samples were stored at the treatment temperature in the paper bag.

c) ND: not detected.

Table 8.

Effect of processing on the residues of methyl bromide and total bromide in wheat and soybeans fumigated with methyl bromide (*a*)

Commodity (Origin)		Residues (ppm w/w)			
		Before processing		After processing	
		Methyl bromide	Total bromide	Methyl bromide	Total bromide
Wheat, hard red winter (USA)	Untreated	ND	10	ND	11
	Treated	0.006	17	ND	15
Soybean (China)	Untreated	0.003	4	ND	ND
	Treated	0.004	29	ND	1

a) Bread was made from wheat and tofu (bean curd) was made from soybean. Data averaged from three replicates. Fumigation conditions were as follows; Wheat: Dosage 24 g m^{-3}, Temperature 15°C, Exposure period 48 hours, Load 0.56 t m^{3}. Soybean : Dosage 34 g m^{-3}, Temperature 15°C, Exposure period 48 hours, Load 0.5 t m^{-3}. Samples were stored at the treatment temperature.

b) Analyzed 3 days after treatment.

c) Analyzed 7 days after treatment.

d) Analyzed 14 days after treatment.

Table 9.

Residues of phosphine on some cereals fumigated with aluminium phosphide under various conditions in the laboratory (*a*)

Commodity (Origin)	Moisture content %	Fumigation conditions Temperature (°C)	Exposure period (days)	Days after treatment	Phosphine residues found (ppm w/w)
Maize	12.6	25	5	7	0.034
(USA)	12.6	25	5	14	0.012
	13.6	25	5	1	0.043
	13.6	25	5	6	0.014(*b*)
	12.6	15	8	6	0.014
	12.6	15	8	6	0.025
	12.6	15	6	7	0.028
	12.6	5	7	7	0.022
	13.6	5	9	1	0.030
	13.6	5	9	6	0.006
Wheat (Canada)	10.6	25	5	1	0.010
"	10.6	25	5	6	0.009
(USA)	12.1	25	5	7	0.008
"	12.1	15	6	7	0.018
"	12.1	5	7	7	0.005
(Canada)	10.6	5	9	1	0.010
"	10.6	5	9	6	0.011
Sorghum	12.5	25	5	7	0.026
(Argentina)	12.5	15	6	7	0.054
	12.5	5	7	7	0.023
Malt	6.3	25	5	7	0.037
(Belgium)	6.3	15	6	7	0.021
	6.3	5	7	7	0.002
Soybeans	10.6	25	5	1	0.047
(USA)	10.6	25	5	6	0.007
	10.6	5	9	1	0.058
	10.6	5	9	6	0.023

a) Dosage : 2 g m^{-3} as phosphine, Load : 0.17 kg L-1. Data averaged from 2 or 3 replicates.

b) Milled.

72.2% respectively.

The residues of phosphine in maize, wheat, sorghum, malt and soybeans ranged between 0.002 ppm and 0.058 ppm, 1 to 7 days after treatment under any fumigation conditions (Table 9). No consistent relationship was found between temperature, exposure period and residues. Aeration and milling proved very effective in removing any residues present.

Maximum Residue Limit of Fumigants in Japan

The maximum residue limit of unchanged methyl bromide has not been established. In Japan the MRL of total bromide in raw wheat fumigated with methyl bromide is 50 ppm. The MRL of phosphine in rice, wheat, beans and other raw cereals fumigated with aluminium phosphide is 0.1 ppm. Both total bromide and phosphine residues in wheat after plant quarantine fumigation did not exceed these MRLs.

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SESSION 2.

ENTOMOLOGY OF CONTROLLED ATMOSPHERE STORAGE

Papers by:

F. I. Attia	-	Australia
P. Williams: W. Minett: P. Savage: A.D. Wilson: S. A. Buchanan and V. Guiffre	-	Australia
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MULTIPLE AND CROSS-RESISTANCE CHARACTERISTICS IN PHOSPHINE-RESISTANT STRAINS OF *RHYZOPERTHA DOMINICA* AND *TRIBOLIUM CASTANEUM*

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ABSTRACT

Phosphine-resistant strains of lesser grain borer, *Rhyzopertha dominica* (Fabricius), and rust-red flour beetle, *Tribolium castaneum* (Herbst) were tested for cross-resistance to DDT, lindane, malathion, fenitrothion, carbaryl and bioresmethrin. DDT and bioresmethrin were applied topically, and the impregnated filter paper method was used to test the other insecticides.

Comparisons with laboratory susceptible strains showed that two phosphine-resistant strains of *R. dominica* with resistance factor (RF) 8-10x, and one of *T. castaneum* (RF 5.3x), had no cross-resistance to all insecticides used. Another phosphine-resistant strain of *R. dominica* (RF 8.6x) was also resistant to malathion and fenitrothion (RF 7.2; 4.8x respectively Furthermore, a phosphine-resistant strain of *T. castaneum* (RF 6.4x) was also resistant to lindane and malathion (RF 5.2 and 77.9x respectively). These data show that there is no obvious correlation between the response to phosphine and the DDT, lindane, organophosphorus, carbaryl and bioresmethrin resistance status in these insects, but some strains have multiple resistances to pesticides including phosphine.

INTRODUCTION

Grain insects are usually controlled by insecticides applied to the grain on intake at the storage in Australia, but as storage time increases the residues can fall below the lethal level and re-infestation can occur. Turning of the grain and re-treatment with the currently used grain protectants is expensive and can also cause increased selection pressure on insects with a more rapid development of resistance to these insecticides. Where this selection has occurred fumigation with phosphine is the usual method of disinfestation.

Phosphine has been highly effective against strains of grain insects that are resistant to contact insecticides. However, resistance to phosphine has now been identified (Attia and Greening, 1981) and it is essential, therefore, to determine whether or not development of this resistance will exacerbate resistance to grain protectants. This paper provides data on cross-resistance to insecticides in phosphine resistant strains of lesser grain borer, *Rhyzopertha dominica* (Fabricius) and rust-red flour beetle, *Tribolium castaneum* (Herbst).

TABLE 1

Dose response data+ obtained by the impregnated filter paper method (FP) or the topical application method (TA) for several insecticides using phosphine-resistant strains of *R. dominica* and *T. castaneum*[1]

Strain and RF of phosphine	DDT (TA)		Biores (TA)		Lind (FP)		Mala (FP)		Fenitro (FP)		Carb (FP)	
	LD50	RF[2]	LD50	RF	KD50	RF	KD50	RF	KD50	RF	KD50	RF
R. dominica												
CRD2 (S)	0.025	-	0.00032	-	0.018	-	0.44	-	0.15	-	0.063	-
NRD184 (x7.7)	0.034	1.4	0.00041	1.2	0.022	1.2	0.49	1.1	0.21	1.4	0.064	1.0
NRD197 (x8.6)	0.035	1.5	0.00040	1.2	0.027	1.5	3.15	7.2	0.73	4.8	0.075	1.2
NRD459 (x9.9)	0.031	1.5	0.00031	1.0	0.021	1.2	0.48	1.1	0.22	1.5	0.079	1.3
T. Castaneum												
NTC138 (S)	0.28	-	0.035	-	0.28	-	0.17	-	0.11	-	0.81	-
NTC395 (x6.4)	0.38	1.4	0.045	1.2	1.46	5.2	13.2	77.9	0.14	1.3	0.83	1.0
NTC414 (x5.3)	0.35	1.3	0.039	1.1	0.29	1.0	0.26	1.5	0.13	1.2	0.91	1.1

1 LD50 expressed as (%) with lindane, malathion, fenitrothion and carbaryl (FP), but μg/adult with DDT and bioresmethrin (TA).

\+ The slope of ld-p lines for susceptible and resistant strains ranged from 3.7 to 8 and X^2 values for goodness of fit were non-significant (P greater than 0.05).

2 Resistance factor (RF): LD50 for resistant strain divided by LD50 for susceptible strain.

MATERIALS AND METHODS

Insect Strains

R. Dominica:

Strains NRD184, NRD197 and NRD459, collected from N.S.W. in May 1974, June 1974 and March 1980 respectively, were diagnosed with the discriminating concentration (DC) recommended by F.A.O. (Anon., 1975) to be resistant to phosphine. The survivors of the DC were kept for breeding and were subjected to 9, 10 and 5 generations of selection respectively with phosphine. Resistance factors (RF's) to phosphine of x7.7, x8.6 and x9.9 respectively were thus obtained. They were compared with a standard susceptible strain CRD2.

T. castaneum:

Strains NTC395 (RF 6.4), NTC414 (RF 5.3), were diagnosed resistant to phosphine as described above. They were collected from N.S.W. in 1977 and 1978 and selected with phosphine for 8 generations. For comparison a susceptible strain NTC138 was used.

Cultures of susceptible and resistant strains of *R. dominica* and *T. castaneum* , established from field samples, were maintained at 27°C and 65% relative humidity. The diet used for *R. dominica* was whole wheat grain with 12% moisture content. *T. castaneum* was cultured in a mixture of finely ground wholemeal flour and dried yeast powder (12:1 by weight). Adults 2-3 weeks old were used for testing.

Chemical bioassay procedures

The insecticides used in these tests were technical grade (Table 1), dissolved in either ethyl methyl ketone or in Risella 17 oil for dosing. Using a method developed by Needham and Devonshire (1973), an automatic micro-applicator with a Hamilton gas-tight syringe of 250 micro litre capacity fitted with 0.2 mm O.D. stainless steel cannula was used to apply 0.08 and 0.1 ul of DDT and bioresmethrin dissolved in ethyl methyl ketone to the abdominal sternites of adults of *R. dominica* and *T. castaneum* respectively.

After treatment adults were placed in vials 4 cm diameter and 6 cm deep, supplied with flour and yeast (12:1), covered with perforated lids and held at 27 $\pm$ 2°C and 55% RH. Mortality was assessed 5 days after treatment. Adults showing no response to prodding with a needle were recorded as dead.

The insecticide impregnated filter paper was used to assess the response of the strains to lindane, malathion, fenitrothion and carbaryl, according to a method described by Champ (1968). Exposure periods of 24 h were used for *R. dominica* and for *T. castaneum* 5 h were used, with malathion, fenitrothion and carbaryl, and 24 h with lindane. After exposure the insects were examined and the numbers of adults responding were recorded. The criteria of response was mortality, as determined 5 days after for DDT and bioresmethrin and was knockdown for lindane, malathion, fenitrothion and carbaryl. Other results have shown that when insects are knocked down by these insecticides about 90% are killed.

Dose response data were analysed using the probit method of Finney (1971).

RESULTS AND DISCUSSION

Table 1 shows the LD50 values for each insecticide treatment and resistance factors for each phosphine resistant strain of *R. dominica* and *T. castaneum*. It can be seen from Table 1 that two phosphine-resistant strains of *R. dominica*, NRD184 (RF x7.7) and NRD459 (RF x9.9) had no cross-resistance to DDT, lindane, organophosphorus, carbaryl and bioresmethrin. The differences in response were greater than 1.5x at the LC50 level. Another phosphine-resistant strain, NRD197 (RF 8.6x) was also resistant to malathion and fenitrothion (RF 7.2 and 4.8x respectively). A similar situation exists with a phosphine-resistant strain (NTC414) of *T. castaneum*(RF 5.3x), which had no cross-resistance to all insecticides tested. Strain NTC395 (RF 6.4x) was also resistant to lindane and malathion with RF 5.2 and 77.9x respectively (Table 1).

The data presented in this paper and those published by Attia and Greening (1981) indicate that there is no correlation between the response to phosphine and responses to DDT, lindane, organophosphorus, carbaryl and bioresmethrin resistances in these species. Some strains, though, have multiple resistances to pesticides including phosphine. The results agree with the data given by Kem (1979) from his studies on the cross-resistant strain of *T. castaneum* in India. His results showed that there was no cross-resistance to contact insecticides. Also Monaro *et al.* (1972) showed no appreciable cross-resistance to other fumigants (except chloropicrin) in a strain of *S. granarius* selected for resistance to phosphine.

This study indicates that phosphine should be effective for controlling grain insects which survived treatment with other insecticides in sealed storages.

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COMMERCIAL POTENTIAL OF METHYL BROMIDE AND CARBON DIOXIDE MIXTURES FOR DISINFESTING GRAIN

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ABSTRACT

The commercial potential of methyl bromide and carbon dioxide gas mixtures, under recirculation, for grain insect control was investigated in a trial in a 2000 t welded steel silo. The trial aimed to achieve a *Ct*-product of 50 g/h/m^3 of methyl bromide in an atmosphere of 20% or more of carbon dioxide within 24 h. This is one third of the minimum recommended *Ct*-product for application of methyl bromide alone. Where the desired *Ct*-product or higher was achieved in an atmosphere containing 40-60% carbon dioxide all stages of *Tribolium castaneum*, *Rhyzopertha dominica*, *Sitophilus oryzae* and *S. granarius* were killed. The trial further indicated that bromide residues on grain can be reduced by at least two-thirds as a consequence of using a lower concentration of methyl bromide.

INTRODUCTION

Laboratory studies on the effects of mixtures of methyl bromide and carbon dioxide on insects have indicated that the toxicity of methyl bromide can be increased in the presence of carbon dioxide (Cotton and Young, 1929; Jones, 1938; Bond and Buckland, 1978).

More recently laboratory experiments by Williams (1982) demonstrated that the methyl bromide concentrations required to kill adults of the stored grain beetles *Sitophilus oryzae* (L.), *Rhyzopertha dominica*, (F.) and *Tribolium castaneum* (Herbst) can be reduced by a factor of less than 2 when the fumigant is used in conjunction with an atmosphere containing 20% or more of carbon dioxide in 24 h fumigations. Such gas combinations were effective under a wide range of temperatures and relative humidities. Further experiments demonstrated similar effects with immature stages of the beetles. Tests in 15 t wheat silos indicated that a dosage of 5 g/m^{-3} of methyl bromide (5 x lower than the standard recommended dose) in an atmosphere containing 20% or more of carbon dioxide, under continuous recirculation for 24 h, would control grain insects.

Calderon and Carmi (1973) carried out commercial trials with gas mixtures in which methyl bromide (liquid at 50 g/m^3) in conjunction with carbon dioxide (dry ice at 250 g/m^{-3}) was applied to the surface of grain in silos for insect control. Gas distribution depended on gravity and convection currents within the silos, with a minimum treatment time of 96 h for disinfestation. This treatment avoided the use of gas recirculation but required a higher dosage of methyl bromide and longer treatment time than for use of recirculated methyl bromide (Monro, 1969).

This paper describes a commercial trial, at Sanger in southern New South Wales, in which the effectiveness of methyl bromide in conjunction with carbon dioxide, under recirculation, was compared with a similar concentration of methyl bromide applied alone.

MATERIALS AND METHODS

Experimental Silo

A silver-painted, welded steel cylindrical silo (2000 t) with a conical roof welded to its walls was used for the trial. The silo had a concrete floor with a central opening for grain discharge and a Y-shaped perforated metal aeration duct. To guard against gas leakage the wall-to-floor joint was sprayed with a polyvinyl chloride resin formulation (Envelon). The outloading gate valve was treated with molten beeswax which set to form a seal that later could be readily broken to allow grain to be outloaded. A hinged steel plate lined with foam rubber was made to seal the manhole at the top of the silo. A pressure decay test (Banks and Annis, 1977) was carried out prior to fumigation and soap solution used to check for leaks. Leaks found were sealed with silicone rubber. Silo dimensions and details of the load are given in Table 1.

Monitoring Physical Conditions

Gas concentrations within the silo were sampled periodically from 20 locations (Fig. 1) using 4 mm (i.d.) semi-rigid nylon sampling lines attached to steel cables. Carbon dioxide concencentrations were measured by a thermal conductivity detector (Gow-Mac Portable Gas Analyser, Model 20-602) which sampled directly from the gas stream drawn through the gas lines by an electric pump. Methyl bromide concentrations were measured by taking 100 micro litres samples from the gas lines and injecting them into a Packard 427 Gas Chromatograph fitted with FID detectors and dual glass columns 1.5m x 2mm (i.d.) packed with Ucon oil (LB 550X) on Chromsorb W. HP. (60-80 mesh). Chromatograph temperatures were 60°C for the oven and 80°C for both the injection ports and detector block.

Ambient temperature and the grain temperatures at the gas sampling locations were monitored using a multi-point recorder (recording systematically from each point every 15 minutes). Grain moisture content was measured by determining the loss of weight on drying 2 g of ground wheat for 1 h in a ventilated oven at 130°C. Bromide levels in the wheat before and after fumigation were determined by X-ray fluorescence techniques (see Table 1).

Table 1.
Experimental Data

Silo dimensions	
Height to eaves	17.6
Height of cone	4.0m
Diameter	13.9m
Volume	287m^3
Load	
Grain	ASW grade wheat
Moisture content on intake	10.7% wet basis
Mass	2367t
Height of head space	1m
Volume of head space	50m^3
Load space	2822m^3
Estimated intergranualar volume (assuming 40% porosity)	1129m^3
Total gas volume	1179m^3
Average grain temperature	
- during experiment 1	31.7°C (13 -44.8°C)
- during experiment 2	30.0°C (10 - 23.5°C)
Average ambient temperature	
- during experiment 1	19.2°C (13 - 31.0°C)
- during experiment 2	15.9°C (10 - 23.5°C)
Insect species infest grain	R. dominica T. Castaneum Oryzaephilus surinamensis
Bromide levels on grain	
from surface before fumigation	8 ± 0.5 ppm
from surface after experiment 1	9 ± 1.0 ppm
from surface after experiment 2	12 ± 0.7 ppm
from location 13 after experiment 1	13 ± 0.7 ppm
from location 13 after experiment 2	15 ± 0.5 ppm

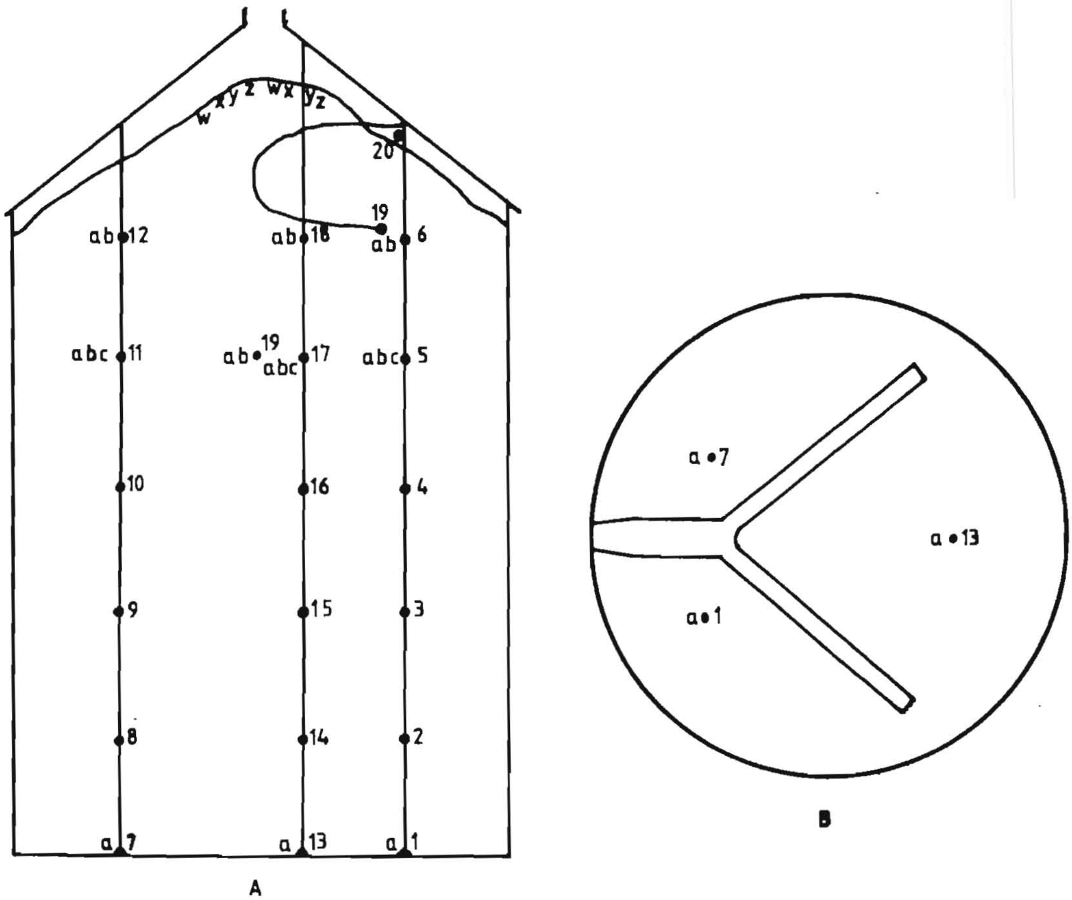

Figure 1.

A. Diagram of silo showing locations of gas sampling lines, thermocouples and insect cages.

B. Configuration of aeration duct and location of sampling points and insect cages on the silo floor.

- 1-20 Locations of gas sampling points and thermocouples.
- 19* Intended location of point 19 gas line and thermocouple.

Spear Cage	Pipe Cage	Insect Species
a	w	T. castaneum
b	x	R. dominica
c	y	S. granarius
	z	S. oryzae

Bioassays

Two types of insect cages were placed in the grain; spear shaped stainless steel cages 240mm long and 37mm in diameter fitted with steel gauze panels, and polyvinyl chloride pipe cages 75mm high and 167mm in diameter the ends of which were covered with steel gauze. The spear cages were probed into the grain, some being positioned during filling of the silo. The spear cages at the base of the silo were introduced via steel pipes 3.6m long and 51mm (i.d.) welded to the silo wall with 3.44m projecting inside the silo. The pressure of wheat at the ends of these pipes was so great that only the spear tips of the cages could be pushed into the wheat. The pipe cages were placed in the wheat just under the surface of the bulk at the top of the silo. Locations of the cages are shown in Fig. 1.

The bioassay insects were *T. castaneum* (strain CTC 12), *R. dominica* (CRC 118), *S. oryzae* (CSO 231), and *S. granarius* (VSG 24). All strains except VSG 24 were insecticide resistant. The cages each contained a single insect species, the spear cages for *T. castaneum* held 60 g of wheat and 10 g of breeding medium (wholemeal flour and yeast) containing an undetermined number of eggs, to which were added 200 adults (2-4 weeks old), 50 mature larvae and 50 young pupae. Spear cages carrying *R. dominica* and *S. granarius* held 70 g of wheat containing immature stages and 200 adults (2-4 weeks old).

Pipe cages carrying *T. castaneum* held 950 g of wheat and 50 g of wholemeal flour containing an undetermined number of eggs, to which were added 500 adults (2-4 weeks old), 50 mature larvae and 50 pupae. The pipe cages for *R. dominica* and *Sitophilus* spp. held 1000 g of wheat containing immature insects and 500 adults (2-4 weeks old).

Gas Recirculation System

The silo was fitted with rigid 0.23 m (i.d.) polyvinyl chloride piping for gas recirculation. Piping carried gas from a Richardsons E6118 fan (fitted with a 1.1 kW 3 phase motor and rated at 310 Ls^{-1}, 0.75kPa, 2940 rpm, 0.52 kW at 21^{o}C) to the aeration duct in the silo and collected gas for recirculating from the headspace. The fan gave an air change in about 80 minutes. The top of the recirculation duct was fitted with a hinged steel cap which was opened after fumigation to assist ventilation. The base of the duct was fitted with a water filled pressure safety valve (minimum pressure 0.75 kPa).

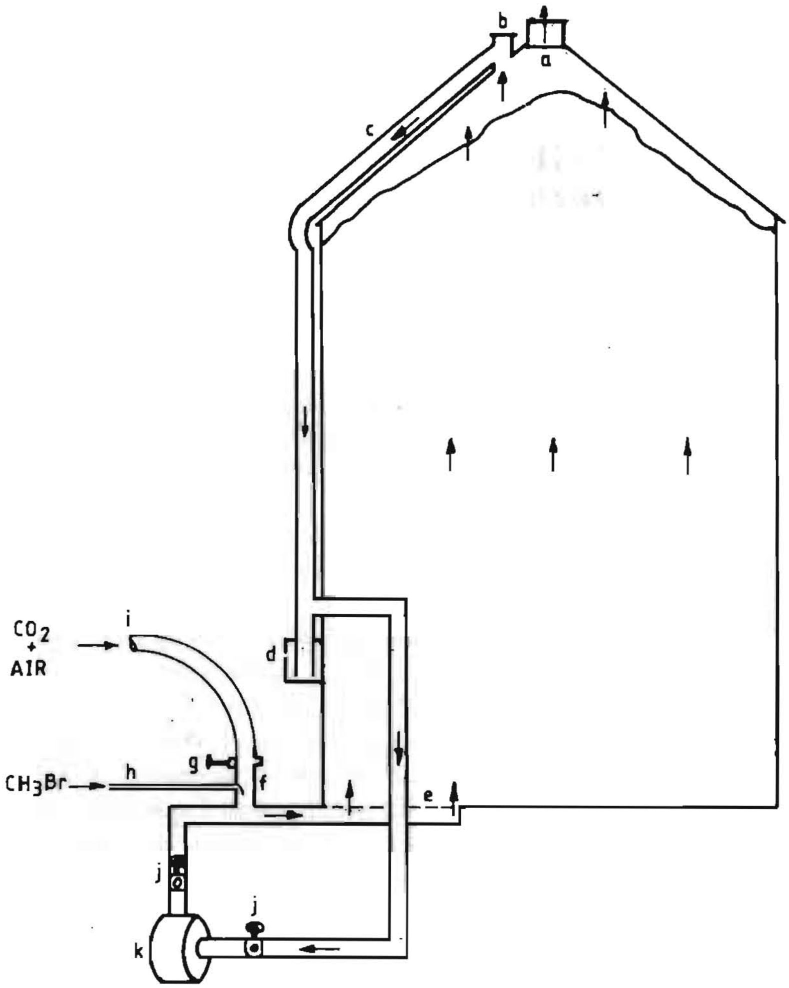

Figure 2.
Diagram of silo showing gas input and recirculation systems
a. manhole sealing plate,
b. cap on recirculation duct,
c. recirculation duct,
d. safety valve,
e. aeration duct,
f. steel pipe,
g. gate valve,
h. methyl bromide hose,
i. carbon dioxide hose,
j. ball valve,
k. gas recirculation fan.

Gas Introduction

It was decided to modify the carbon dioxide application technique developed by Wilson *et al.* (1980) so as to introduce a methyl bromide and carbon dioxide mixture into the silo. On the basis of laboratory and small scale field trial results, Williams (1982), it was decided to aim at obtaining a *Ct*-product of 50 g/h/m^{-3} of methyl bromide in an atmosphere containing 20% or more carbon dioxide in 24 h.

Experiment 1

Carbon dioxide was provided from a road tanker (4 t) connected to a vapouriser capable of delivering up to 3 t h^{-1}. The vapouriser was set to produce a 60% carbon dioxide in air mixture. This mixture was heated to 30^{o}C (range 29.0 - 20.5^{o}C) in the vapouriser and passed through a flexible steel hose 75mm (i.d.) coupled to a pipe at the base of the silo. The gas then passed through an open gate valve, below which a copper pipe 10 mm (i.d.) carried methyl bromide into the gas stream. The methyl bromide was dispensed from a cylinder, placed on scales (minimum graduation 0.1 kg) to measure dosage, through a dispenser and a vapouriser connected to a semi-rigid nylon hose leading to the copper pipe at the base of the silo.

The carbon dioxide and air mixture was introduced into the silo for a period of 10 min. Methyl bromide was then gradually added to the gas stream. During the purge air escaped from the headspace via the gas return duct to the recirculation fan which was connected to the fan after completion of the purge. Purging was stopped and the bin sealed when the carbon dioxide level in the headspace reached 20%. By the time this was done the carbon dioxide level in the headspace had risen to 60%. The purge lasted 63 min. during which 1.71 t of carbon dioxide and 8 kg of methyl bromide were introduced into the silo.

On completion of the purge the manhole cover and the gate valve on the gas delivery pipe were closed, the gas return duct to the recirculation fan was connected to the fan and the valves on either side of the recirculation fan were opened and the fan was started. Gas was recirculated for 24 h during which a further 8 kg of methyl bromide was introduced (4 kg after 6 h and another 4 kg after 12 h) to enable the desired *Ct*-product to be achieved. After 24 h the fan was turned off, the manhole cover and the cap on the recirculation duct opened, the valve on the gas intake side of the fan was closed and the pipe disconnected from the fan. The fan was then restarted and used to ventilate the silo for 15 h after which the methyl bromide level was below 15 ppm and grain could be safely handled.

Experiment 2

Experiment 2 was carried out in a similar way, but methyl bromide was applied alone without CO_2, into the recirculation duct and distribution by the recirculation fan. The dosage applied was exactly the same and was applied at the same time intervals as in experiment 1.

Table 2.

Cumulative products (ghm^{-3}) for methyl bromide at different sampling locations within the silo.

Sampling Location	Cumulative *Ct*-Product for Methyl Bromide Experiment 1 with carbon dioxide	Experiment 2 with Methyl bromide alone
1	61.1	76.2
2	56.4	71.4
3	62.0	56.9
4	60.4	59.8
5	62.7	35.3
6	51.0	32.0
7	63.1	80.5
8	63.5	78.7
9	52.9	55.1
10	62.0	49.9
11	59.1	35.5
12	46.3	30.7
13	61.9	26.0
14	55.8	44.2
15	67.6	40.8.
16	57.1	34.8
17	54.1	34.6
18	38.2	36.4
19	47.1	37.8
20	25.0	3.5
Totals:	1107.3	900.1
Average:	55.4	45.0

RESULTS

Grain Condition

The grain moisture content, temperatures and bromide levels are given in Table 1. Grain temperatures of 35°C and over in the central region of the silo were probably due to an insect infestation in the wheat prior to fumigation. The bromide levels after experiment 1 were under two-thirds of those to be expected after a normal methyl bromide fumigation (Monro, 1969).

Gas Distribution

The carbon dioxide assisted greatly in achieving even distribution of methyl bromide in the grain, much better distribution being obtained than in the comparison trial, experiment 2, where the methyl bromide without CO_2 was distributed by the recirculation fan (Table 2). This was most noticable at location 13 (Fig. 1) at the base of the silo in between the arms of the Y-duct where slow gas distribution had been expected because of pressure equalisation created by the gas flow from each duct arm. Carbon dioxide levels were maintained at 40-60% at all locations throughout the recirculation period.

Bioassays

In experiment 1 insects in the spear cages at locations 5, 6, 11, 12, 17, 18, and 19 (see Fig. 1) were exposed to *Ct*-products of 38.2 - 62.7 g/h/m^{-3} of methyl bromide and all were killed. Gas flow to the insect cages at the base of the silo (locations 1, 7 and 13) was restricted, especially in cage 13 where the pipe carrying the cage was exposed to the sun and air pressure within the pipe would have further restricted ingress of methyl bromide and carbon dioxide. In this cage 96% of adults and 57% of larvae survived but in the other cages all insects were killed.

In the pipe cages under the grain surface where the *Ct*-product could have been as low as 25 g/h/m^{-3} all insects were killed, excepting 2 mature larvae or pupae of *S. oryzae* and 165 mature larvae and pupae of *S. granarius* which were reared to the adult stage by incubation of the grain.

At the end of experiment 1 a 1 kg grain sample from the surface was examined for insects and all were found to be dead.

In experiment 2 most caged insects at all locations survived.

DISCUSSION

This study has demonstrated that methyl bromide and carbon dioxide gas mixtures under recirculation can provide effective insect control with a *Ct*-product of 50 g/h/m^{-3} of methyl bromide in an atmosphere of 40-60% carbon dioxide. This *Ct*-product is one third of the minimum recommended for use of methyl bromide alone. It was ineffective when applied without carbon dioxide under the trial conditions.

Carbon dioxide was shown to be an efficient carrier for the methyl bromide and ensured good gas distribution during the purge. Distribution of methyl bromide alone by the gas recirculation fan was not as good. The gas distribution obtained by the purge may best be maintained by recirculating gas from the base of the silo to the headspace with a small fan in the manner used for carbon dioxide treatments (Wilson *et al.*, 1980). A further trial using this recirculation system is planned.

The low methyl bromide concentrations observed in the headspace are believed to have resulted from a combination of sorption of methyl bromide during its passage through the grain, gas leakage around the recirculation duct at the top of the silo and the recirculation fan sucking air into the silo through the leaks. Thus the trial indicated the importance of effective silo sealing for fumigations. The problem of low concentrations of methyl bromide in the headspace should be overcome by sealing the leaks at the top of the silo, by increasing the methyl bromide input to about 20–25 kg, and by reducing the recirculation rate and recirculating gas from the base of the silo into the headspace. Bromide residue levels would still be expected to be about two-thirds of those to be expected from a normal methyl bromide fumigation.

Costs of the methyl bromide and carbon dioxide treatment were estimated to be similar to those incurred for carbon dioxide alone (about 30 ¢ t^{-1} for gas and labour). Advantages of using the methyl bromide and carbon dioxide gas mixtures are the short treatment time compared with that required for carbon dioxide alone and the reduced bromide residues on grain compared with those for methyl bromide alone.

ACKNOWLEDGEMENTS

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EFFECTS OF OXYGEN ON THE TOXICITY OF CARBON DIOXIDE TO STORAGE INSECTS

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ABSTRACT

Atmosphere of 60-80% carbon dioxide (CO_2) in air often prove more lethal to insects than CO_2 alone. The toxicity of CO_2 can thus be enhanced by the presence of oxygen (O_2). The efficacies of gas mixtures containing up to 80% CO_2 and 20% O_2 with any balance as nitrogen, of similar CO_2 mixtures in air, and of CO_2 alone were compared. Eggs of *Ephestia cautella* and *E. kuehniella* aged 2, 20 and 44h were obtained by regulating oviposition in light/dark cycles and were tested at 15 and 25°C. At both temperatures, 2-h-old eggs were killed within 18 h by any mixture containing over 40% CO_2. Eggs aged 20 h or more were killed by any mixture containing over 30% CO_2 within 48 h at 25°C but required 5-7 days for control in mixtures containing 40% or more CO_2 at 15°C.

Increasing O_2 from 4 to 20% had little effect on the lethality of 80% CO_2 to eggs at 15 or 25°C, but an increase to 100% CO_2 shifted the most tolerant age group from 44 to 20 h. Implications for practical control are discussed.

INTRODUCTION

The effects of modified or controlled atmospheres on pests of stored products have received much attention in recent years, but in some areas there is a lack of precise information because of difficulties in comparing data gathered in different ways by different authors regarding the influence of temperature, and the relative susceptibility of different species and life stages.

Early work by Bailey (1956; 1957;1965) included an examination of carbon dioxide (CO_2) concentrations of up to 60% in the presence of 21% oxygen (O_2). Working mostly with 14-day exposures at 29-32°C, he found that all insects died in O_2 concentrations less than 3-4%, or with 40-60% CO_2 (depending on the species) in the presence of 21% O_2 (Bailey, 1956; 1965). Subsequently, other workers concentrated largely upon the effect of atmospheres obtained by replacing air with CO_2, mostly at high temperatures. Hence Press and Harein (1967) describe the effects of purging peanut storage towers with nitrogen (N_2) or CO_2 to control *Tribolium castaneum* (Herbst), Pearman and Jay (1970) described the effects of a 46% CO_2 atmosphere in air at various humidities, and Marzke *et al.*, (1970) described the effects of various mixtures of O_2, N_2 and CO_2 (modelled on the replacement of air with either N_2 or CO_2) on three insect pests at various temperatures. Apart from the last mentioned paper, work on the effects of cool temperatures on the performance of atmospheric gas mixtures is scarce. Harein and Press (1968) working with *T. castaneum* and *Plodia interpunctella* (Hubner) included tests at 15.6°C in their examination of the effect of low O_2 concentrations in the presence of 36% or more CO_2, and Jay (1980) tested immature *Sitophilus oryzae* (L.) at 1.6, 4.7, 10.4 and 15.7°C in 60 and 98% CO_2 in air.

In looking at the replacement of air with CO_2 (or N_2) much emphasis has been placed on the effects of low O_2 concentrations. It is widely known, however that atmospheres of 60-80% CO_2 in air can be more lethal to some insects than near 100% CO_2 (Lindgren and Vincent, 1970; Jay and Cuff, 1981). Recently it has been shown that the toxic effects of low O_2 concentrations are much increased by the presence of relatively low levels (10-35%) of CO_2 (Calderon and Navarro, 1979; 1980; Navarro and Calderon, 1980). Similar results have been obtained in atmospheres generated by burning gas in air (Storey, 1977; 1980).

The effect of O_2 at concentrations up to 21% on the toxicity of CO_2 atmospheres has been looked at infrequently since the early work by Bailey over 20 years ago. AliNiazee (1971) tested 80/20% and 90/10% mixtures of CO_2 and O_2 at 26.7°C and found the latter mixture to be as lethal to *Tribolium confusum* J. duV. adults as a 98/2% mixture, and Lindgren and Vincent (1970) found the toxicity of an 80/20% mixture of CO_2 and O_2 to be comparable with that of 80% CO_2 in air against *Sitophilus oryzae* and *Sitophilus granarius* (L.) adults, again at 26.7°C. Both mixtures were much more toxic than 100% CO_2.

Bailey and Banks (1980) expressed the view that in contrast to low O_2 concentrations, there may be little temperature dependency for high CO_2 concentrations in the presence of O_2. The present contribution presents the early results of a programme studying the effects of O_2 on the toxicity of mainly high concentrations of CO_2 to storage pests at 15 and 25°C.

MATERIALS AND METHODS

Experiments were performed on eggs of *Ephestia cautella* (Walker) and *Ephestia kuehniella* Zeller. Eggs were obtained by utilizing the ovipositional response in light/dark cycles whereby most eggs are laid in the first part of the dark period (Bell, 1981). It proved possible to obtain eggs aged to within one hour in sufficient numbers for experiments by collecting the yield after the first hour of darkness. Cultures were conditioned in a 15-h light, 9-h dark cycle at 25°C, 60% r.h. with the dark phase commencing at the hour chosen for the subsequent isolation of eggs for several days before setting up adults. During a photoperiod, between 100 and 150 adults were anaesthetised briefly with CO_2 and dropped into a nylon sieve. A PTFE-coated glass dish with a cotton wool drinking pad taped inside was then taped over the sieve to confine the adults. Eggs laid through the sieve were collected in a dish which was emptied immediately prior to the start of a dark period. After remaining in the dark for one hour the eggs obtained were counted on to watch glasses in lots of 100. Neither damaged eggs nor clumps were used. Glasses bearing eggs were then held at 25°C until reaching the age chosen for treatment. Eggs aged 2, 20 or 44 h were treated at 15 and 25°C. For treatment, each watch glass bearing eggs was placed on a platform inside a 15 cm diameter perspex dish at the experimental temperature. Each dish was fitted with inlet and outlet apertures for the flow of gas from a gas blending apparatus, and was sealed using vacuum grease by a perspex lid. Spratt (1979) described the apparatus used to provide a continuous, regulated flow of a mixture of gases. Before entering the sealed chamber, the mixture of CO_2 and O_2 or air was passed over a saturated solution of sodium and potassium chloride to bring it to about 70% r.h. After flushing for five minutes, the flow rate was adjusted to 50 ml/min. Eggs were exposed for up to 3 days at 25°C and 7 days at 15°C. Details of the measurement of gas concentrations using a Taylor Servomex oxygen analyser are given by Bell *et al.* (1980). Control eggs were exposed to turbulent air at 15 or 25°C and 70% r.h. in the constant temperature room containing the gas flow apparatus.

After the allotted period of exposure, the chamber was opened and the watch glasses were placed on petri dishes which contained food. After a few minutes in the exposure room these were returned to the rearing room at 25°C, 60% r.h. Starting three or four days later, the number of unhatched eggs was counted each day until no further hatch occurred. In some experiments the petri dishes were retained to observe development of larvae to adults.

RESULTS

Eggs of both species were quite susceptible to exposure to CO_2 in the presence or absence of O_2. At 25°C, gas mixtures containing 40% or more CO_2 did not permit hatch of any age group of eggs after 48-h exposures. An 18-hr exposure to 60% CO_2, 20% O_2 and 20% N_2 killed all 2-h eggs of both species. The survivals observed after 24-h exposures are shown in Table 1 and hatch was delayed by about one day after these exposures. Eggs of *E. cautella* aged 2 h at the start of exposure were more susceptible to all mixtures tested than 20 or 44h old. Eggs aged 20h were the most tolerant when exposed to pure CO_2, or, in the case of *E. cautella*, to mixtures containing 30% or less CO_2 in the presence of 20% O_2. With 40-80% CO_2 in the presence of O_2, 44-h-old eggs of both species became the most tolerant age group. A few tests with CO_2 in the presence of O_2 on 68-h eggs revealed a fall in tolerance by this age in both species.

Table 1. Survival (%) of eggs of *Ephestia cautella* (E.c.) and *E. kuehniella* (E.k.) exposed to gas mixtures for 24 h at 25°C.

Species	Age of eggs (h)	% Survival When Exposed To A Mixture Of - ($CO_2/O_2/N_2$)						
		20/60/20	30/50/20	40/40/20	60/20/20	60/32/8	80/0/20	100/0/0
E.c.	2	1	6	0	0	0	0	4
	20	98	87	0	9	0	37	78
	44	97	52	*	67	64	64	42
E.k.	20	*	*	60	1	33	31	98
	44	*	*	*	63	*	42	1

* Not tested

At 15°C, a shift was again seen in the age group most tolerant to CO_2 in the presence or absence of oxygen. Eggs of both species aged 2h were susceptible and none survived 18-h exposures to any mixture containing 40% or more CO_2. Table 2 gives the number of days required for less than 10% survival of eggs of *E. cautella* and *E. kuehniella* aged 20 or 44h. In general about a day longer was required for complete control and no eggs survived exposure for 7 days. When hatch was less than 10%, very few larvae developed to the adult stage, and with less than 5% hatch all larvae died before moulting. The most tolerant age group in pure CO_2 was 20h, and in CO_2 with O_2, 44h. At 15°C, eggs of *E. kuehniella* were as or more susceptible than those of *E. cautella*.

Table 2. Days for less than 10% survival of eggs of *Ephestia cautella* (E.c.) and *E. kuehniella* (E.k.) at 15°C.

Species	Age of eggs (h)	Days For Less Than 10% Survival When Exposed To Indicated ($CO_2/O_2/N_2$)				
		40/48/12	80/0/20	80/10/10	80/16/4	100/0/0
E.c.	20	5	4	4	4	5
	44	6	6	6	6	3
E.k.	20	5	*	*	*	4
	44	6	5	6	5	3

* Not tested

DISCUSSION

Eggs of *Ephestia* moths are easy to obtain in large numbers within an hour and provide a useful tool for physiological studies. As development proceeds, several changes occur in the response to CO_2. In the first hours after oviposition, eggs are highly susceptible to concentrations of 40% CO_2 or above in the presence or absence of O_2. A similar phase of high susceptibility is seen in eggs held in N_2 (Bell *et al.*, 1980). Eggs of Pyralid moths are fertilized as they descend the oviducts immediately prior to oviposition but it is unlikely that nuclear fusion occurs until several hours afterwards The sensitivity of the fertilizing sperm cell to anoxia or metabolic disruption caused by CO_2 may very well explain the extreme sensitivity of very young eggs to treatment. As embryogenesis proceeds, the susceptibility to CO_2 decreases at first (Bell *et al.*, 1980) and it continues to do so up to age 44h, in the presence of O_2 concentrations of 4-20%. However in the absence of O_2 it starts to fall again between 20 and 44h after oviposition. The results for *E. cautella* indicate that the presence of O_2 may in some circumstances enhance the toxic action of CO_2. During anoxia the development of the eggs virtually ceases (Price and Bell, 1981) and survival depends on the capacity of the embryo to accumulate glycolytic products and reduce its need for active metabolism. In the presence of O_2, however, although development may be delayed, there is no evidence that it ceases completely and death probably results from progressive CO_2 poisoning which either hampers the utilization of O_2 by inhibiting enzymes such as succinic dehydrogenase (Edwards, 1968) or causes oxidative metabolism to accumulate toxic products. The desiccating action of CO_2 at lower humidities can be disregarded for eggs because spiracles are absent. If development continues during exposure and different stages vary in tolerance, individuals will tend to develop through phases of tolerance and die when a susceptible stage is reached. A similar effect has been observed with the fumigant phosphine (Reynolds *et al.*, 1967).

A slightly longer exposure was required to kill all eggs with CO_2 in the presence of oxygen than in its absence when tests were conducted at 15°C, but the age group surviving best was one quite easily killed by anoxia. The age group most tolerant to anoxia was killed more quickly by CO_2/O_2 mixtures. This difference in the stages of development best fitted for survival necessitates further investigation of CO_2/O_2 mixtures. It is known that *Sitophilus* spp. are more tolerant to CO_2 exposure than most other pests and that 80% CO_2 in air or O_2 is much more toxic to these species at high temperatures than CO_2 alone (Lindgren and Vincent, 1970). In recent unpublished tests on *S. granarius* at Slough conducted by E. C. Spratt, an 80/20% mixture of CO_2 and O_2 killed adults faster at 15°C than 80% CO_2 in air, and very much faster than CO_2 alone. The period of exposure necessary for control of this species at 15°C by CO_2 in the presence of O_2 was in fact not so very much longer than at 25°C.

The time for action of CO_2 in the presence of air or oxygen at high temperatures in many species is little different than that for CO_2 alone, but results such as those described for *Sitophilus* spp. indicate that at low temperatures the difference may be more marked. If this is so then purging storage structures with CO_2/O_2 mixtures may give better results at low temperatures than purging with CO_2 alone. However, much work needs to be done on a wider range of species and stages. Up to this time much emphasis has been placed on adults and when compared with other life stages they tend to be fairly tolerant to anoxia. In the presence of O_2, however, the spectrum of tolerance to CO_2 among species and stages may be different than in the absence of oxygen, as indicated by the shift of the least susceptible age in the current tests on eggs.

Carbon dioxide, unlike nitrogen, does not rely solely on achieving anoxia to be lethal and 100% CO_2 is more toxic to moth eggs than 100% N_2 (Bell *et. al.*, 1980). In addition, CO_2 may be more toxic when O_2 is present although this aspect of increased toxicity requires further clarification. Hitherto, CO_2 has been used in a manner that more or less replaces air and hence O_2, although it has been recognised that CO_2 remains effective far longer than N_2 as air leaks back into the purged structure. The feasibility of purging structures with a mixture of ca. 10-20% O_2 in CO_2 will depend on the advantage gained in the initial stages of the treatment. This advantage may be small or localised when only 60-80% of the total air is replaced with pure CO_2. As the effects of anoxia in conjunction with CO_2 tend to disappear when oxygen levels rise above 4% (Calderon and Navarro, 1980), this period of potential advantage extends only until the CO_2 concentration drops to 80% at all points as the purged volume is replaced by air leaking back into the storage.

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RESPONSE OF SEVERAL SPECIES OF INSECTS TO MIXTURES OF PHOSPHINE AND CARBON DIOXIDE

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ABSTRACT

Exposure of several species of insects to low levels of phosphine (50 or 200 mL/L) in CO_2-air mixtures (25 or 75% CO_2) enables a quicker kill than does exposure to either CO_2-air mixtures or to phosphine in air. These species are *Tribolium castaneum*, *Tribolium confusum*, *Rhizopertha dominica* and *Trogoderma granarium*. This result is sometimes due to synergism - i.e. CO_2 potentiates the action of phosphine - and sometimes due to the relative order of susceptibilities of different insect stages to one component of the mixture. Under the conditions studied mixtures of phosphine and CO_2-air were not more effective at the LT_{99} level than phosphine alone in controlling pupae and eggs of *Sitophilus granarius* and *Sitophilus oryzae*.

INTRODUCTION

The temperate middle-European climate, while enabling insect control by aeration for much of the year, results in summer conditions that are too sultry for the use of aeration but cool enough (15-25°C) to require high concentration by time products for inert atmospheres or phosphine. It was in view of this background that we investigated the effects of mixtures of phosphine, carbon dioxide and air mixtures against adults, pupae, larvae and eggs of *Sitophilus granarius* (L.), *Sitophilus oryzae* (L.), *Tribolium castaneum* (Herbst), *Tribolium confusum* (duVal), *Rhizopertha dominica* (F.) and *Trogoderma granarium* (Everts) at the low temperature of 19°C and at 70% relative humidity. Insects were exposed to mixtures of gases for defined periods and then held at 25°C, 70% rh for end point mortality, in the case of adults, 3 weeks mortality, in the case of external larvae, complete transformation to the next stage, in the case of external eggs or pupae and complete emergence to adults in the case of internal stages of *Sitophilus* species and of *R. dominica*. A fuller account of the experimental procedure is given by Desmarchelier (1984).

RESULTS

The stages most resistant to 75% CO_2, 25% air were pupae of *Sitophilus* species, and *T. granarium* and larvae of *T. granarium*. Adults and pupae, but not eggs and larvae, of *S. granarius* were significantly more tolerant to CO_2 than corresponding stages of *S. oryzae*.

Table 1 - Time to mortality (LT_{50}) for stages of 6 species exposed to 75% CO_2, 25% air at 19°C, 70% rh.

Species	LT_{50} (h)			
	Adults	Pupae	Larvae	Eggs
Sitophilus granarius	63	207	68	56
Sitophilus oryzae	15	63	48	73
Tribolium castaneum	39	99	51	28
Tribolium confusum	45	75	47	15
Rhizopertha dominica	27	50	31	94
Trogoderma granarium	40	238	312	115

In studies on the effect of 25% CO_2, 75% air on the 4 stages of each of the six species studied, mortality after 7 days exposure was generally low, but 100% in the cases of eggs of *T. castaneum* and *T. confusum* and adults of *S. oryzae* and between 50 and 99% for adults and larvae of *R. dominica*.

Selected data on the toxicity of mixtures of CO_2, air and phosphine to *S. oryzae* and *S. granarium* is shown in Table 2. The data are similar for each species in that addition of CO_2 accelerates the speed of action of phosphine against adults and, to a lesser extent, against larvae, but does not accelerate the speed of action of phosphine against the stages most tolerant to phosphine, namely eggs and pupae.

Table 2 - Time to 50% mortality (LT_{50}) for stages of *S. oryzae* and *S. granarius* exposed to mixtures of carbon dioxide, air and phosphine at 19°C, 70% rh.

CO_2 in air (%, V/V)	PH_3 (mL/L)	Species	LT_{50} (h)			
			Adults	Pupae	Larvae	Eggs
75	0	S. oryzae	15	63	48	73
0	200	"	3.5	12	11 *	44
25	200	"	0.51	12	9 *	44
75	200	"	0.47	10	7 *	44
75	0	S. granarius	68	207	8 *	56
0	200	"	4.8	32	7.1 *	21
25	200	"	0.24	36	4.8 *	19
75	200	"	0.75	32	3.7	22

*Exposed to 50 mL/L phosphine

The position could, however, be different when one considers the addition of a small amount of phosphine, for example 50 mL/L, to a CO_2 fumigation. This amount is sufficient to kill the larvae - the LT_{99} values in 50 mL/L phosphine, 75% CO_2, 25% air are 34.3 and 14.4 h - respectively for larvae of *S. oryzae* and *S. granarius*. This amount of phosphine could be important in preventing control failures associated with e.g., the development of larvae into pupae during the experiment, or isolated cold spots where CO_2 is incompletely effective. The numerical preponderance of larvae vis-a-vis pupae or eggs merits consideration in this context.

Similarly use of 50 mL/L phosphine will control larvae of *R. dominica* (Table 3). Carbon dioxide accelerates the action of phosphine against larvae and adult stages.

Table 3 - Time to 50% mortality (LT_{50}) for stages of *Rhizopertha dominica* exposed to mixtures of carbon dioxide, air and phosphine at 19°C, 70% rh.

CO_2 in air (%, V/V)	PH_3 (mL/L)	LT_{50} (h) for:			
		Adults	Pupae	Larvae	Eggs
75	0	27	50	3.4	94
0	200	3.7	7.6	8.6*	8.3
25	200	1.7	3.3	5.6*	7.9
75	200	1.5	4.2	4.7*	8.1

*PH_3 concentrations were 50 mL/L for larvae

For *Trogoderma granarium* the LT_{99} values are presented graphically (Figure 1). Here the time to 99% kill in a mixture of 50 mL/L phosphine and 75% CO_2 is only approximately half the time required in CO_2 alone (cf. Table 1) or in phosphine alone. Under the conditions studied, carbon dioxide does not alter the sensitivity of phosphine to pupae but the time to 99% mortality of the phosphine-tolerant larvae decreases rapidly with increasing CO_2 content.

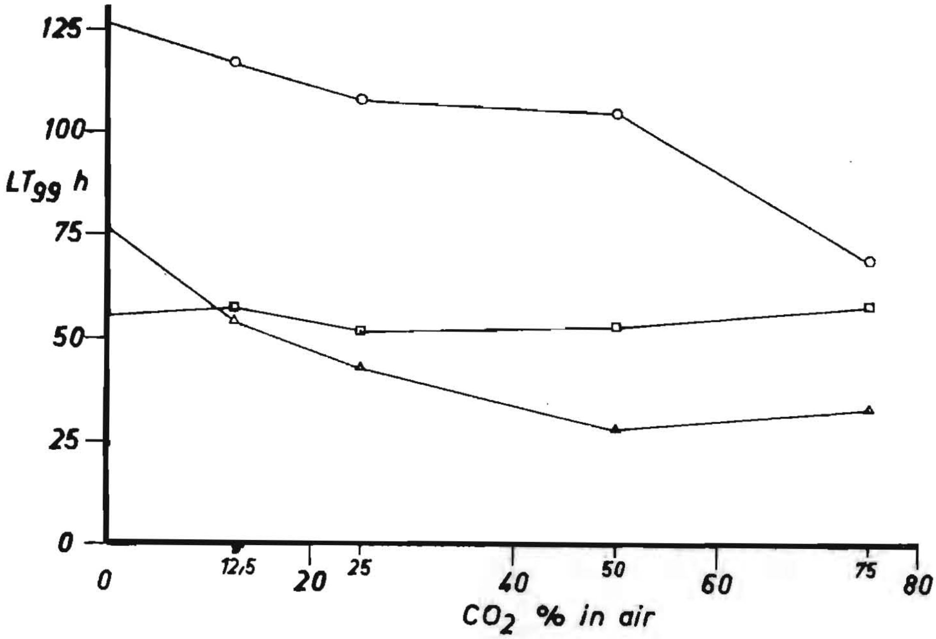

Figure 1 - Time to 99% mortality (LT_{99}) for adults (o), pupae (□) and larvae (△) of *Trogoderma granarium* exposed to 50 mL/L phosphine in CO_2-air mixtures, at 19°C, 70% rh.

Mixtures of carbon dioxide and phosphine result in a rapid kill of *Tribolium* species, due to synergistic effects between the two against larvae and adults, and to the sensitivity of pupae to phosphine, which is maintained in CO_2, and to the sensitivity of eggs to CO_2. Results for *T. castaneum* are given in Figure 2, together with results for adults of *T. confusum*. For this species, time to mortality decreases with CO_2 concentration over the range 0-75% CO_2, whereas for adults of *T. castaneum* the speed of kill is dependent on CO_2 concentration in the range 0-25%. Time to mortality for larvae of *T. castaneum* is also dependent on CO_2 concentration over the range tested, whereas CO_2 does not effect the toxicity of phosphine to pupae.

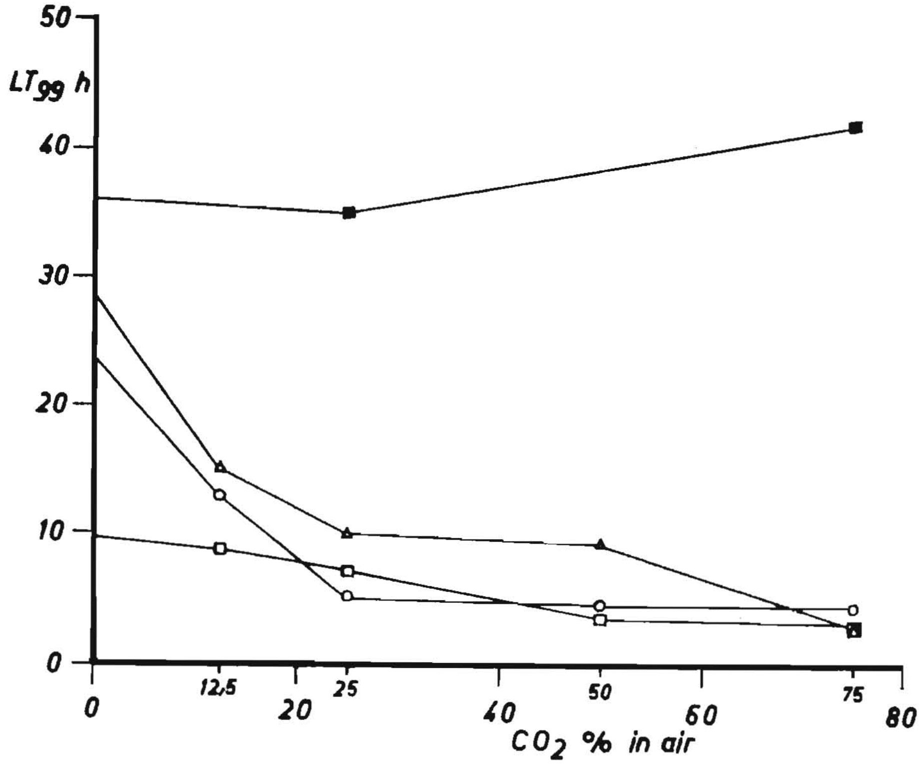

Figure 2 - Time to 99% mortality (LT_{99}) for adults of *Tribolium confusum* (▲) and adults (o), pupae (■) and larvae (□) of *T. castaneum* exposed to 50 mL/L phosphine in CO_2-air mixtures at 19°C, 70% rh (Results for *T. castaneum* adults were calculated as four times the measured value at 200 mL/L phosphine).

DISCUSSION

For *Tribolium* species, a mixture of 25% carbon dioxide and 50 mL/L phosphine offers a clear economic alternative to either high CO_2, or high phosphine, concentrations. A similar comment applies to mixtures of high CO_2 and low phosphine concentrations for the control of *T. granarium* and *R. dominica*. If similar values are confirmed for other species, especially moth species, the use of low phosphine, CO_2 air mixtures could find a use in disinfesting commodities where *Sitophilus* is not a problem. Such products include commodities with high oil content, such as cocoa and nut products, where high levels of phosphine can cause residue levels in excess of 0.1 mg/kg.

The use of low levels of phosphine to kill internal larvae in a CO_2 fumigation has been discussed. It should here be pointed out that the LT_{99}, but not the LT_{50}, of adults of *S. granarius* in 75% carbon dioxide, 25%

air, or in other gas mixtures containing 5% oxygen, is greater than the LT_{99} in air. However at 200 mL/L phosphine this protective effect of low O_2 levels is not observed.

In summary, there would seem to be a niche in stored products for mixtures of phosphine and carbon dioxide.

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SESSION 3.

PRESERVATION OF QUALITY IN CONTROLLED ATMOSPHERE STORAGE

Papers by:

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PILOT SCALE EXPERIMENTS ON HALF-WET MAIZE STORAGE UNDER AIRTIGHT CONDITIONS: MICROBIOLOGICAL AND TECHNOLOGICAL ASPECTS

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ABSTRACT

Fungi of cereal grains are generally recognized to be strictly aerobic. Several recent publications indicate that these fungi can grow to some extent even under very low oxygen tensions. In laboratory scale experiments, we have shown that it is possible to store grains at 21% moisture content in hermetic structures (w/w) for several months without appreciable changes in microflora. Accordingly, a pilot-scale experiment was carried out in a hermetic mini-silo of about 1 m^3 of capacity, at the same moisture content. Analytical results show that in such conditions, a natural anaerobiosis is obtained within 4-5 days (O_2 0.5%) after a very slight alcoholic fermentation. Subsequently, during a six months period of storage the technological and nutritional qualities of the grain remain unchanged. During the next six months the lactic bacteria and the yeast show weak, but noticeable, metabolic activity and growth. In spite of this unexpected microbiological growth the qualitites of the grain were still acceptable after one year of storage. Microbiological analysis show that mould counts were very low at the end of the experiment, no mycelial growth occured (checked by determination of ergosterol in grains) and no mycotoxins were produced. This grain storage technique seems therefore of interest because of the increasing cost of drying. A new experiment is now being conducted on a larger scale. Same preliminary results are presented.

INTRODUCTION

During the last 10 years, the cost of maize drying, harvested in France with a moisture content between 35 and 45% wb, has increased very much. In order to minimize that cost, several processes have been developed. At first energy-saving drying technologies have been evolved: dryeration, high temperature dryers, two-step dryers (MULTON and LASSERAN, 1979). But, generally speaking this tendancy has been to increase the temperature of incoming air. This has led to a decrease of the quality of maize, due to the thermal shock; a problem of particular concern in the case of maize used for starch production. As alternatives two wet storage techniques have been developed: (i) the "silage", which is mainly lactic fermentation, exclusively used for cattle feed, and (ii) the "cribs", a natural and slow drying process. Unfortunately, the slowness allows moulds to grow and produce toxins, when the climate is not appropriate. Wet storage using propionic acid is too expensive and irradiation is not satisfactory.

Noting all these disadvantages, we have studied in our laboratory a method which consists in partially drying the maize to 20–22% mc and storing this 'half wet' maize in air tight silos. It appears that between one third and one half of the cost of drying may be saved, and the thermal degradation avoided.

A previous study conducted in our laboratory using a small quantity of grain (1kg) (Richard-Molard *et al.*, 1980) has shown that in such conditions of humidity and airtightness, the oxygen of air trapped inside the sealed box is quickly converted to CO_2 by the respiration of moulds and the grain itself. Generally speaking the higher the moisture content and the temperature, the quicker is this phenomenon. When the partial pressure of O_2 has become very low (0.5%) respiration is stopped and moulds decrease and disappear progressively, killed by anaerobiosis. According to the literature, at that moisture content (22%), the water activity is too low to allow development of bacteria: only some yeasts can grow, but very slowly. Therefore all living processes are stopped and good preservation of grains is guaranteed.

Then the question was to check if the same effects could be observed with grain in bulk, and what are the changes in technological, nutritonal and microbiological qualities under these conditions. Two pilot-scale trials described here, were carried out to observe these changes. If these experiments were successful it appeared to us that the technique had potential for large scale application to the storage of partially dried grains destined for industries where the first step of the process is to temper the grain and which require grain without thermal degradation. These are mainly the starch industry and the cattle feed industry, making pellets and granules.

PILOT EXPERIMENT IN SMALL METAL SILOS (1 m^3)

Experimental procedure

Five 1 m^3 metal sealed bins, specially built for these experiments were completely filled with maize (one with dry maize, as reference, and four with half-wet grain).

The openings on the top for filling and at the bottom for emptying, were closed by screwed metal cover plates, the airtightness of which was ensured by a rubber gasket.

Three small tubes, closed by a valve, and connected to a device measuring the concentration of O_2, were fitted at the top, the bottom and centre of each bin. Three temperature probes were also arranged along the axis of the cell. These silos were placed inside the pilot plant area of the Research Centre, so that the temperature variations of external air were largely reduced and no noticeable thermal gradient could occur.

Maize

The maize used in this trial (unknown variety) was grown in Nantes area, (Western coast of France, Atlantic climate). After harvesting at about 40% moisture content, it was gently dried at a moderate temperature (80°C) to 21.5% moisture content in a discontinuous farm dryer.

METHODS

Moisture determination

Standardized method AFNOR NF-V-03-708, identical to ISO-6540, ICC 110-1 and OIML (No. 8) has been used (Multon, 1982).

Microbiological determination

Under anaerobic conditions, moulds which are supposed to grow or to metabolise, cannot sporulate (Tabak and Cooke, 1968). So counting conidia by the classical suspension-dilution method cannot be used to demonstrate a possible mould activity. This method has nontheless been used as a control for anaerobiosis. To some extent, the "Ulster" method (Musket and Malone, 1940) takes into account mycelial growth but is not really quantitative. For this reason the mycological analysis has been complimented with a gas-chromatographic determination of specific volatile compounds which indicate a fungal activity and with the high pressure liquid chromatography analysis of fungal ergosterol to show mycelial growth.

Enumeration of bacteria

The classical microbiological methods were employed for enumeration of the following bacteria groups: general mesophilic bacteria (PCA), Enterobacteriaceae (VRBG), Lactobacilli (Rogosa Agar), Anaerobic (TGY* Agar) and especially sporulated Clostridia.

Yeasts and moulds

The suspension-dilution method was used for yeast counts (Cahagnier, 1973). For the Ulster method (malt extract agar), 400 grains were analysed each time, 200 grains being superficially disinfected, 200 grains being not. Disinfected grains lead to aberrant results, probably due to increasing permeability of stored kernels (Pelhate and Theriault, 1979). For this reason those results are not considered here.

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* TGY : Trypticose, Glucose, Yeast extract agar.

Collection and analysis of volatile organic compounds

The volatiles were desorbed under vacuum, collected in a dry-ice cooled trap and recovered with methylene chloride. The extract was then concentrated by solvent evaporation and analysed by gas-chromatography, using a glass capillary column (50m length, 0.5mm id) coated with carbowax 20m. The oven temperature was programmed from 70°C to 150°C at a rate of 2°C/minute with helium as carrier gas (Richard-Molard *et al.*, 1976).

Fungal ergosterol assay

Fungal ergosterol was extracted with methanol from grounded kernels, saponified with KOH, extracted from methanol with petroleum ether, purified and analysed by HPLC using a 5u-Spherisorb Column (Seitz *et al.*, 1977) Cahagnier and Poisson, 1982).

Mycotoxins

The mycotoxins were determined with the multidetection method of Stoloff (1971).

O_2 concentration

O_2 concentration in intergranular atmosphere of the silo was determined directly with a paramagnetic O_2 meter (La Gazometrie **) working on a sample of air, which was pumped out and reinjected into the silo.

DETERMINATION OF TECHNOLOGICAL QUALITY

Grain fitness for industrial starch extraction. The industrial starch value of maize was estimated by extraction in a laboratory pilot plant, according to method described by Beaux and Le Bras (1982). The efficiency of extraction and quality of extracted starch was estimated through 13 different tests.

Nutritional quality

The nutritional value was estimated, as a first aproach, with growth trials, carried out on pullets. Two homogeneous groups of 24 pullets were fed for 4 weeks with a feed made of 22% soybean, 6% mineral and vitamins and 72% maize (experimental stored maize for one group, and the same maize but dried immediately after harvest, for the other group). Nutritional value was assessed by the quantities ingested and by the increase in weight of the pullets.

** La Gazometrie, 33 rue Antoine Marie Colin, 94400 Vitry-sur-Seine, France.

Biochemical parameters

Very classical methods were used for biochemical determinations:

- for starch content: Ewers' method
- for protein content: Kjedhal method (N x 625)
- for lipid content and ash: standardized EEC method
- for fat acidity: standardized AFNOR method (extraction in 95% ethanol, neutralization by KOH/ethanol, with phenolphthalein as indicator; results in mg H_2SO_4/100g dry matter.)

RESULTS AND DISCUSSION

Changes in temperature, moisture content and dry matter. Since the experimental silos were situated inside the pilot plant, the influence of outside temperature variation was not significant, and the temperture of the grain (17°C) did not change during the storage period. Table 1 shows that the moisture content and dry matter (measured by weight of 1000 grains) did not change either.

Table 1 - Moisture content and dry matter as a function of time.

Storage time Days	0	34	93	150	280	326
Moisture content (% wb)[a]	21.28	21.20	21.70	21.73	21.67	21.80
Dry matter[b] grams	243.3	241.5	241.9	238.5	237.6	240.6

a average of 12 samples

b dry weight of 1000 grains

SPEED OF DECREASE OF O_2 CONCENTRATION

Theoretical calculation. It is possible to compute, theoretically, the consumption of O_2 and the time necessary for the concentration of O_2 to decrease to zero. The full silo, if internal volume ca 0.93m^3, contained 690 kg maize. As 40% of the apparent volume is occupied by intergranular atmosphere, the total volume of air existing inside the silo is, including a small head space, about 0.400 m^3. The total volume of pure oxygen is then:

$$0.4 \times \frac{21}{100} = 84 \text{ litres.}$$

There are

$$\frac{84}{22.4} = 3.75 \text{ moles}$$

of O_2 inside the silo (neglecting correction of temperature).

With respect to the stochiometric equation of starch oxidation (and assuming as an approximation, that maize is constituted only of starch):

$$C_6H_{12}O_6 + 6O_2 = 6CO_2 + 6H_2O + \text{energy}$$

one can say that for each mole of O_2 used, there is one mole of CO_2 produced. Therefore, at the end of the process, when all oxygen is used, there are 3.75 moles of CO_2 in the silo, ie a mass of:

$$3.75 \times 44 \simeq 165\text{g of } CO_2 \qquad (1)$$

According to Srour (1982) the rate of production of CO_2 (q) in bulk grain is given by the following equation:

$$q = k \exp(a\theta) \qquad (2)$$

where q is expressed in mg CO_2/100 g dry matter/day, θ is final temperature in ^{o}C, k is a constant, being a function of moisture content and species (for maize at 21.5%, $k \simeq 4$), and a is a constant, being a function of the type of grain (for cereals, $a = 0.1385$).

Under the conditions of our experiment (final $\theta \simeq 17^{o}C$), we compute that the speed of production of CO_2 resulting from respiration is given by

$$\begin{aligned} q = 4.\exp(0.1385 \times 17) &\simeq 42.13\text{mg } CO_2/100 \text{ g dry matter/day} \\ &= 421 \text{ g } CO_2/\text{tonne dry matter/day.} \end{aligned} \qquad (3)$$

The silo contained 0.69 tonnes of maize at 22% moisture content, ie 0.54 tonnes of dry matter. Therefore the total speed of production of CO_2 inside the silo was 227 g/day (3). From (1) and (3) it is easy to compute that all the oxygen might have been converted into CO_2 after about:

$$\frac{165}{227} = 0.77 \text{ day.}$$

EXPERIMENTAL DETERMINATION

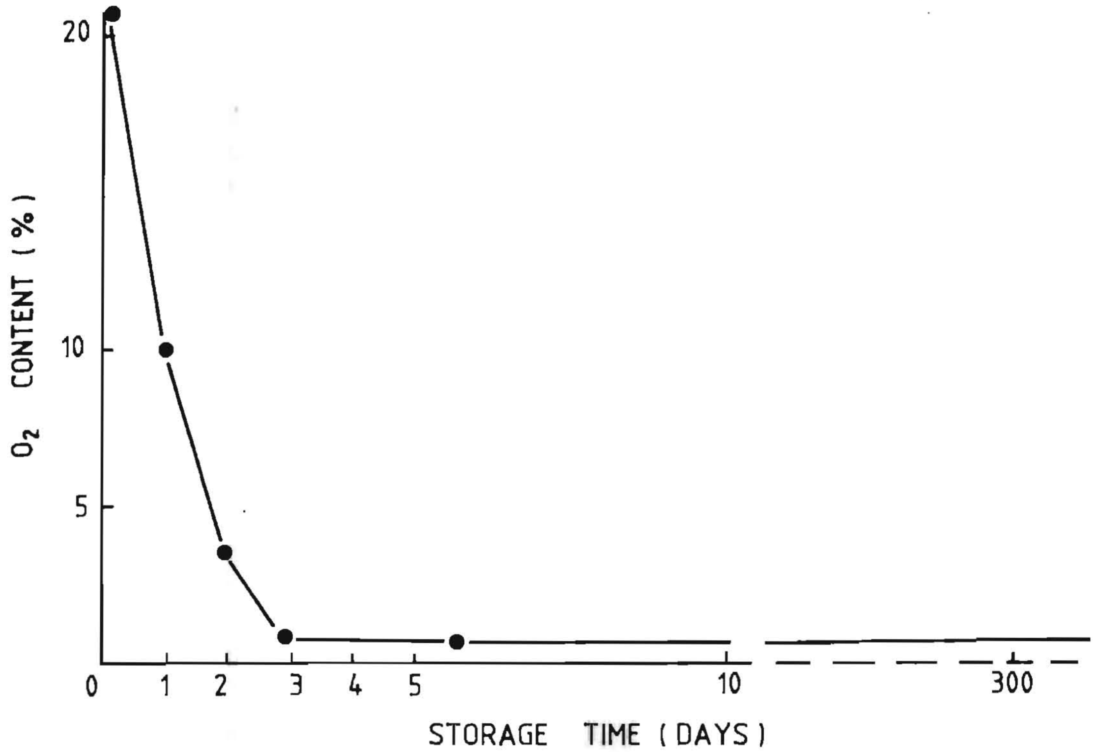

Figure 1 - Decrease of O_2 concentration inside the silo as a function of time

Figure 1 shows the decrease of O_2 concentration as a function of time of storage. It is clear that O_2 concentration is lower than 0.5% in about 3 days, 3 times longer than predicted by the theoretical calculation. This difference is certainly due to the fact that the equation (2) is representative of CO_2 production in aerated conditions. In confined conditions the metabolism and therefore the CO_2 production rate (or oxygen consumption) decreases progressive as the CO_2 increases.

This confirms the concept of the storage and shows the system is well sealed.

MICROBIOLOGICAL ACTIVITIES AND METABOLISM DURING STORAGE

Micro-organism growth

The results obtained are shown on Figure 2. We observed a continuous decrease of the number of mesophilic and aerobic, facultative anaerobic bacteria growing on TGY medium. In contrast there was pronounced development of *Lactobacillus* reaching a level of 3.3 x 10^4 germs/gramme (g/g) after 5 months, but decreasing thereafter.

The yeasts decreased from the beginning of the trial, but after 5 months, some species of *Candida* were growing.

Moulds, when estimated by simple counting, showed a very important, and rapid reduction of the total number, except for the *Mucorales*, which remained constant for about 1 month.

The 'Ulster method' gives a semiquantitive estimate of the mycelium growth of the different species. It was noted particularly that the fields species (*Epicoccum, Cladosporium, Verticillium, Cephalosporium*) disappeared very quickly. Fusarium remained a longer time on the grains, but disappeared completely before the end of the trial.

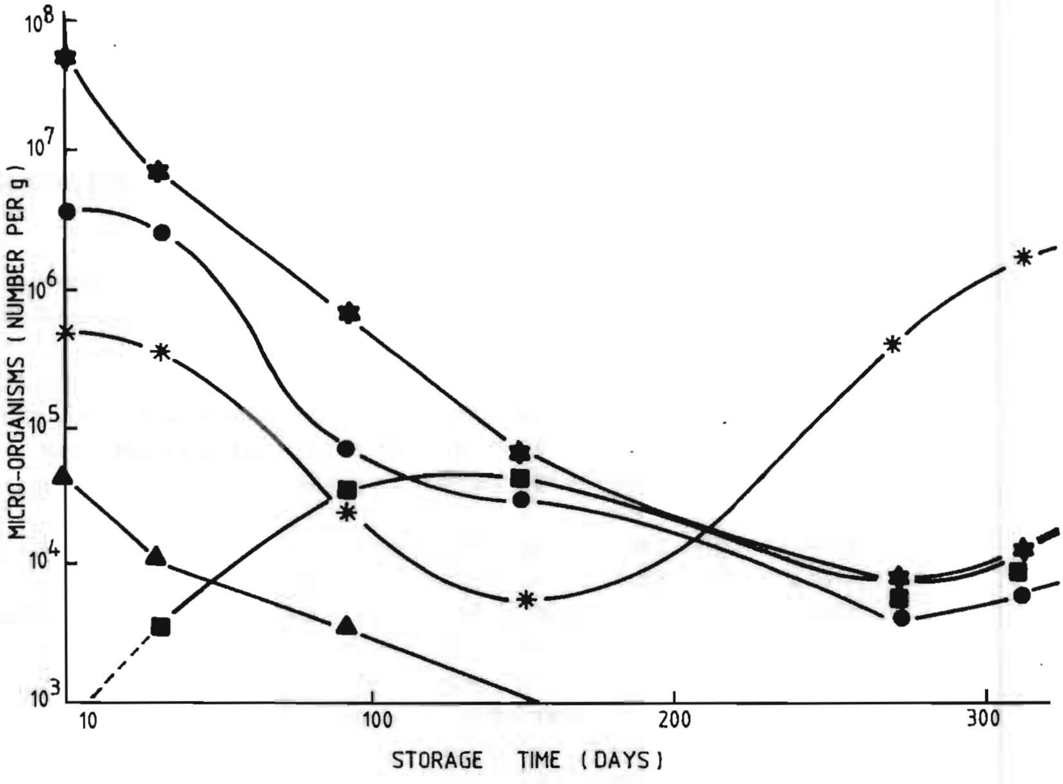

FIGURE 2 - MICROBIOLOGICAL EVOLUTION OF MAIZE, AS A FUNCTION OF STORAGE TIME.
- MESOPHILIC BACTERIA
- OPTIONAL ANAEROBIC BACTERIA
- LACTOBACILLUS
- YEAST
- MOLDS

Storage fungi, sparsely represented by some *Penicillium*, also disappeared very quickly, except again the *Mucorales*, characteristic of an intermediate flora well adapted to the lack of oxygen (Pelhate, 1978). The genus *Rhizopus* seemed particularly tolerant, but did not grow in the conditions of experiment. Anyway, at the end of the trial, when the silos were emptied, the grain was free of bacteria and moulds and only yeasts were present. The external appearance of this maize was excellent.

Ergosterol content

The biosynthesis of ergosterol requires oxygen (Weete, 1973). It is a good indicator of mould development (Cahagnier *et al.*, 1983). The average ergosterol content (four determinations) obtained from each silo was not significantly different (1.1, 1.09, 1.08, 1.07, 1.06 and 1.1 μg/g of grain).

The initial value is typical of a late harvested grain, and the constancy of this value during storage shows clearly that (i) there was no fungal evolution, and (ii) no entry of oxygen had occured during the trial. In fact, a small quantity of oxygen could have entered without causing a measurable increase of the concentration of oxygen in the silo since moulds used O_2 at the same speed as it comes in. However, in that case, ergosterol production would appear as a consequence of the mould activity.

Changes in volatile compounds of intergranular atmosphere

Only certain specific volatile compounds have been looked for. No presence of oct-1-ene-3-ol, an indicator of mould growth, was observed.

Some traces of isoamyl alcohol and B-phenyl-ethanol indicated a small alcoholic fermentation during the first days of storage, which stopped after some days.

Volatile compounds thus produced, remained in the grain and gave them a slight and characteristic smell of fermentation.

Mycotoxins

All the titrations of mycotoxins (aflatoxin B, ochratoxin A, sterigmatocystine, zearalenone) during and at the end of storage were negative.

CHANGES IN BIOCHEMICAL CHARACTERISTICS OF THE GRAIN

Table 2 shows the changes in the 7 biochemical characteristics which have been considered during this experimentation.

It can be seen that no important changes of these biochemical characteristics occurred during storage, except for the fat acidity, which increased slightly but continuously. This could be due to the lipase activity

Table 2 Changes in the main biochemical characterisitics as a function of time of storage.

	Storage Time (days)					
	0	34	93	150	280	326
Fat acitidy	0.025	0.031	0.037	0.062	0.069	0.079
Dry matter	84.8	84.5	85.1	84.2	83.3	83.6
Proteins % (d.b.)	9.36	9.38	9.60	9.64	9.50	9.30
Lipids % (d.b.)	4.62	4.65	4.52	4.89	4.68	4.87
Starch % (d.b.)	74.1	74.9	76.0	74.2	74.3	76.1
Cellulose % (d.b.)	2.5	-	3.8	3.3	2.5	2.7
Mineral Matter	1.10	1.30	1.20	1.20	1.35	1.25

Table 3 Changes in sugar fraction as a function of time (expressed in gramme/100 grammes of dry matter).

Time (days)	Sugars soluble in alcohol		Glucose	Fructose	Sucrose
	Orcinol	HPLC	H P L C		
0	1.25	1.30	0.08	0.06	1.00
93	0.51	0.56	0.13	0.15	0.18
150	0.52	0.53	0.20	0.23	0.10

It could be observed that sucrose is slowly hydrolysed into glucose and fructose during the experiment.

The lack of maltose and the ratio of glucose/fructose suggests that there was no noticeable amylolytic activity.

Table 4 Changes in starch value of maize as a function of time of storage (study made in starch pilot plant; results reported for a moisture content of 15%).

Test	Time of storage (in days)					
	0	34	93	150	280	326
Starch yield (% d.b.)	63.8	65.6	64.8	66.3	65.9	65.0
Starch recovery rate (%)	86.1	87.6	85.3	89.4	88.6	85.4
Protein content of starch (% d.b.)	.78	.63	.65	.58	.55	.61
Protein content of gluten (% d.b.)	27.0	28.9	27.4	27.8	27.2	26.7
Gluten yield	15.6	15.1	16.0	15.7	16.3	16.5
Protein recovery rate (%)	44.8	46.3	45.7	45.5	46.7	47.4
Filtration speed of gluten	12.0	13.1	12.2	11.7	11.9	11.6

TABLE 4 (continued)

Germ yield (% d.b.)	5.8	5.9	6.0	6.0	6.2	6.2
Fat content of germs (% d.b.)	56.2	56.3	55.2	56.1	55.0	54.6
Envelope yield	5.91	4.90	4.90	3.80	3.59	4.79
Turbidity	32.5	44.0	58.0	69.0	88.0	83.5
Sedimentation	58	77	72	67	93	78
Fitness to water tempering	39.8	39.9	40.3	40.4	39.8	40.5

of grains and micro-organisms, these enzymes being active in the abscence of O_2. More probably, such an increase is due to some organic acidity (eg. lactic acid, produced by microorganisms which was also extracted by 95% alcohol.

Nevertheless the fat acidity increase is rather small and does not indicate any significant alteration of the grain.

The changes in the sugar fraction (Table 3) are further evidence of a slight fermentation during the first month of storage, as suggested by microbiological data.

CHANGES IN TECHNOLOGICAL QUALITY

Starch value

The results of evaluation of starch values of the grain are given in Table 4.

When considering these results, the following observations could be made:

- the starch yield remained almost constant :
- grain stored for 326 days gave a starch as good as grain just harvested;
- the protein content of starch is similar to those obtained with a maize gently dried at 70-80°C;
- the fitness to water tempering, an important parameter to the starch processing industry, remains constant;
- the turbidity test showed a small deterioration of the protein : this change could be due to progressive acidification of the grains by organic acids (fermentation) leading to a partial insolubilization of proteins. Such a change has already been reported (Pelhate and Theriault, 1979 ; Carantino, 1979).

Nevertheless, generally speaking, the fitness of maize for industrial processing (as measured in a pilot extraction facility) remained very good throughout the storage period.

Nutritional value

The nutritional tests were made with selected pullets (homogeneous samples with respect to the weight, after four weeks ; during the first 12 days, the animals were fed *ad libitum* with a starting feed for chicks). The results of tests are reported in Table 5.

Table 5 Results of nutritional tests on pullets (R:for reference maize, S:for half-wet stored maize). Average values and standard deviation.

Storage Time (months)	5		11	
Diet	R	S	R	S
Dry matter ingested (g)	2,092.1 (± 39.4)	2,179.6 (± 31.6)	1,988.3 (± 40.0)	1,907.9 (± 44.9)
Weight increase	1,016.7 (± 21.2)	1,119.5 (± 17.1)	1,022.5 (± 28.4)	1,011.2 (± 26.2)
Nutritional efficiency (x)	0.496 (± 0.004	0.514 (± 0.003)	0.514 (± 0.001)	0.530 (± 0.047)

(x) Nutritional efficiency is the ratio of dry matter ingested over weight increase.

After 5 months of storage, the three tests showed better results for the hermetically stored grains than for the reference samples. The difference is highly significant. On the other hand, after 11 months, the results from the two first tests were slightly better for the reference maize, but the differences are very small and not significant for the weight increase and nutritional efficiency. Thus it appears clear that the hermetic storage of half-wet maize improves the nutritional value of maize during the first 5 months. After one year, the stored maize has the same nutritional value as the reference (fresh) maize.

Since the hermetically stored grain has a much better hygienic quality (absence of moulds and toxins) than crib-stored maize, this technique appears to be of great interest for the cattle feed industry.

STUDY, NOW IN PROGRESS, TO DEVELOP A STORAGE TECHNIQUE USING AIRTIGHT PLASTIC SILOS (10 m^3)

The experiments, reported above, have demonstrated the economic and scientific interest of airtight storage of half-wet maize. But it has been also emphazised that, in order to be successful, this method demands that the concentration of O_2 be kept always below 1 or, better, 0.5%. Therefore the sealed silo must be absolutely airtight. As the use of metal silos is probably too expensive for large industrial development, we have decided to check, in a small model (10 m^3), the reliability of a silo made with non-rigid, supple plastic sheets placed over a metal frame. Such structure could be 5 times cheaper than a simple metal silo.

For this trial, we collaborated with a French company, specialising in fabrication of plastic sheets, but hitherto mainly for military purposes and conditioned storage rooms for fruits (Societe Bachmann*).

Experimental procedure

An experimental silo, of approximately 10 m^3, pyramidal shaped, has been built and set up at Boigneville, in the I.T.C.F. experimental research station.

The plastic sheets were made of PVC. The permeability of the material is less than:

- for O_2 : 42.8 $cm^3/m^2/24$ h, at 20°C, under differential pressure of 100 g;
- for CO_2 : 284 $cm^3/m^2/24$ h, at 20°C, under differential pressure of 100 g;
- for H_2O : 12 $g/m^2/24$ h, at 38°C, with differential R.H. of 95%.

The roof was doubled inside the silo by a light plastic awning, separating the grain from the roof. This system creates a bed of air, isolating the grain from changes of temperature, and preventing water condensing on the roof from dropping on the grain. The plastic material used for the silo was chosen for its highly airtight/insulating properties and non-absorbancy of radiant energy. This experimental silo has been equipped with various accessories, as shown in Fig. 3.

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* Bachmann Company : 69 rue Daniel Casanova, 94200 YVRY (France)

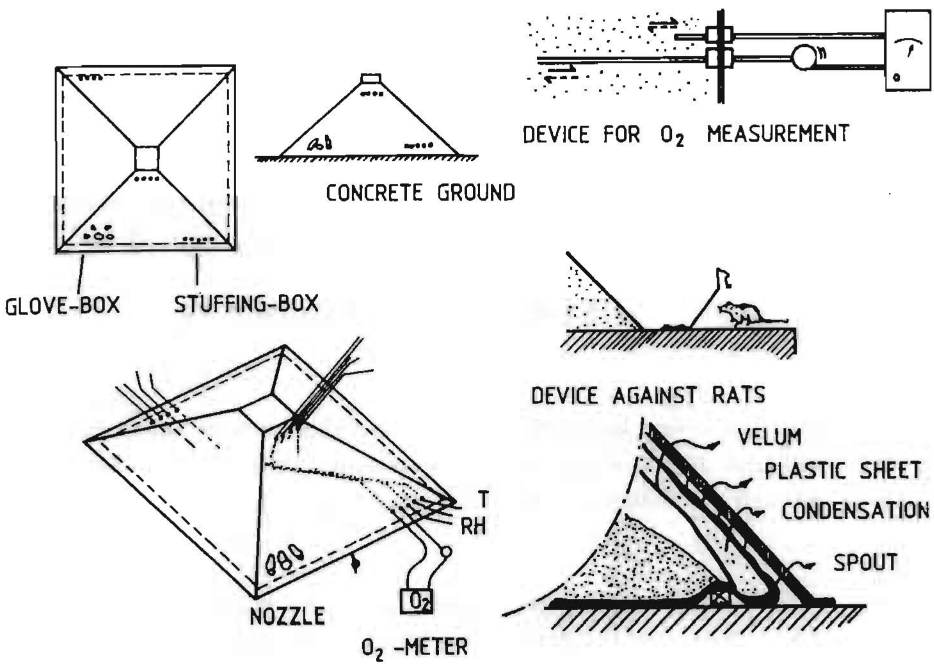

Figure 3. Arrangement of the different sampling systems in the experimental silo.

They are:

- a glove box and an air-lock allowing samples of grain to be taken without any introduction of oxygen (air) inside the silo ;
- temperature probes and relative humidity probes ;
- several tubes which could be connected to the device measuring the residual oxygen concentrations inside the silo ;
- some nozzles for removal of the water which eventually condensed and collected in the water receiver.

The maize used for this trial was grown on ITCF experimental fields. It was harvested at about 38% and then partially dried by ITCF to 19% moisture content.

The silo was filled at the end of November 1982.

Measurements were taken from each probe regularly once a week. When necessary, a sample of grain was taken for analysis.

Preliminary results and discussion

This trial, scheduled to run for 1 year, has now been in progress for 5 months. It is thus not possible to give complete conclusions for the trial but one can say that up to now it has been perfectly satisfactory.

The calculation used for prediction of the time taken for the oxygen to be removed can be used here. In this instance, with a final temperature of 5^{o} and larger volume, it is predicted that the process would take 9 days.

On two occasions an interesting incident occured : oxygen level was found to be too high (4 to 5%) and at the same time some mould development was observed. On inspecting the silo, a small leak was found in the stuffing box where probe wires pass through the wall. When this leak was sealed up by the technician, the level of oxygen very quickly decreased again below 1% and the moulds regressed.

It is interesting to note that these two minor incidents, due to experimental problems, paradoxically confirmed the good working of the system.

TENTATIVE CONCLUSION : FURTHER DEVELOPMENTS EXPECTED

In seven months the silo will be emptied and the grain analysed from a microbiological and technological point of view. In particular the starch extraction qualities will be measured in pilot scale.

If all the results are good and if the state of plastic is also good, then a full scale trial (5000 m^3) will be organized, in collaboration with a starch making company. This will test the industrial feasability and the economic place for the system in the post-harvest handling structure existing in France.

ACKNOWLEDGMENTS

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- Mr. Janny, general director of the Bachmann Company, fabricator of the

experimental silo.

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- Mr. Bertrand (Lab. Technologie des Aliments pour Animaux, INRA) who provided the nutritional evaluations.

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STORAGE OF MALTING BARLEY (C.V. CLIPPER) IN A NITROGEN ATMOSPHERE

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ABSTRACT

Two lots of malting barley (950 tonnes, c.v. Clipper) were stored under nitrogen (0.5% O_2). One was held for 5 months under nitrogen, the other for 9 months. A third lot was held under normal storage to act as a control. The trial was carried out by The Barley Marketing Board in conjunction with the C.S.I.R.O. Division of Entomology during 1980.

The aim of the trial was to find a lower cost alternative storage method to refrigerated aeration for the medium term storage of malting barley in Queensland. Although tanker-delivered nitrogen gas was used in the trial, and costs were consequently high, it was envisaged the 'lower cost' aim could have been satisfied, in the future, by the use of atmospheres produced by combustion or biological means on-site.

It was concluded that there was neither a significant beneficial nor detrimental effect from the nitrogen atmosphere itself on the germinability of the stored barley. However, there was substantial actual loss of germinability of the grain stored under nitrogen attributable to pregermination.

INTRODUCTION

Queensland's grain growing belt, being in the sub-tropics, has a grain storage environment unique in Australia. This is particularly relevant for the storage of malting barley. High intake temperatures coupled with a warm humid climate for the first few months of storage make the quality preservation in Queensland of barley for malting a rather delicate affair. The important quality parameter, is of course, germination. Barley of low germination obviously cannot be used to produce malt as the malting process is dependent, in simple terms, on the germination of barley, albeit under strictly controlled conditions.

The germination energy of barley, of the cultivar Clipper, unaerated and un-turned, initially at 98%, may gradually drop in storage down to 80% after twelve months. The storage temperature, initially around 30°C, does not normally fall below 20°C within this period. In the past, this problem has been overcome to varying degrees by turning the grain and by use of aeration. However, refrigerated aeration has proved to be the most successful solution to the problem to date. Because it is expensive to refrigerate large masses of grain, an alternative would be desirable.

The object of this trial was to test if storage in an inert atmosphere of 0.5% or less oxygen was viable commercially as an alternative. Although nitrogen delivered by tanker as a liquid was used in this instance as an inert gas source, it was envisaged that further economies could be obtained in the future by the use of combustion gases or biologically produced atmospheres.

Published information supported the feasibility of use of nitrogen to preserve the germination of malting barley. Glass et al. (1959) showed that in wheat stored at 30°C the onset of deterioration as measured by viability was delayed somewhat by storage in nitrogen as compared with storage in air. Roberts and Abdalla (1968) showed that the oxygen had a deleterious effect on the viability of barley in storage. Shejbal and Di Maggio (1976) in Italy demonstrated that barley at 30°C and 12% moisture lost germinative energy and capacity much faster when stored in air as compared with in nitrogen. Furthermore, Storey et al. (1977) showed that the storage of barley under inert or air atmospheres for six months at 27°C and 50% relative humidity had no adverse effect on the quality of malt produced from the stored barley.

During the trial it was found that the grain designated as a control was not strictly comparable with that stored under nitrogen as the latter had an unexpectedly high level of pregermination. This finding complicated the analysis of the data obtained but also gave information on this poorly recognised factor in commercial barley storage for malting.

MATERIALS AND METHODS

Trial Conditions

At the Barley Marketing Board's Harristown complex, there are eleven white-painted, welded steel bins capable of holding 950 tonnes of barley each. Three of these bins were selected for the trial. Two (designated Bin A and B) were modified prior to inloading so that they were sealable (pressure test:decay time (full), 1500-750 Pa, 7.8 mins each) and possessed a gas introduction system and safety valves. The modifications and gas introduction were carried out according to the procedures given in Banks and Annis (1977). A third bin in the complex, Bin C, was selected for use as a control.

Bins A, B and C were filled with cleaned malting barley (c.v. Clipper) of the 1979/80 crop. Bins A and B were then purged with nitrogen so as to attain an atmosphere within each bin with less than 0.5% oxygen (nitrogen input rates 1.2 and 4.8 m^3, usage 1.26 and 1.01 $m^3\ t^{-1}$ for Bins A and B respectively). Bin A was kept under this low oxygen atmosphere for 5 months and Bin B for 9 months before outloading and sampling. To maintain 0.5% oxygen, it was necessary to continuously bleed nitrogen into both bins. This maintnenance rate varied between 10 L min^{-1} and 40 L min^{1-} at various stages of the trial (average 26 L min^{-1}).

The grain was sampled on the loading of each bin. The loading rate was approximately 100 t h^{-1} with samples and temperature measurements taken every 15 minutes. All samples were tested for moisture content and germination. Selected samples also tested for pregermination precentage. Selected samples were also micro-malted and analysed by two separate malt laboratories, while some commercial malting was also carried out on the barley stored for five months under nitrogen (Bin B).

Analysis Methods

Temperature measurements were made with a thermocouple in the grain stream as each bin was loaded or unloaded.

Moisture contents were determined as per Institute of Brewing Method 1.2 : Moisture Content (oven method: 103°C to 104°C for 3 hours) (Anon. 1977).

Germination percentage was measured as per Institute of Brewing Method 1.4.2 : Germinative Energy (Anon. 1977)

Pregermination level was determined by E.B.C. - Analytica Method 2.6 'A Determination of Pregerminated Grains in Barley' (Anon. 1979).

Malt analyses were carried out in conformity with Institute of Brewing Methods (Anon. 1977).

RESULTS AND DISCUSSION

Table 1 summarises the germination, temperature, moisture content and pregermination data obtained for the three bins during the trial. The grain in each bin showed decrease temperatures of similar magnitude over the trial periods, while moisture content increased slightly in the same proportion in all three bins. Significant germination loss, as is evident from Table 1, was experienced in the two bins held under nitrogen, Bin A and Bin B, while Bin C, normal storage, showed no change in final germination. It is of interest to see an increase in the 24 h germination of Bin C for the 9 month period. It is also important to note here, that Bin C had significantly lower grain temperatures throughout the trial than Bins A and B, possibly enhancing storeability in Bin C.

The substantial loss of germination while under nitrogen was unexpected. Two approaches were adopted in an attempt to explain the observation. Firstly, all inloading and outloading samples were retested so that comparisons could be made between samples held in the laboratory and

Table 1 Analysis of quality of bin, before and after storage.

Quality Parameter	5 MONTH TRIAL						9 MONTH TRIAL					
	Bin B (N_2)			Bin C (Air)			Bin A (N_2)			Bin C (Air)		
	Jan 80	Jun 80	% change	Jan 80	Jun 80	% change	Jan 80	Oct 80	% change	Jan 80	Oct 80	% change
Moisture Content (%)	11.9	12.3	+3	12.0	12.3	+3	11.8	12.2	+3	12.0	12.3	+3
Temperature (°C)	28.3	21.0	-26	26.7	19.4	-27	30.1	22.4	-26	26.7	17.8	-33
Germination (%)												
4 ml-												
24 h	80.6	61.4	-24	78.7	69.4	-11	82.0	58.3	-29	78.7	86.5	+10
48 h	93.3	83.6	-10	95.8	90.5	-5	94.7	80.7	-15	95.8	95.6	0
72 h	94.7	88.2	-7	97.3	96.1	-1	96.7	85.7	-11	97.3	97.2	0
8 ml-												
72 h	52.7	77.7	+48	54.2	88.0	+62	57.5	64.0	+12	54.2	87.7	+62

Table 2. Mean germination and pre-germination. Analysis by bin.

		Moisture content (%)	Temperature (°C)	Observation soon after sampling Germination tests 4 ml 24 h	4 ml 48 h	4 ml 72 h	8 ml 72 h	Retest after laboratory storage Germination tests 4 ml 24 h	4 ml 48 h	4 ml 72 h	8 ml 72 h	Pre-germination (%)
Mean	Bin A	11.8	30.1	82.0	94.7	96.4	57.5	48.1	71.3	77.9	50.5	7.2
STD Dev	Initially	0.6	5.6	4.5	3.8	1.4	15.0	15.9	15.6	13.3	18.7	4.7
No. Samples		46	45	47	47	47	47	27	27	27	27	27
Mean	Bin A	12.2	22.4	58.3	80.7	85.7	64.6	42.4	64.3	74.8	47.8	3.9
STD Dev	After 9 mth	0.4	2.8	13.5	12.9	9.8	15.7	17.8	18.9	18.5	19.2	2.9
No. Samples	storage under N_2	42	42	42	42	42	42	42	42	42	42	42
Bin B												
Mean	Bin B	11.9	28.3	80.6	93.3	94.7	52.5	60.6	75.7	79.5	61.8	3.4
STD Dev	Initially	0.8	2.7	13.2	9.9	8.7	11.6	25.8	22.9	20.4	24.8	4.1
No. Samples		40	41	41	41	41	41	19	19	19	19	19
Bin B												
Mean	Bin B	12.3	21.0	61.4	83.6	88.2	77.7					
STD Dev	After 5 mth	0.9	2.6	21.2	18.1	15.4	20.3					
No. Samples	storage under N_2	40	39	40	30	30	40					
Bin C												
Mean	Bin C	12.0	26.7	78.7	95.8	97.3	54.2	74.1	90.0	93.8	73.5	0.8
STD Dev	Initially	0.7	4.9	5.2	2.6	1.8	11.6	6.2	4.9	3.8	16.7	1.4
No. Samples		46	46	46	46	46	46	32	32	32	32	32

Table 2 (continued)

		Moisture content (%)	Temperature (°C)	Observation soon after sampling Germination tests 4 ml 24 h	4 ml 48 h	4 ml 72 h	8 ml 72 h	Retest after laboratory storage Germination tests 4 ml 24 h	4 ml 48 h	4 ml 72 h	8 ml 72 h	Pre-germination (%)
Bin C												
Mean	Bin C	12.3	19.4	69.4	90.5	96.1	88.0					
STD Dev	After 5 mth	0.5	1.9	19.4	5.5	2.7	5.3					
No. Samples	storage	48	48	48	48	48	48					
Bin C												
Mean	Bin C	12.3	17.8	86.5	95.6	97.2	87.7	64.8	89.0	96.2	73.1	0.7
STD Dev	After 9 mth	0.4	0.6	5.1	2.8	1.7	5.3	10.7	4.7	1.8	7.4	1.1
No. Samples	storage	28	26	28	28	28	28	28	28	28	28	28

grain stored in the bins. Secondly the sampling profiles of the grain from the bins were examined, isolating comparable zones for micro-malting work and more definitive comparison of effect of storage atmosphere.

The average germination of samples of barley taken from Bins A,B and C measured soon after sampling, were compared with that for the same samples stored under laboratory conditions (dry, in paper packets, air conditioned area) until March 1981. The results are shown graphically in Fig. 1.

It can be seen that the grain in each bin is significantly different in terms of storage potential and that the germination decline in the barley stored in the bins under nitrogen was similar with that stored in the laboratory. It can be concluded that the storage method did not influence the retention of germinability appreciably.

Bin Profiles

Germination profiles for each bin are shown graphically in Figs. 2, 3 and 4. As can be seen by comparing initial loading results and fifteen month re-test results, some parcels of grain exhibited a substantial potential for loss of germination. In fact, both Bins A and B contained parcels of grain which were initially low in germination and progressively got worse. As luck would have it, Bin C, the control bin, contained no parcels of grain with an inherent potential for germination decline.

An attempt was made to account for this 'germination loss' potential by testing all available samples for pre-germination percentage. These results are also shown graphically in Figs. 2, 3 and 4 and, at the best, show only a casual relationship with viability decline. A more definite relationship is evident from the mean results for each bin (Table 3). Additional pre-germination data is given in Table 2.

Table 3 Pregermination levels before and after storage under laboratory conditions (4 ml test, 72 h assessment)

	On Inloading	After Lab. Storage		
Bin	Germination at Jan 80 %	Germination at Mar 81 (%)	Loss rate (% per mth)	Pregermin -ation (%)
Bin A	96.4	77.9	1.23	7.2
Bin B	94.7	79.5	1.01	3.4
Bin C	97.3	93.8	0.23	0.8

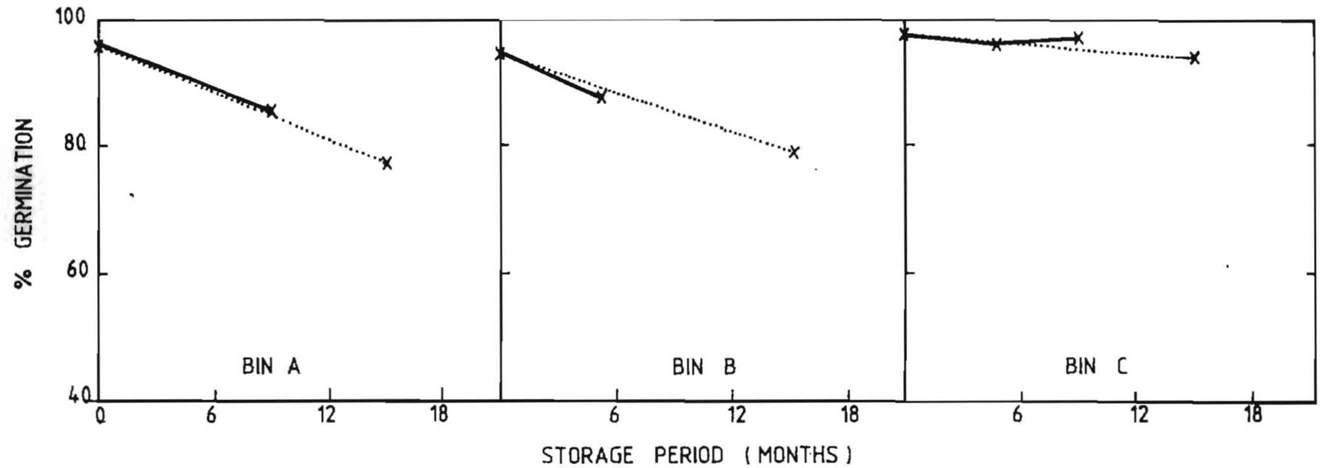

Fig. 1 Germination decline - bin storage (-) vs. storage in laboratory (.......). 4 ml test, 72 hr assessment.

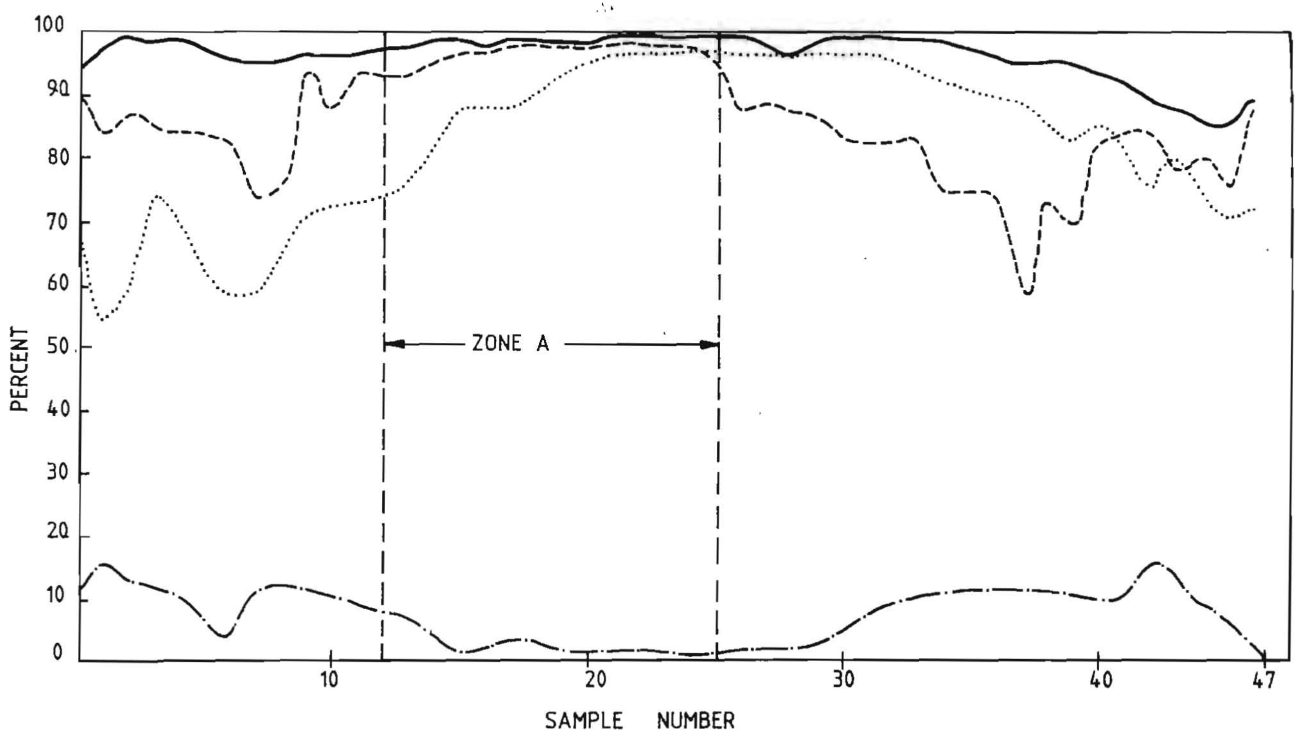

Fig. 2 Profile of germination and pregermination (-.-.) for Bin A. 4 ml test, 72 hr assessment (-, on loading; -----, after 9 months storage under nitrogen; ...., on-loading samples stored in laboratory).

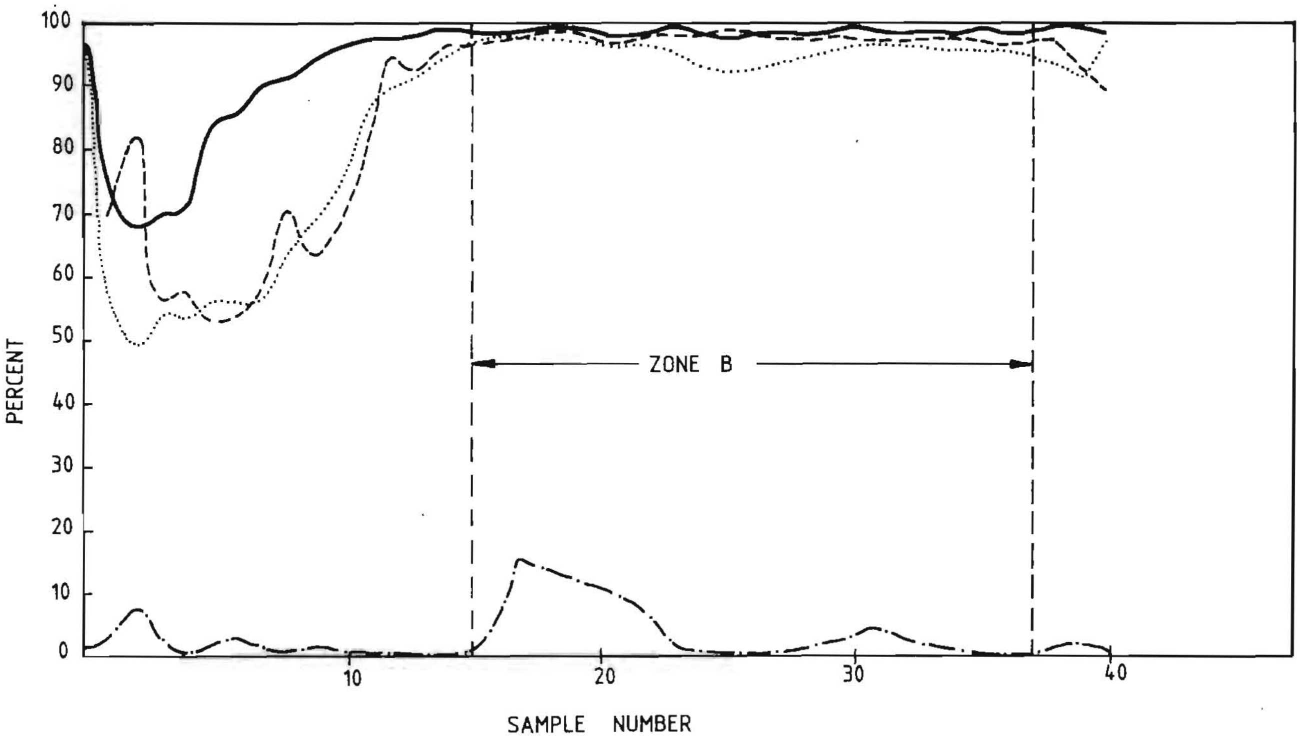

Fig. 3 Profile of germination and pregermination (-.-) for Bin B. 4 ml test, 72 hr assessment. (-, on loading; ----, after 5 months storage under nitrogen; ...., on-loading samples stored in laboratory).

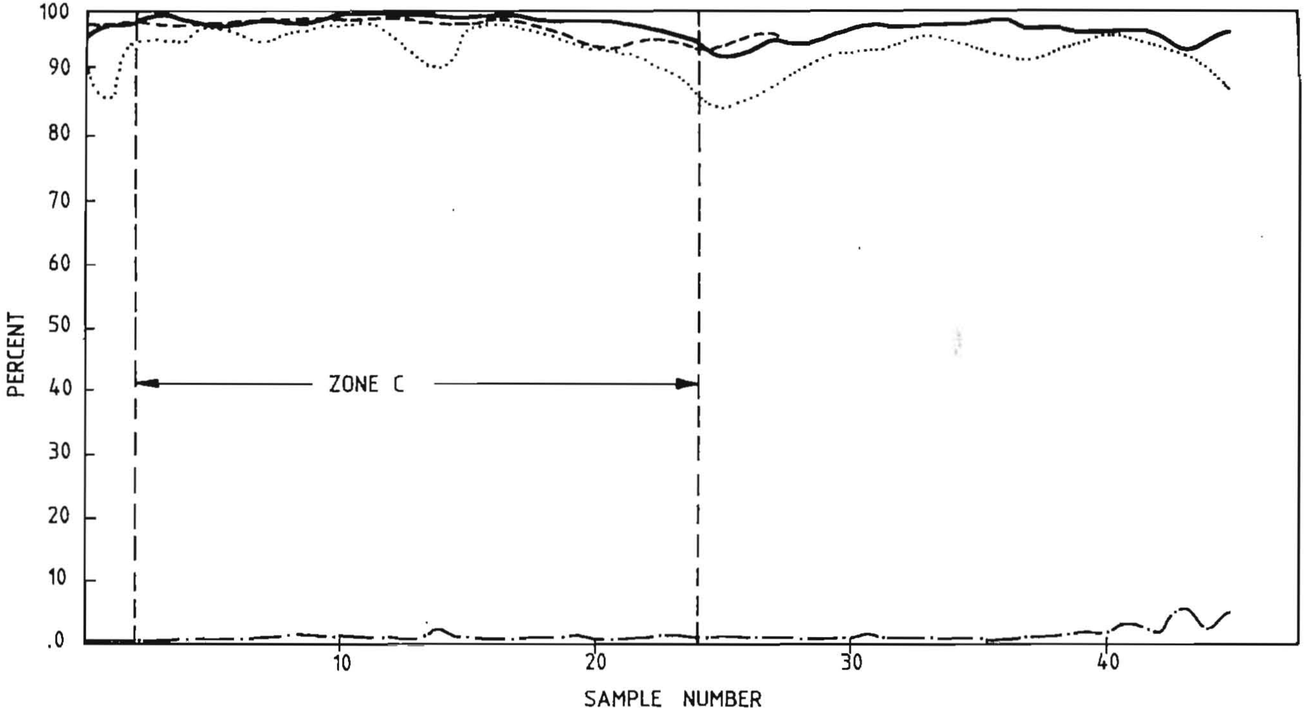

Fig. 4 Profile of germination and pregermination (-.-.) for Bin C. 4 ml test, 72 hr assessment. (-, on-loading; -----, after 9 months storage; ......, on-loading samples stored in laboratory).

In carrying out the pregermination testing, problems of reproducibility were encountered and this may well have hindered conclusive definition of casual relationship between pregermination and germination loss.

It is interesting to note here that the only comparable work done by us on grain stored under refrigerated aeration shows a loss rate of 1.19%/mth with a pregermination level of 17.2% (Appendix 1).

Zoning

Because of the variation in germination of the samples the concept of zoning was introduced, so that comparable grain could be isolated for micro-malting analysis. Grain samples on inloading and outloading were labelled as to which portion in the bin they represented. Given that the bins emptied from the top and the grain flow was at a constant rate, then it was considered probable that certain zones could be identified as the grain was transferred from one bin to another. The other proviso was that a zone should not involve any of the first three or last three samples of a transfer as it was logical to expect a certain amount of mixing here. The zone in Bin C was isolated because part of that bin had to be outloaded in June for reasons external to the trial.

The zones selected are shown in Figs. 2, 3 and 4, with the zone limits based on fifteen month re-test data and pregermination data from the laboratory - stored samples. Table 4 gives data similiar to that shown in Table 1, but with the analysis by zone rather than by bin. As can be seen from Tables 4 and 5 there is little difference between the germination (4 ml - 72 h) results of the treated and untreated zones after either 5 or 10 months of storage under nitrogen. Further data on grain from Zones A, B and C is given in Table 5. Of some interest in the results from 9 months storage under nitrogen is the fact that the percentage increase in 8 ml data is significantly less than the air control. Also, it is still of interest to see the 4 ml - 24 h results with a positive value for Bin C after 9 months storage.

Malt Analysis

Micro-malting and subsequent malt analysis was carried out by two malt laboratories on barley from Zones A, B and C. We were also fortunate to obtain the malt analysis of commercial malt produced from Bin B in two separate malthouses. The micro-malting work is summarised in Table 6 while Table 7 shows the results of commercial malt produced from barley stored for 5 months under nitrogen compared with standard control malts for the respective malthouses.

As can be seen from both tables, the storage under nitrogen had no adverse affects on subsequent malt quality. In fact, it is encouraging to

Table 4. Analysis of quality by zone.

Quality parameter	5 MONTH TRIAL						9 MONTH TRIAL					
	Zone B (N_2)			Bin C (Air)			Zone A (N_2)			Zone C (Air)		
	Jan 80	Jun 80	% change	Jan 80	Jun 80	% change	Jan 80	Oct 80	% change	Jan 80	Oct 80	% change
Moisture content (%)	11.4	11.8	+4	12.0	12.3	+3	11.8	11.9	+1	11.8	12.3	+4
Temperature (°C)	28.0	20.8	-26	26.7	19.4	-27	29.8	25.5	-14	26.8	17.9	-33
Germination (%)												
4 ml-												
24 h	87.0	73.7	-26	78.7	69.4	-12	84.5	73.6	-13	82.1	87.2	+6
-48 h	97.7	95.0	-13	95.8	90.5	-6	97.9	93.9	-4	97.1	96.1	-1
-72 h	98.6	97.7	-1	97.3	96.1	-1	98.9	96.6	-2	98.2	97.3	-1
8 ml-												
72 h	57.6	89.4	+55	54.2	88.0	+62	70.6	82.1	+16	56.1	87.3	+56

Table 5 Mean germination and pre-germination. Analysis by zone.

		Moist-ure content (%)	Temper -ature (°C)	Observation soon after sampling Germination tests 4 ml 24 h	4 ml 48 h	4 ml 72 h	8 ml 72 h	Retest after laboratory storage Germination tests 4 ml 24 h	4 ml 48 h	4 ml 72 h	8 ml 72 h	Pre-germin-iation (%)
Zone A												
Mean	Before treatment	11.8	29.8	84.5	97.9	98.9	70.6	61.1	84.7	89.4	68.5	1.7
STD Dev		0.5	1.1	3.5	1.1	0.9	9.0	9.0	9.6	7.8	19.2	2.4
No. Samples		14	12	14	14	14	14	6	6	6	6	6
Zone A												
Mean	After 9 mth storage under N_2	11.9	25.5	73.6	93.9	96.6	82.1	63.9	86.9	93.5	69.3	2.7
STD Dev		0.4	0.8	5.3	3.8	1.5	6.3	10.1	5.7	4.5	9.4	2.1
No. Samples		12	12	12	12	12	12	12	12	12	12	12
Zone B												
Mean	Before treatment	11.4	28.0	87.0	97.7	98.6	57.6	82.1	95.2	97.0	82.1	5.0
STD Dev		0.4	1.4	3.1	0.9	0.6	10.0	3.8	2.8	2.3	5.0	5.9
No. Samples		23	23	23	23	23	23	7	7	7	7	7
Zone B												
Mean	After 5 mth storage under N_2	11.8	20.8	73.7	95.0	97.7	89.5					
STD Dev		0.3	2.1	6.0	1.9	0.7	4.4					
No. Samples		23	23	23	23	23	23					

Table 5 (continued)

Zone C												
Mean	Initially	11.8	26.8	82.1	97.1	98.2	56.1	76.1	93.0	95.8	82.6	0.3
STD Dev		0.5	0.9	3.5	1.7	1.2	14.0	5.3	3.1	2.5	6.6	0.6
No. Samples		22	22	22	22	22	22	15	15	15	15	15
Zone C												
Mean	After	12.3	85.7	95.2	98.0	91.6	18.8					
STD Dev	5 mth storage	0.4	3.5	2.3	1.1	3.1	2.1					
No. Samples		22	22	22	22	22	22					
Zone C												
Mean	After	12.3	17.9	87.2	96.1	97.3	87.3	66.4	89.5	96.4	73.4	0.7
STD Dev	9 mth storage	0.4	0.6	2.9	1.9	5.3	5.3	11.0	5.1	2.0	7.7	1.2
No. Samples		22	20	22	22	22	22	22	22	22	22	22

Table 6 Micro - Malting Results for barley stored in air and under nitrogen.

Laboratory	Zone	Barley Sampling date	% Extract (dry)	Modification index (%)	Diastase (^{o}L)
Laboratory 1	Zone A	Initially Jan 80	77.5	33.9	65
(0.1 ppm GA + 100 ppm bromate)	"	After Storage Oct 80	78.3	34.6	67
	Zone B	Initially Jan 80	77.0	32.9	68
	"	After storage Jun 80	78.1	37.7	76
Laboratory 2	Zone A	Initially Jan 80	81.0	44.1	77
(0.2 ppm GA + 150 ppm bomate)	"	After storage Oct. 80	80.9	43.9	78
	Zone B	After storage Jun 80	81.3	50.6	86
	Zone C	Initially Jan 80	82.5	52.4	80

Table 7 Commercial Malting Results for barley stored for 9 months under nitrogen.

Malthouse	Barley	% Extract (dry)	Modification index (%)	Diastase (^{o}L)
Malthouse 1	Zone B	85.4	44.8	74
	Malthouse control	84.5	48.4	63
Malthouse 2	Zone B	79.1	37.7	79
	Malthouse control	79.0	37.1	69

note, that the malt enzyme parameter, diastase, is higher for the 'nitrogen' barley in every case where a comparison can be made.

CONCLUSIONS

Although inert atmosphere storage of barley has no detrimental effects on the malting quality of that barley, it has no advantage over the present techniques employed for the medium term storage of malting barley in Queensland.

This trial has indirectly produced evidence which more positively identifies pregermination as a factor in germination decline of barley in commercial storages.

ACKNOWLEDGEMENTS

This trial work was sponsored by The Barley Marketing Board (Q'land) and without support from senior Management and Board Members would not have been possible. Dr. Jonathan Banks provided the inspiration, the impetus, and certainly a large part of the energy required to conduct this trial and for this, I thank him very much.

Thanks must also go to the following companies and organisations and their staff for assistance in this project: C.S.I.R.O. Division of Entomology, The Commonwealth Industrial Gases Limited, Carlton and United Breweries Limited, Queensland Malting Company Pty Limited and Barrett Burston (Australia) Limited.

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APPENDIX 1

Pregermination levels and germination tests for barley (c.v. Clipper) stored for 7 months under refrigerated aeration (Brookstead, 80/81 season).

Sample	Moisture content (%)	Temperature (°C)	Germination 4 ml test 24 h	48 h	72 h	Pregermination (%)
A 4	10.2	17.6	68	86	94	34
A 8	10.7	17.2	75	91	93	25
A12	10.9	17.6	74	92	97	16
A16	10.9	17.3	68	86	92	27
A20	9.6	18.3	65	82	89	26
A24	10.6	17.9	77	88	92	25
A28	10.2	17.9	61	84	90	30
A32	9.9	19.6	73	85	88	31
A36	10.8	18.9	72	82	90	29
A40	10.1	18.3	55	86	92	23
A44	10.6	18.2	60	91	93	31
A48	10.1	17.4	77	91	94	13
B 4	11.2	13.6	66	86	91	9
B 8	10.6	13.4	75	86	93	8
B12	10.1	13.1	73	87	82	9
B16	9.9	12.9	75	86	93	9
B20	9.8	12.6	68	91	94	10
B24	10.2	13.2	72	87	90	10
C 4	10.6	12.3	71	82	90	12
C 8	9.8	12.6	74	87	84	34
C12	10.2	12.3	28	79	92	22
C16	11.1	11.7	75	86	90	14
C20	10.6	11.2	76	86	91	17
C24	9.8	10.6	73	83	84	4
D 4	10.9	12.3	65	77	88	5
D 8	10.7	12.6	69	84	89	11
D12	10.7	11.7	74	87	91	10
D16	10.5	11.4	70	87	96	4
D20	10.4	11.6	75	86	93	10
D24	10.2	10.7	80	93	97	9
Mean	10.4	14.5	69.5	86.1	91.7	17.2
STD. DEV.	0.4	3.0	9.6	3.7	2.3	9.8

THE USE OF CARBON DIOXIDE FOR QUALITY PRESERVATION IN SMALL SHEETED BAG STACKS

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Abstract: Successful trials of the combined use of carbon dioxide and well sealed flexible PVC sheeting for insect control in 100-200 t stacks of bagged cereal products have been reported elsewhere. For smaller stacks, the surface area to volume ratio is increased, and effective gas concentrations may be more difficult to maintain. A trial carried out in Lae, Papua New Guinea, showed that the method was suitable for smaller stacks (10-20 t). An average concentration of > 90% CO_2 was obtained by adding approximately 4.0 kg of carbon dioxide per tonne of commodity. The loss rate constant of CO_2 from the enclosed gas was between 0.012 and 0.025 day^{-1} in the three stacks treated. The treatment helped preserve the quality of green coffee beans in this hot and humid environment, where rapid degradation normally occurs if this commodity is stored in open stacks.

INTRODUCTION

Insect control in bag stacks of stored products may be thought of as having two phases; disinfestation and prevention of reinfestation. Both of these operations present difficulties in bag stacks. Disinfestation is usually carried out by fumigation with either methyl bromide or phosphine under 'gasproof sheeting'. This sheeting is typically a plain sheet of PVC or polythene draped over the stack and sealed to the floor using long, heavy and flexible weights, e.g. 'sand snakes' or chains. Usually, no attempt is made to establish the level of gastightness other than a visual inspection for obvious leaks, accompanied occasionally by testing for detectable concentrations of fumigant outside the enclosure.

Enclosures formed in this fashion may be significantly leaky even when apparently well sealed (Annis *et al.* in press). This leakiness may lead to areas of low fumigant concentration and consequently an imperfect fumigation.

Conventionally, the cover is removed after fumigation, and the stack is consequently exposed to possible reinfestation. Prevention of reinfestation is usually attempted by a combination of store hygiene, application of pesticide to the outer surface of the stack by spraying and general fogging of the storage environment with pesticides. Such measures are often unsuccessful and may lead to the need for repeated fumigations.

A treatment consisting of fumigation under a substantially gastight enclosure followed by prolonged storage within this 'insect proof' enclosure should reduce the number of operations involved in protecting the product from insect damage when stored for long periods. To avoid degradation of quality in such an enclosure, it is essential that any insect infestation must be eliminated shortly after the enclosure is sealed. Insects, apart from causing physical damage to the product, may cause localised increases of heat and moisture which could lead to levels of these factors which are conducive to mould growth, with consequent quality degradation and risk of mycotoxin formation.

There should be only a small change in the total amount of water contained within the enclosure during the storage period, provided that all insects are killed, and the product is dried to a level at which its endogenous respiration is low (Oxley 1948). Furthermore, if diurnal and seasonal temperature variations are not great, the water activity within the stack will not change significantly and there should be little risk of liquid water forming ("sweating" or condensation). Where a product is drier than the equilibrium moisture content of the ambient air, a well sealed enclosure may offer a partial barrier to moisture ingress. This is especially desirable when the contained atmosphere is occasionally flushed out with a dry gas.

Accordingly, a trial was set up at Lae, Papua New Guinea (PNG), in collaboration with the PNG Coffee Industry Board, to determine whether high grade green coffee beans could be stored without deterioration for prolonged periods in sealed sheeted stacks in a coastal area where the relative humidity is normally high. Under normal storage conditions (unsheeted bag stacks) in these areas, high quality coffee beans suffer substantial quality degradation after only a few weeks of storage. A further aim of this trial was to show that the process designed for large stacks (100 - 200 t) (Annis *et al.* in press) was applicable to stacks in the 10 - 20 t range.

METHOD

The trial was conducted in a well ventilated, corrugated iron warehouse normally used to store a wide range of products not usually liable to infestation. While the trial was in progress, there was an infestation of *Tribolium castaneum* (Herbst) in some stacks of chicken feed held in the warehouse. Routine insect control measures were not carried out in the warehouse during the course of the trial.

Two stacks each of approximately 18 tonnes of green coffee beans (1 stack *Coffea robusta*, and 1 stack Y grade *C. arabica*) and one stack of approximately 9 tonnes (mixed *C. robusta* and *C. arabica*) were built on thick (0.76 mm) polyvinyl chloride (PVC) floor sheets. All the stacks were found to have a moderate infestation of *T. castaneum* and larval *Ephestia* sp. when they were enclosed.

THE ENCLOSURE

The stacks were enclosed within tailored fumigation enclosures made of nylon-reinforced, PVC sheeting (Wavelock 41), and the outer edge of each enclosure was bonded to the edge of the PVC floor sheet with a urethane sealant. After checking the enclosure for obvious leaks, a small vacuum cleaner was connected to the gas inlet pipe built into the enclosure to create a negative differential pressure of about -1500 Pa (15 cm water gauge) with respect to atmospheric pressure. The inlet was then sealed and the pressure change over time recorded (see Annis *et al.* in press, for a detailed account of sealing and pressure testing such enclosures). The decay from -500 to -250 Pa took 11, 10 and 7 minutes for stacks 1, 2 and 3 respectively. The pressure test standard for a carbon dioxide (CO_2) treatment using a single gas addition to disinfest a full, rigid sealed structure with a capacity between 300 and 10 000 t is 5 min, for a halving of differential pressure (Banks and Annis, 1980).

GAS INTRODUCTION AND INITIAL GAS HOLDING

In all stacks, CO_2 was added as 'snow' obtained from 30 kg cylinders of food grade gas fitted with eductor tubes. The 'snow' was piped so that it would spread between the timber layers of the base pallets and sublime without contacting the lower bags. Carbon dioxide was added to each stack until the gas leaving an exit vent made in the top of the stack contained > 90% CO_2. The enclosures were then completely sealed. The introduction process used between

60 and 70 kg of CO_2 (2-2.3 cylinders) per 18 t stack less than 45 kg (1.5 cylinders) for the 9 t stack.

At 20°C, the regime required for successful insect control is an initial CO_2 concentration above 70% and a concentration above 35% for > 10 days (Banks *et al.* 1980). All three stacks maintained a concentration of above 35% CO_2 for longer than 25 days, and had CO_2 loss rate constants 0.024, 0.012 and 0.025 day^{-1} for stacks 1, 2 and 3 respectively.

RESULTS AND DISCUSSION

The stacks were opened after 26 weeks of sealed storage and the coffee and the others areas of the enclosed stacks were found to be free of insects. The coffee in stacks 1 and 2 was judged by local graders to have maintained its high quality, and had not suffered any of the humidity-related damage expected when coffee is stored in the humid coastal areas for more than a few weeks. The contents of stack 3 had suffered significant moisture damage, as evidenced by "caking" in the top two rows of sacks and a "distinct off odour". The timber pallets used for the base of this stack were saturated with water when the stack was made, while those used for the other two stacks were dry. This was the only difference noted in the treatment of this stack from the other two, and may have been partially responsible for the moisture problems.

All bags and stack covers were free from rodent damage when they were unsealed despite the presence of many rats in the warehouse. The level of rodent infestation was clearly high because there was substantial rodent damage to many of the outer bags in all of the test stacks within 2 days after they were uncovered. This observation suggests that the sealed and treated enclosures on this occasion offered the product some protection against rodent attack.

CONCLUSION

This trial demonstrated that a well sealed, flexible PVC enclosure combined with a CO_2 treatment can be used to disinfest stacks of bagged product as small as 9 tonnes. The enclosure's "insect proofness" is such that it appears to give long term protection against reinfestation. The treatment also offers a barrier to the damaging humidity normally found in coastal, tropical areas, and allows prolonged storage of products normally damaged by this type of environment. The method is sensitive to faults in operation, and additional research is required before many of the problems encountered can be evaluated and corrected and the technique recommended for routine storage.

ACKNOWLEDGEMENTS

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SESSION 4.

STORAGE SEALING TECHNIQUES (1)

Papers by:

W. Woodcock	–	Australia
I. Alexander	–	Australia
W. Glet	–	West Germany
E. R. Sutherland and G. Thomas	–	Australia
D. Lloyd	–	Australia
C. O'Neil	–	Australia
D. M. Ellis	–	Australia
D. J. Calverley	–	United Kingdom

THE PRACTICAL SIDE OF SILO SEALING

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ABSTRACT

The sealing of a 'horizontal' grain storage shed is described and practical details of the process are given. For a successful sealing, it is necessary to pay strict attention to many apparently insignificant items. These include very careful initial preparation of the surfaces to be treated, correct application of polyurethane foams for filling large voids in the structure, sealing of imperfections in the floor, and appropriate selection of sealing systems to treat problem regions such as around skylights and moving parts (eg. distributors for grain). Photographs are included illustrating the various features that require sealing in a horizontal storage.

INTRODUCTION

There are many types of silos : concrete vertical cells, welded or bolted metal cells, 'horizontal' storages made of concrete and sheet metal on a metal frame work or sheet metal supported by a wooden and metal structure. Each of these methods of construction can be used for a range of storage sizes and shapes. This paper gives details of sealing of a warehouse-type, 'horizontal' storage and describes problem areas encountered and how these areas can be treated. The information is based on the experience gained in the sealing of one such storage that was carried out successfully 3 years ago. Many of the problems met are similar to those found in other types of silo.

PRELIMINARY CONSIDERATIONS

The sealing system used must fulfill these criteria:

Must be flexible, and able to bridge and seal gaps up to 2 mm with no failure.

Must withstand high and low temperatures.

Must have good adhesion.

Must be suitable for use with foodstuffs.

Must not creep or perish.

Must be U.V. radiation resistant.

Must have good abrasion resistance.

Materials must be stable to fumigants.

Must be easily repairable.

Material and application costs must be reasonable.

Must have 10 year life or longer.

Several different products in combination can be used to achieve the seal.

Cleanliness of the structure is of utmost importance for good adhesion of the coating, so a complete, high pressure water or detergent wash, inside and out, is essential.

The type (galvanised, aluminium, colourbond etc.) and condition (new, weathered) of the metal cladding determine if primers must be used. There are many different products available for silo sealing. They may be water or solvent-based and have various specifications. Manufacturers of the products provide technical data sheets and application rates. The products may be applied to certain parts of the silo at rates exceeding the specification, as a double safeguard.

Polyurethane foam, foamed in situ, is used by all companies involved in silo sealing to fill the main voids and gaps in the structure and to strengthen and bridge weak areas. The foams used have fire-retardant properties. Internally, foam of 1kg m^{-3} density is used. Externally, 1.5 kg m^{-3} density foam is applied and, when used, flexible foam is applied at 0.5kg m^{-3}.

There are three general approaches to silo sealing : complete internal seal with external heat reflectant coating, complete external seal, and part internal and part external sealing. The latter method is lowest in cost and is most favoured.

After cleaning down, the following items must be attended to before sealing is attempted:

All nuts, bolts, screws to be checked. If missing, replaced.
All badly damaged, badly rusted, roof cladding to be replaced.
All gutters and downpipes checked. Replace any parts damaged or badly

rusted.
Any loose flashings, roof cladding, expansion joint covers, ridge capping etc. to be fixed firmly.
Extraction fans on roof to be removed and a cap fitted over the hole left.
Fibreglass skylights replaced or sealed where necessary.
Ridge vents, where fitted, to be capped.
Internal rubber door seals to be removed.

DETAILS OF SEALING PROCESS

Filling of large gaps with polyurethane foam (internal treatment)

The largest gaps and voids in a horizontal silo of the type discussed here are on top of the concrete wall where it meets the metal-clad curtain wall. (Fig. 1) Polyurethane foam is applied internally to the top of the wall to give a fillet which both seals this region and also is steep enough (45^{o}) to shed grain, preventing the build-up of dust and grain residues that is normally a problem there.

When polyurethane foam is to be applied over large gaps, it is advisable to fill these first with pre-moulded, flexible polyurethane foam to prevent the uncured foam blasting through the gap. This also cuts material cost and reduces the cost of cleaning up afterwards.

The lap joins in the metal cladding and at the gable ends are sometimes foamed with polyurethane. This reduces total time for sealing and usage of material on external surfaces. The join between the curtain wall and roof cladding (Fig. 2) is foamed. It is recommended that the foam extend 20 cm down the wall and 20 cm onto the roof cladding to strengthen the area and reduce movement there.

The ridge line of the roof (Fig. 3) is filled with polyurethane foam to a depth of 10cm, extending between the top purlins on each side. This treatment not only seals but strengthens the region. The ridge is walked on frequently during the sealing operation.

The 'penthouse', where the grain conveyor enters the storage, has an abundance of problem areas to seal. It is coated completely internally with foam. This system also reduces temperature build-up.

Large voids and gaps around girders passing through the curtain walls are filled with foam. The region where the base plate holding the roof truss, and also the top section of the roof truss, goes partially through the wall should also be sealed.

Electrical conduits entering or leaving the silo should be sealed into the structure with foam. Electrical circuit check points or circuit panels should not be foamed, but should be fitted with rubber or silicone rubber gaskets so they are sealed, but openable.

The roof ventilation fans and ridge vents are removed and the holes left capped off using corrugated iron, fibreglass sheet or polystyrene foam. This capping is then sealed to the silo with polyurethane foam.

All exposed polyurethane foam is coated with a sealant membrane for protection against damage and degradation.

Other sealing carried out internally

The grain distribution chute (Fig. 4) is sealed by taping a heavy duty plastic sheet or bag over the end. The chain lines used for manual operation of the valve from outside are sealed with silicone rubber, butyl mastic or acrylic sealant from a cartridge gun at the point where the chains go through the penthouse wall.

Fibreglass skylights are lighter and flimsier than the roof cladding and should be fixed on all corrugations to avoid tearing under wind pressure or pressure resulting from temperature variation when the shed is sealed up.

At certain times of year condensation may build up on the underside of the metal roof cladding and the fibreglass skylights. It can then run down the cladding. If the horizontal lap joins between the roof sheets are not sealed, moisture can build up behind the external membrane there, bringing a danger of corrosion and possible membrane breakdown.

The concrete walls can be treated either internally or externally. Or, for best results, both internally and externally. The external application should be done first to avoid rain water passing through unsealed cracks and collecting between the internal membrane and the wall. A good primer is required giving excellent adhesion to concrete and capable of covering hairline cracks, pitted regions and both porours and shiny impervious areas. The top coat must have good flexibility and abrasion resistance and be applied at least 500 microns dry thickness. Experience has found thinner membranes to be unsuccessful.

External sealing operations

Skylights

Skylights should be fixed down with pop-rivets. A cloth, fibreglass or bitumen tape should be applied over the edges of the skylight to reinforce the membrane applied in this weak area (Fig. 5). Skylights may deteriorate badly under the intense sunlight of Western Australia. In some instances they must be replaced. Sometimes, it may be possible to repair them using a polyester resin coating to rebond the fibres. A heat reflectant coating over the pannel eliminates further deterioration. Hairline cracks in skylights are a problem. These are caused by incorrect fitting, rough handling and fixing with screws or bolts or damage from workmen standing on the skylight.

Penthouse

When treating the penthouse it is first necessary to ensure that all metal flashings and claddings are bolted or pop-riveted securely. Sealants applied by cartridge dispensers are very useful for preliminary sealing around window frames etc. before the sealing membrane is applied.

Polyurethane foam has been found to be a successful gap filler when used externally. Unfortunately in Australia the abundance of bird life, especially parrots, is a problem. They peck into it, removing the protective membrane and eventually exposing parts of the foam. This allows moisture to penetrate into the foam and leads to rapid deterioration under our sunlight.

The region where the penthouse fits into the main roof is a problem (Fig. 6). The full foaming internally eliminates most leaks. However the penthouse must also be sealed externally here (e.g. with a flexible membrane sealant with bitumen or cloth tape reinforcement) to prevent rain getting behind the foam inside.

Almost all the thermal movement in a storage takes place under the expansion joint cappings (Fig. 7) This makes the region particularly difficult to seal. Flexible polyurethane foam (0.5 kg m^{-3} density) can be sprayed into the joint from below and a flexible membrane (e.g. a styrene-acrylic emulsion system) applied with reinforcement over the top of the join. The foam provides a very good seal by itself.

Gable Ends

The gable ends can be sealed using a flexible spray-applied membrane externally. Unfortunately it may be necessary to apply several coats of sealant to achieve specified thickness because thick films tend to run on the vertical surface. The seams in the gable end may be sealed internally with polyurethane. In this case a thinner external coating is acceptable. The place where the metal cladding of the upper part of the gable end meets the concrete wall (Fig. 8) is filled with polyurethane foam completely. This removes a haven for birds, moths, spiders etc. and prevents wind damage to the sealing membranes. If this region is not filled it is possible for high winds to blow up into this region causing excessive movement of the cladding.

Roof

The region where the roof cladding joins the walls at the gutter line is a critical area for sealing and many of the problems encountered occur in this region (Fig. 9). Two systems of sealing have been used successfully there. After removal of the gutters, each lap join is unbolted, the top sheet is lifted and a piece of felt or reinforcing material, dipped in the membrane sealer used (eg. an acrylic), is placed between the two sheets. The cladding is then bolted down again. Alternatively bitumen sealing tape can be used in place of the felt. The tape should be at least 2mm thick. It should be applied so that it seals between the sheets for at least 15cm along the join and for 15cm across the sheets at the open ends. This sealing should be carried out before any polyurethane foam is applied to the region internally.

The main horizontal laps in the roof should be secured by additional screws or pop-rivets before sealing to ensure that the membrane applied does not split under stress and movement (eg. from wind pressures or traffic during the sealing or inspection). The sealing membrane should be reinforced there (eg. with woven fibreglass tape). All laps should be coated so that the sealing membrane extends for at least 12mm each side of the join and with a thickness of more than 2mm.

All regions subject to movement (eg. around the grain inloading conveyor, where the roof trusses protrude through the curtain wall) must be sealed using a combination of polyurethane foam and flexible spray applied membrane coatings, reinforced with fibreglass fabric or other material. Any polyurethane used externally is coated with a protective sealing coating to prevent degradation by UV-light. A fine steel mesh can be embedded in the coating to prevent damage to the foam by parrots.

Doors

Sealing of the doors is always difficult and has to be redone each season. The main entry doors can be sealed internally with reinforcing tapes coated with a spray-applied flexible membrane. Access doors can be sealed externally using the same system. A new, less awkward system is now under development for sealing doors. This uses a light weight false door made of polystyrene foam protected with fibreglass that clips over the door frame externally. The edges are sealed to the frame using the flexible membrane system and reinforcing tape.

Floors

The concrete floors have been found to leak significantly. After cleaning, the construction joins and cracks are coated with the flexible membrane system and the whole floor is then sealed with a penetrant concrete sealer to fill porosity and hairline cracks.

Heat Reflectant Coat

After sealing a heat-reflective white coating is applied over the whole storage. This not only reduces the fluctuation in internal temperatures but also reduces the thermal expansion and contraction of the structure reducing stress on the sealing membranes.

CONCLUSION

Many minor points have not been covered in this paper. However, using the techniques described above, most problems in silo sealing can be overcome.

The motto of the silo sealing industry is "All Silos are Sealable'.

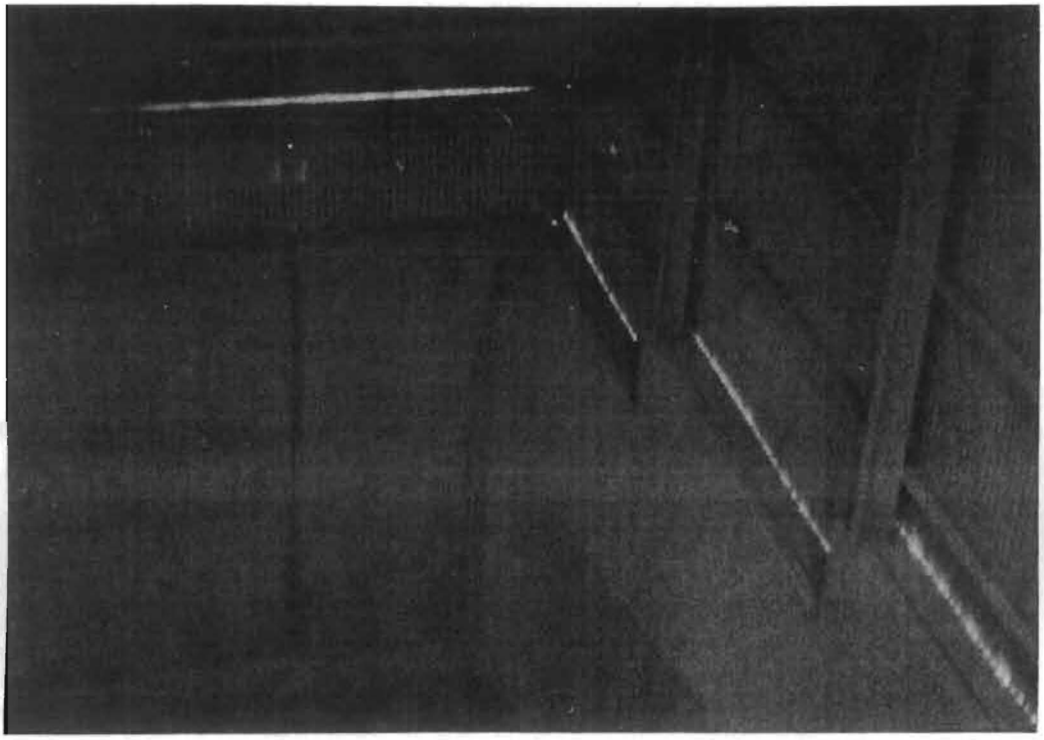

Fig. 1 The join between the concrete wall of the storage and metal cladding of the curtain wall, showing large gaps to be bridged before sealing.

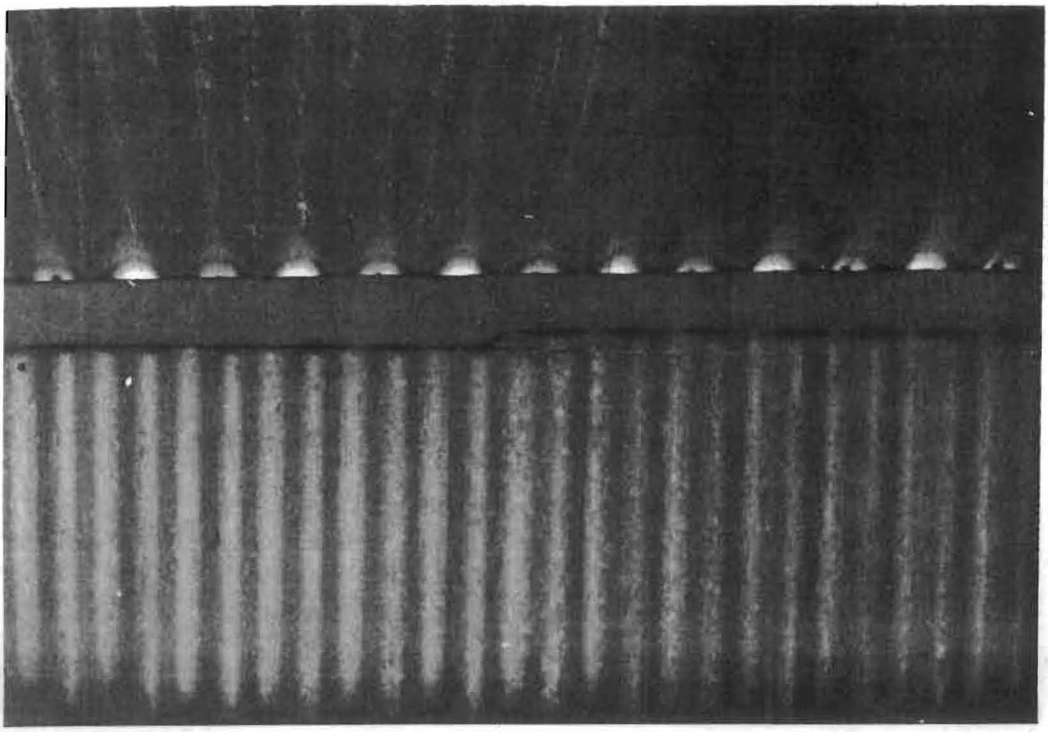

Fig. 2 Internal view of the wall-to-roof region.

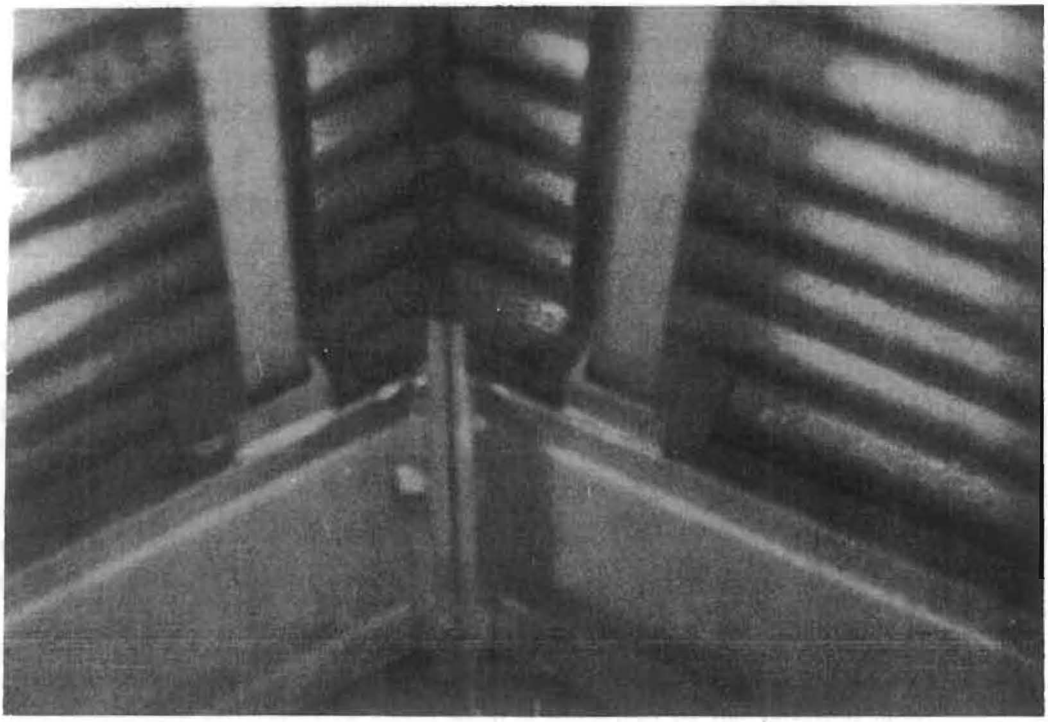

Fig. 3 Internal view of the ridge line of the roof in the penthouse.

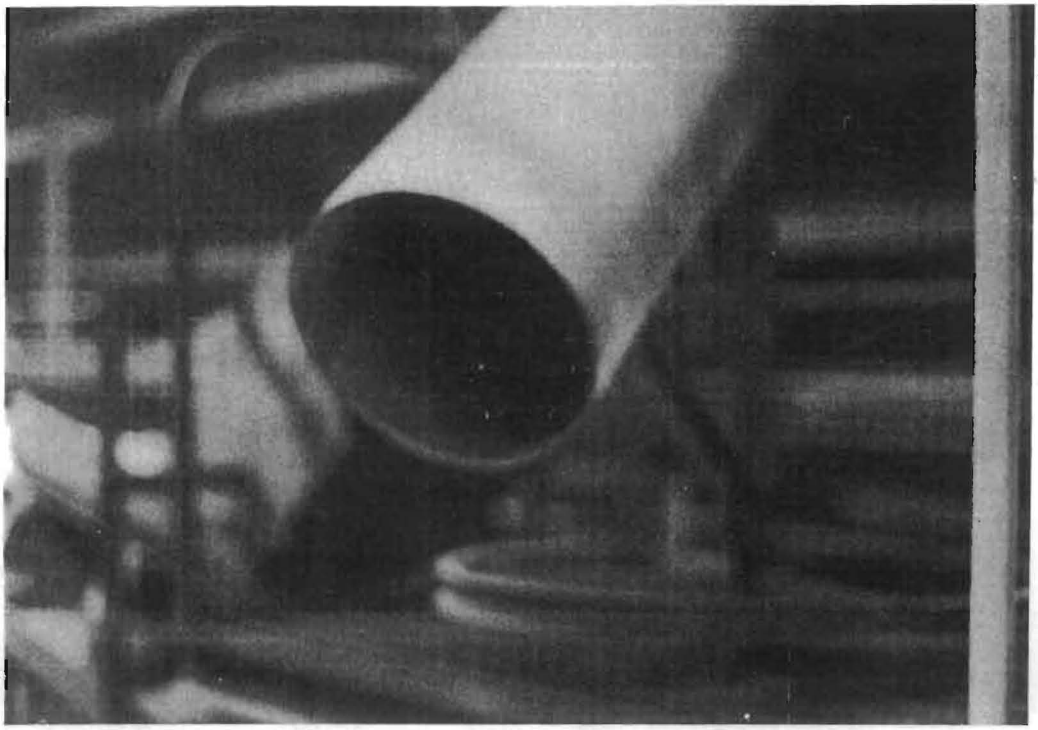

Fig. 4 Distribution chute for grain at the top of the storage.

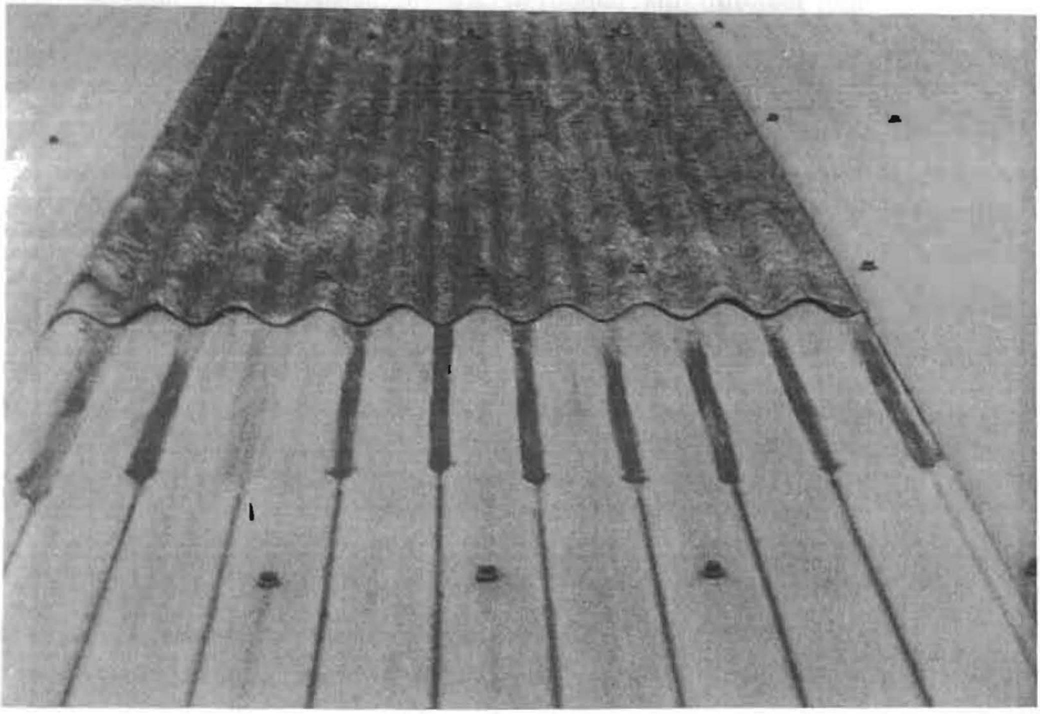

Fig. 5 Region of the roof where the skylight overlaps with the metal roof cladding.

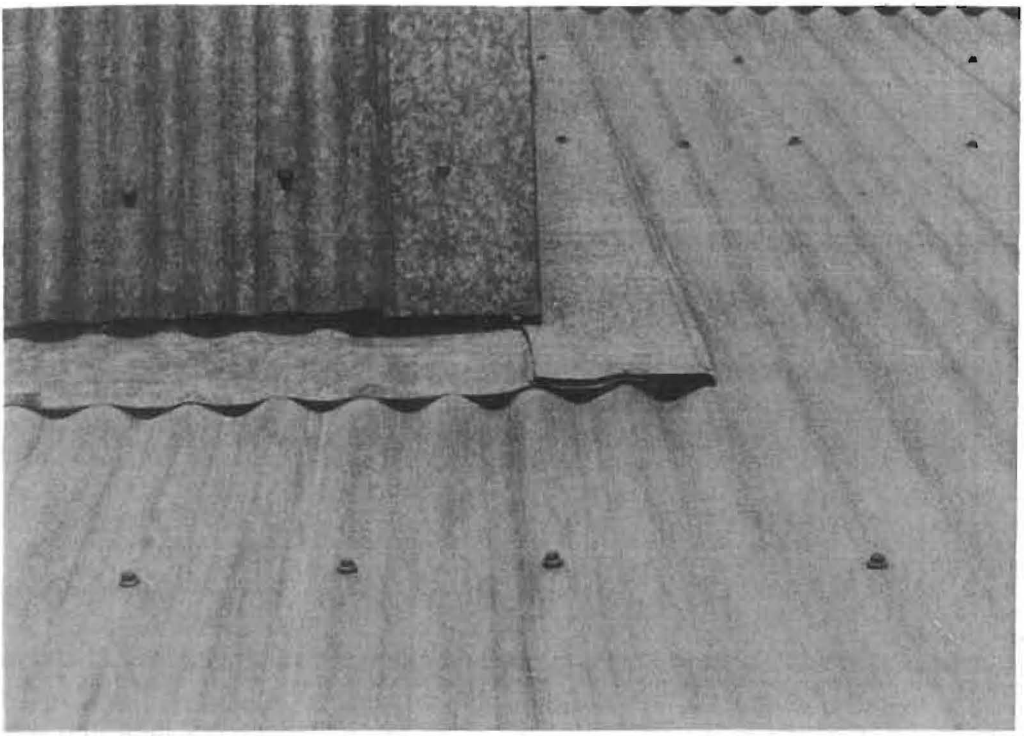

Fig. 6 The join between the penthouse and the storage roof.

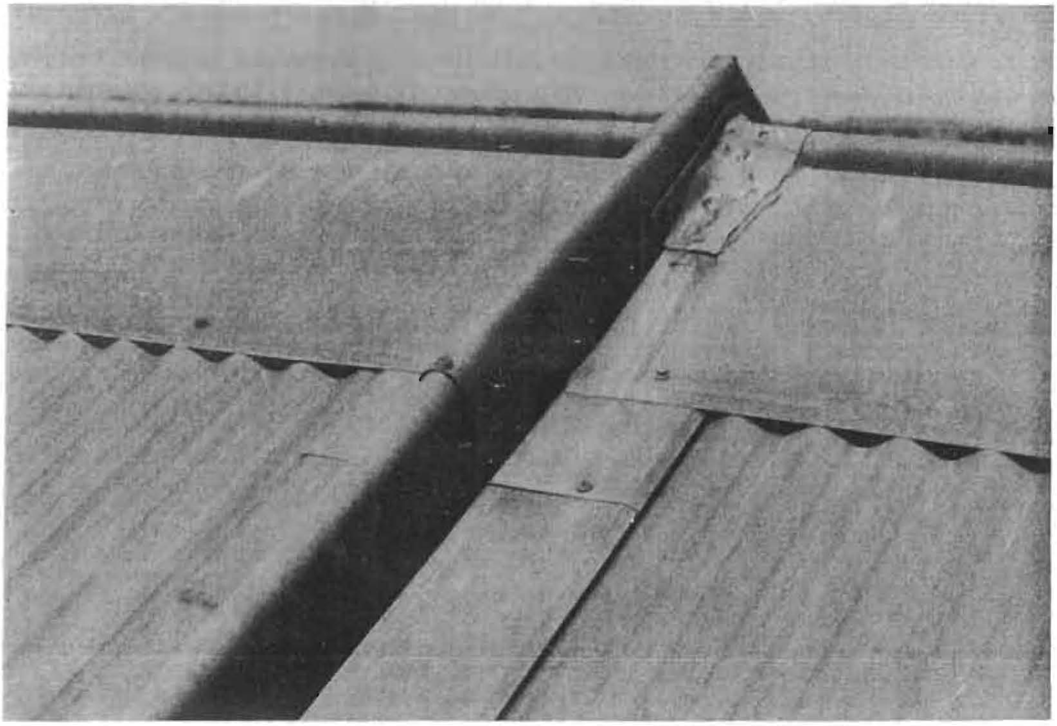

Fig. 7 An expansion join, at the place where it joins the roof ridge.

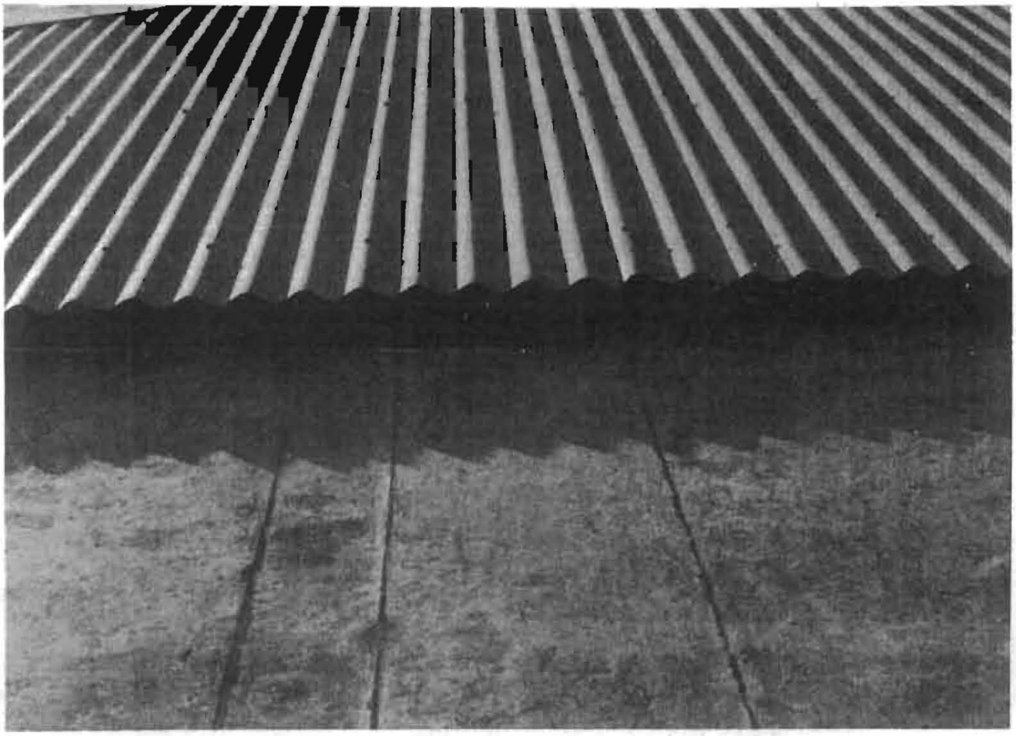

Fig. 8 The region of the gable end where the wall cladding overhangs the top of the concrete wall. Viewed from below.

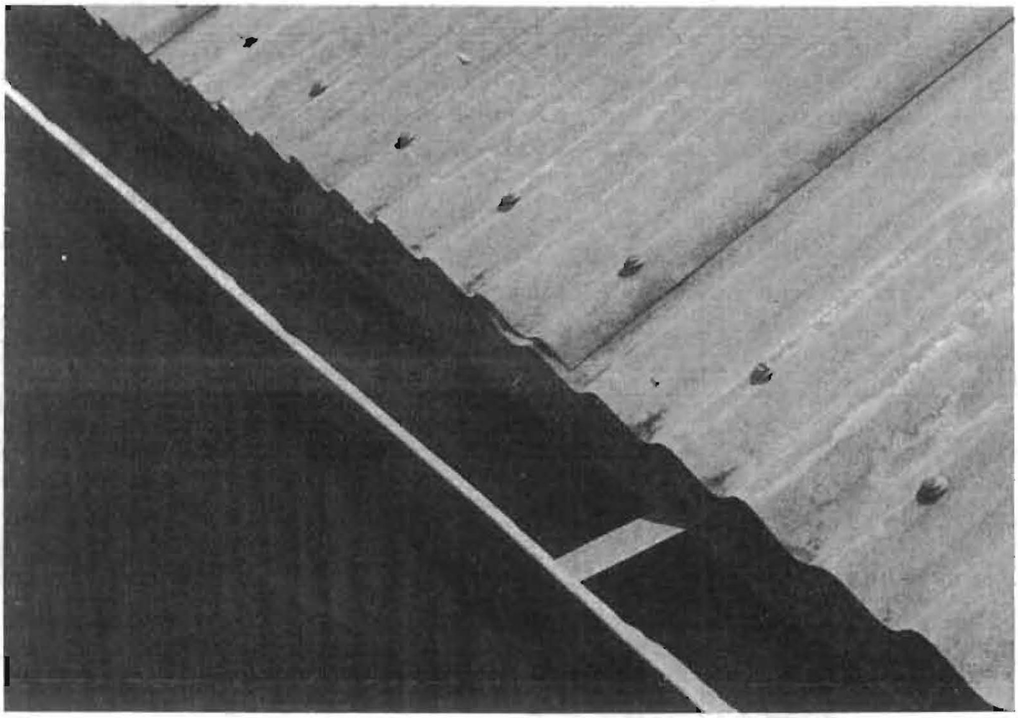

Fig. 9 The lower ends of the roof sheeting at the gutter line.

POLYURETHANE FOAM FOR SEALING GRAIN STORAGES

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ABSTRACT

Polyurethane foam is a useful material for filling voids in the structure of grain storages so that they can easily be sealed by a membrane coating. It can be used as a sealant itself when applied in the full foam system over a complete roof or structure. Details are given of the physical properties of polyurethane foam and how it may be applied to storages.

INTRODUCTION

This paper makes no pretence to account comprehensively for one of the most recent interesting applications for polyurethane foams - sealing of grain storages. The applications of polyurethane foams are practically limitless and this paper is intended only to disseminate some knowledge of practical experience gained in application and to acquaint the reader with a basic technical background on polyurethanes.

CHEMISTRY OF URETHANE FOAMS

The chemistry of urethane foams is very complex. This summary gives a general presentation of the reactions of primary importance.

Polyurethane is the end product of the polyaddition reaction which takes place between a polyol (resin) and an isocyanate resulting in the formation of urethane macromolecules. A blowing agent is added to the reacting mix in order to obtain the cellular structure of a foam. The reaction follows:

$$R - NCO + R'OH \longrightarrow \begin{array}{c} \phantom{R -{}} H \phantom{{}-{}} O \\ \phantom{R -{}} | \phantom{{}-{}} \| \\ R - N - C - OR \end{array} + \text{Heat}$$

R NCO = A polyisocyanate. For production of rigid polyurethane foams the isocyanates proven particularly successful consist of polymers of 4'4 - diphenylmethane di-isocyanate (MDI).

R'OH = A polyhydroxyl compound - polyols, glycols, polyesters. As a general rule the end nature of the polyurethane foam produced is due largely to the character of the polyol selected e.g. flexibility.

Suitable catalysts such as metal compounds or tertiary amines influence the speed of the polyaddition reaction. Besides emulsifiers, polyglycol and silicone polymers are incorporated to control the cell formation.

The reaction of polyol and isocyanate is highly exothermic. If low boiling liquids, (e.g. fluorotrichloromethane) are added to the mix, these are volatilized by the reaction heat which is released and in this way the reaction mix expands.

PROPERTIES OF POLYURETHANE FOAM

Some general physical properties of polyurethane foam are summarised in Table 1.

Thermal Conductivity

The closed cell structure and the presence of fluorotrichloromethane (freon R11) as a blowing agent ensure that urethane foams are unsurpassed with regard to low thermal conductivity.

Chemical Resistance

As a cross linked plastic, polyurethane has good resistance to water, washing liquors, diluted acids and alkalis and aliphatic hydrocarbons.

Temperature Behavior

Under changing temperature, a mechanical stress is exerted on the foam structure of closed cell polyurethane.

With correct choice of material, proper application and appropriate density, polyurethane can be used in a temperature range extending from -200^{o}C to above +100^{o}C.

Mechanical Properties

Excellent dimensional stability, compressive strength and fluexural strength.

The mechanical properties of polyurethane depend to a large extent on density. Generally speaking, linear or very nearly linear graphs of density versus a mechanical property are obtained, except when thermal or chemical effects are introduced (Fig. 1).

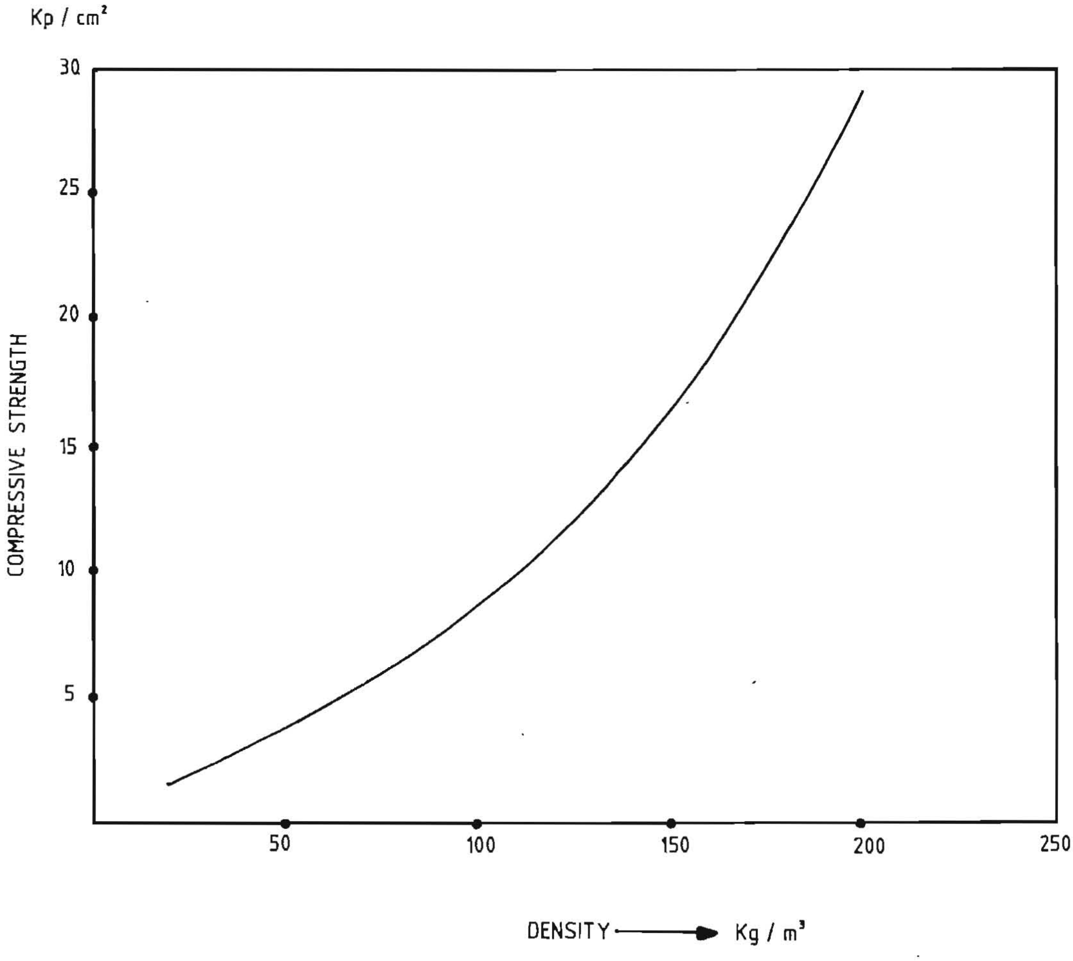
Adhesion

Polyurethane foam produces a strong, permanent bond with many materials.

Table 1 Physical properties of rigid cellular polyurethane (Australian Standard 1366, Part 1 - 1981)

PHYSICAL PROPERTY	UNIT	LIMIT	METHOD
Compressive stress at 10 percent deformation; minimum	kPa		AS2498.3
- Parallel to rise		175	
- Perpendicular to rise		100	
Rate of water vapour transmission; maximum - measured paralled to rise at 38°C	$mg\ m^{-2}s^{-1}$	1300	AS2498.5
Dimensional stability of length; maximum	Percent After 20h		AS2498.6
@ 70°C and 95 ± 5% r.h.		3	
@ -15°C		1	
Closed cell content (uncorrected). Minimum	Percent	85	AS2498.7
Thermal conductivity; maximum (at a mean temperature of 25°C)	$Wm^{-1}K^{-1}$	0.027	AS2122.1
Flame propagation characteristics			
- Median flame duration; maximum	S	8	
- Eighth value; maximum	S	12	
- Media mass retained; minimum	Percent	55	AS2122.1
- Eighth value; minimum	Percent	50	

FIG. 1. COMPRESSIVE STRENGTH OF POLYURETHANE FOAM VARIATION WITH DENSITY (THE USUAL DENSITY FOR SPRAY LIES BETWEEN 35 - 50 Kg m^3. FOR HIGH LOAD BEARING APPLICATION SYSTEMS UP TO 100 Kg m^3 ARE AVAILABLE)



Other properties

Polyurethanes are resistant to rot and, since they have no food value, do not support fungi and do not attract termites, rodents or insects. Being odourless (when cured) polyurethane is suitable for food processing and storage plants.

Table 2 and Fig. 2 give some typical physical properties for a commercial foam.

Table 2. Typical average physical properties of sprayed foam - commercial system

Physical Property	Unit
Cut foam density	45 Kg m^{-3}
% closed cell	94%
Compression at 10%	290 kPa
Water absorption after 7 days	1 vol %
Thermal Conductivity	0.023 W m^{-1} k^{-1}

Dimensional stability: 14 days

	L	W	T	
-30°C	0	0	0	%
+100°C	+0.4	+1.1	+0.3	of
70°C/100% RH	+0.9	+0.4	+0	original

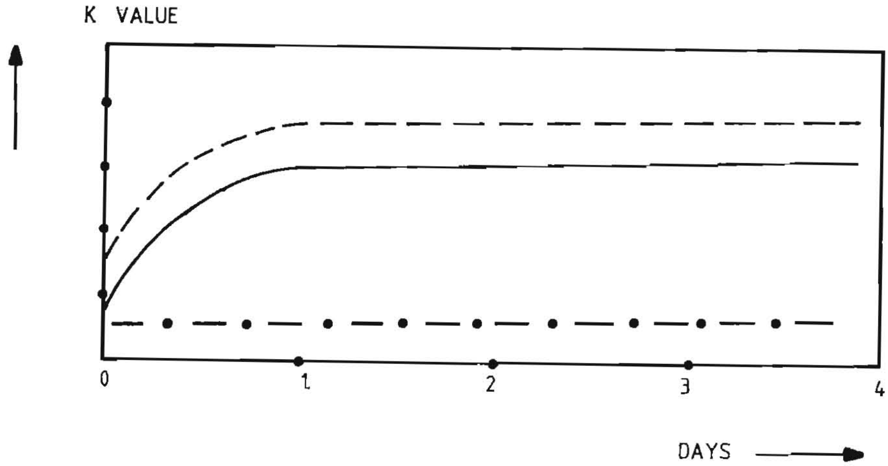


FIG. 2. CHANGE IN THERMAL CONDUCTIVITY WITH TIME FOR POLYURETHANE FOAM
—•—•—•— WITH DIFFUSION - TIGHT FACINGS :
———————— WITH NORMAL SURFACE SKIN
– – – – – – – – WITH CUT SURFACE

APPLICATION - SEALING GRAIN STORAGES

Initially the predominant application for polyurethane foams was insulation. However, polyurethane foam is excellent for filling even the most complex cavities. The reaction mix, which expands 30 - 40 times in volume, can be formulated in such a way that the rising foam will pass through narrow gaps, flow around edges and penetrate into the farthest corners. Combined with the fact that polyurethane can be sprayed or poured, these properties have led to the acceptance of polyurethane as an important component necessary for the process of sealing grain storages.

In practice it should be said that the polyurethane is not used to seal grain storages. It is utilized to fill voids and holes thus providing a suitable surface for the subsequent application of a continuous membrane.

The critical areas of a shed-type or 'horizontal' store to be sealed (filled) prior to membrane application are shown diagramatically in Figs. 3, 4 and 5.

The following areas are treated in the so-called 'partial foaming' system:

Penthouse (Control Room)

The penthouse is generally the weakest structural point and most subject to damage by the environment.

Full foaming is used in practice to achieve:

(a) Void Filling

(b) Minimal temperature/pressure variation

(c) Inherent strengthening of structure.

Ridge Capping (Detail A, Fig. 4)

Polyurethane is used to seal ends of roof sheets and fill voids under ridge capping to prevent build up of grain and provide suitable base for membrane.

Side Wall (Detail B, Fig. 5)

As shown.

End Wall

Sealing of all laps in sheeting and filling gable ends.

Polyurethane is particularly useful for the sealing of old structures. Physical damage, loosened fasteners and general disrepair lead to voids and gaps which cannot be bridged using membranes alone.

In full foam technique the entire roof area and vertical metal cladding are sealed with foam. This full foam technique may be necessary in storages in very bad repair as the foam provides long term structural strength, reducing maintenance costs.

Although in practice the partial foam system is employed for economic reasons it may be that "full foam" technique will be more viable in future. The technical advantages are-:

(a) Minimal variation in temperature within the storages leading to economies in gas usage.

(b) Strengthening of overall construction.

(c) Minimization of expansion/Contraction of the structure.

Insufficient data is currently available to substantiate this concept to say that results obtained from the only fully foamed storage in Western Australia (Burracoppin, 25,000 tonnes capacity) are apparently superior at present to those in which the 'partial foam' system has been used.

EQUIPMENT REQUIRED FOR POLYURETHANE FOAMING

The polyurethane foam is generally applied by the spraying method. Commercially available equipment for spraying urethane foams falls into two categories: low pressure or hydraulic high pressure units. The production of good quality polyurethane foam is highly dependant on efficient mixing of the components. The high pressure equipment is favoured by the majority of applicators as it offers several advantages. These include:

Highly efficient mixing via high pressure counter current injection. Current designs of mixing head (spray gun) are hydraulically controlled, permitting instantaneous shut off. This not only ensures essential automatic clean out of the mix chamber but prevents feed-back into delivery lines.

Extremely accurate metering of component ratios via positive displacement piston pumps or high pressure axial piston pumps.

High pressure units are essentially sealed systems and experience has proven them to be reliable and cost effective. It should be stressed that the application of successful spray urethane systems is no job for amateurs.

PREPARATION OF SUBSTRATE

Sprayed urethane foam can be applied to a wide variety of substrates. This property is one of its most important features. However, it is extremely important that proper adhesion be achieved by correct surface preparation prior to application. All substrates should be clean, dry and free of grease, oil, looses scale or rust. Substrates should be physically tested by scraping to ensure that the area immediately below the surface is of sufficient strength to support the urethane foam once applied. In general, porous surfaces should be sealed and metal surfaces primed.

Surfaces likely to be encountered in grain storages are:

Galvanised Steel

New galvanised steel should be washed with a solvent or acid, then with water and finally primed. Weathered galvanised steel can generally be washed with water and primed.

Whilst polyurethane foam generally does not contribute to corrosion, under some conditions of service it can do so. It is important to be aware that urethane foam is not a vapour barrier. All organic materials will allow some moisture to penetrate to a metal surface, and some additional means of preventing corrosive attack by this moisture should technically be provided. In grain storage application the urethane is protected by the metal sheets on one side and the sealant membrane on the other so, hypothetically, moisture ingress is minimal. It can be readily perceived however that the initial cleanliness of the metal surface and the adhesion of the foam are important factors in reducing the potential for corrosion at the surface.

Concrete

Concrete is one of the most difficult substrates to treat with foam because of the possibility of the presence of excessive moisture. The moisture content should be checked and the suggested maximum content for spraying is 10%. Excessive salt precipitation should also be removed. (e.g. with muriatic acid).

Pre-painted Surfaces

Pre-painted surfaces may not readily accept polyurethane foam because of the smoothness of the surface. Mechanical scoring or abrasive blasting will increase surface area and improve adhesion.

ENVIRONMENTAL CONDITIONS FOR POLYURETHANE FOAMING

Substrate Temperature

Optimum results are generally obtained with substrate temperatures of 20 - 25^{o}C.

Moisture/Humidity/Condensation

Care must be taken whenever the relative humidity rises above 80% as this could adversely effect physical properties, particularly adhesion.

Wind

Wind is generally not of major concern when spraying externally. However, allowance may be necessary for lowering of substrate temperature in calculating curing times.

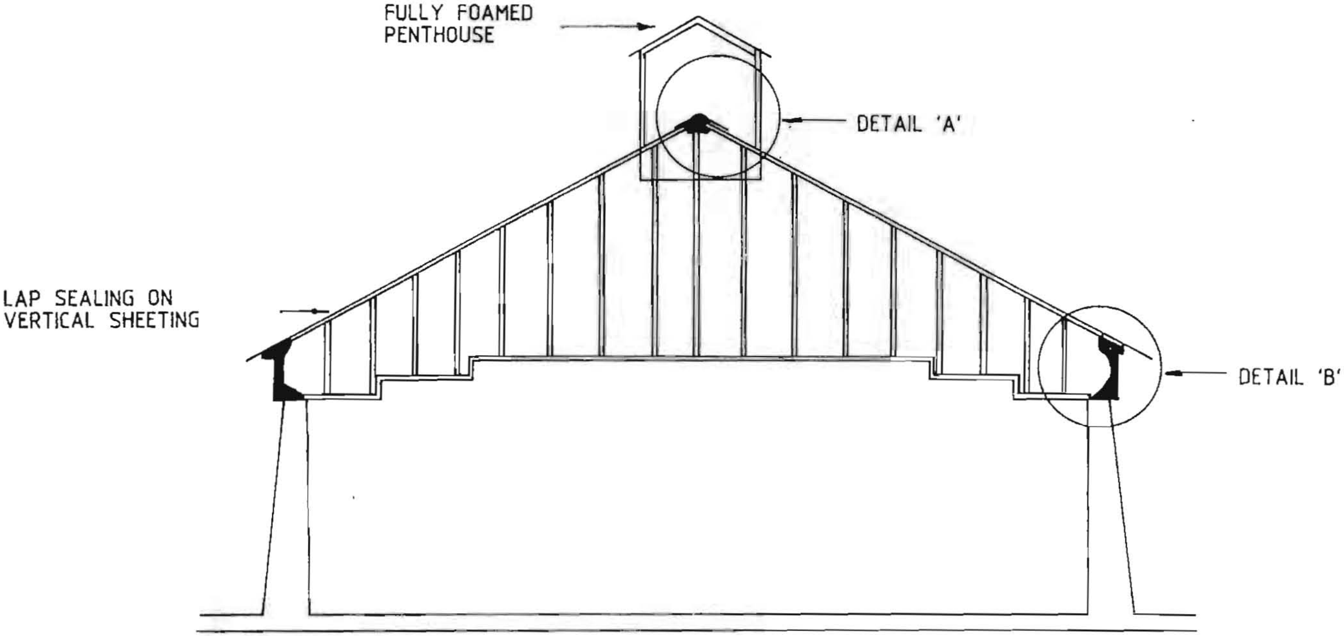
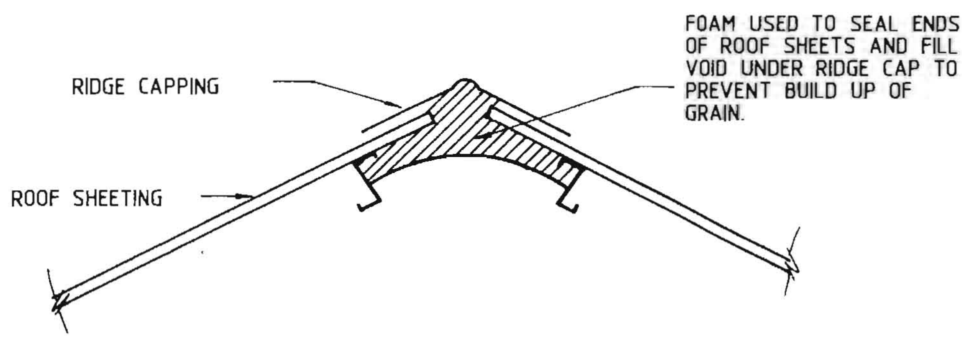


FIG. 3. END ELEVATION OF A TYPICAL HORIZONTAL OR SHED-TYPE STORAGE SHOWING REGIONS WHERE POLYURETHANE FOAM IS USED.



DETAIL 'A'

FIG. 4. DETAIL OF USE OF FOAM POLYURETHANE TO FILL IN VOIDS IN RIDGE CAPPING.

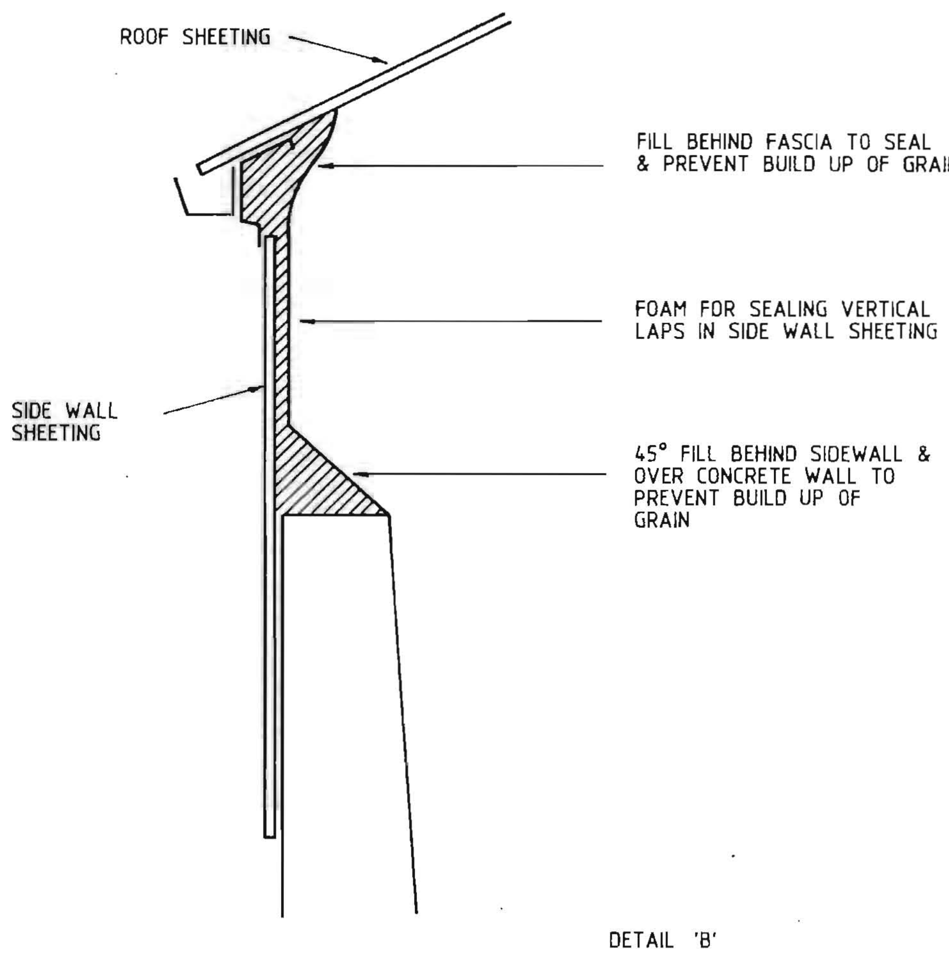


FIG. 5. DETAIL OF USE OF FOAM POLYURETHANE TO FILL WALL - TO - ROOF JOIN IN A STEEL - CLAD HORIZONTAL STORAGE.

SPRAYING TECHNIQUE

It is important that sprayed polyurethane foam be applied in a way that maximizes physical and thermal properties and appearance. This can only be achieved by applicators trained in its proper use and familiar with its limitations.

The spray gun is handled in a similar way to normal paint spraying. The gun is always held perpendicular to the substrate being sprayed. Spraying at an angle can cause elongation of cells in the foam with reduced physical and thermal properties.

At any point where two foam layers meet the edges should be allowed to feather-off as this allows subsequent passes to be blended in giving an even, smooth surface free of ridges and built up sections. A good practice is to allow an overlap of at least 80% on successive passes. If spraying roof areas, cross hatching of successive passes helps in resisting dynamic forces on the foam from the roof.

APPLICATION TIME AND COSTS

Naturally each and every grain storage has different factors influencing time for foam sealing and cost.

If we take a typical 'Warehouse' or 'horizontal' storage of 20,000 tonnes capacity, and discount variables such as location, state of repair, weather conditions etc, it can be assumed that an efficient contractor would complete a 'partial foam' sealing in one week. The average cost is currently around $10,000 to $12,000 for 'partial foam' sealing, i.e. 50 - 60¢ per tonne capacity of grain. The contracting team generally consists of 2 men and cost indicated includes all provisions including travelling, accommodation and meals.

In contrast 'full foam' sealing would require two teams, (4 men), and take around 3 weeks to complete at an indicative cost of $3.50 per tonne capacity. Subsequently, membrane application costs would be somewhat lower. However, overall a 'full foam' system would be 25 - 30% higher in cost. As previously stated, long term data regarding likely cost effectiveness of 'full foam' vs 'partial foam' techniques is not available.

SAFETY PRECAUTIONS

All persons concerned with the handling of isocyanates and isocyanate-containing products must be conversant with the associated hazards and trained in the recommended normal and emergency handling procedures.

The "Threshold Limit Value" or TLV is currently set at 0.02 p.p.m. (0.14mg/m^3) isocyanate vapour in air.

The hazard from vapour and airborne droplets (aerosols) is increased when spraying polyurethane foam. The vapour and aerosols will irritate the eyes as well as the mucous membranes of the nose, throat and lungs. Inhalation must be avoided.

Standard on-site practice should include:

(a) Efficient ventilation. Applicators must be provided with efficient respirators, preferably breathing apparatus with independant air supply.

(b) Overspray should be limited to ensure clean, efficient working conditions and to prevent the spread of material in a hazardous form.

(c) Safety goggles, rubber gloves and protective clothing must be worn.

(d) Suitable eyewash liquid should be near at hand.

(e) Prohibit smoking and eating in proximity to foaming.

(f) Store liquid chemicals out of direct sunlight in well ventilated areas.

Detailed information relative to standard safety precautions is readily available from suppliers of raw materials.

Precautions Against Fire

Like most organic materials, polyurethane foam is combustible. Therefore all safety practices specified against fire hazard during and subsequent to installation must be followed. Manufacturer's and applicator's specifications and codes of practice should be checked and strictly adhered to.

CONCLUSION

Due to the unique properties of polyurethane foam, it is an ideal material to facilitate efficient sealing of grain storages. Full foam sealing may prove to have significant advantages in future, particularly as the excellent insulation value of urethane foams could facilitate pest control via temperature control, such as with the use of refrigerated aeration systems.

ACKNOWLEDGEMENT

I wish to acknowledge the co-operation of Bains Harding Surfacings Pty. Ltd. and Woodkon International in providing information about their experience in sealing grain storages.

EFFECTIVE SEALANTS FOR EXISTING STORAGES FROM FLOOR TO ROOF

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ABSTRACT

Complete sealing has to take into account the main elements of existing storages, e.g. concrete floors, connections to vertical walls, vertical walls with cracks and/or joints, connections to flat or angular roof and roofs with or without insulation. Apart from general requirements an external sealing material has to meet the following specifications: gastight sealing with crack bridging properties, elastic filling of joints and cracks, heat reflection, long lasting UV-resistance and, for metal surfaces, protection against corrosion.

An internal sealing material has to comply with similar requirements plus smoothing of surfaces, formation of abrasion resistant layers, compatibility with foodstuffs, and stability to fumigants. Systems of sealants have been developed which effectively meet these requirements.

As external sealants (walls and roofs) - acrylic resin dispersion as primer - if necessary with rust preventing agents - with subsequent white heat reflecting acrylic coating (eg Wastolan-B white).

As internal sealants - floor - flexible polymer concrete; joins in floor and walls - Acrylic mortar with cement (e.g. Barument); walls - bituminous primer (e.g. Eubit-Plast) plus polychloroprene coating (e.g. Wastolan-A).

These systems (excepting Barument) have proven their effectiveness on grain storages for 4 years in internal application and 2 years in external application in Western Australia. They have also proved successful in the farm silo sealing programme in Western Australia.

1. THE PURPOSE OF SEALANTS AND COATINGS

There are three reasons why sealants and coatings are applied:

a) because of beauty and to make buildings look nicer,

b) to protect buildings against attacks from light, atmosphere, water, chemicals and mechanical treatment by use,

c) to avoid escaping gases and liquids out of storage or to protect materials which are stored in sealed rooms.

In our case the most interest is given to point c) but also point b) is rather important. Of course it is nice to have materials which give satisfaction in these matters and which have a good appearance as well, but beauty is not treated here.

2. BUILD-UP OF SEALANTS AND COATINGS

Three components of sealant are important: the base, the primer and of course the sealant itself.

Mostly the base is not mentioned when sealing is discussed. However the success of the substrate is very important to a sealing system. A coating never can be better than the base to which it must adhere. Defects found in the substrate include porosity, roughness, excessive smoothness, dustiness, coverage with old paints or dirt of all sorts and dampness.

The primer has various tasks. In many cases it only has to overcome defective substrates. A sealant can often be used without a primer when the base is in good condition. However this is rare. Generally, the primer has to bind dust, close pores, increase cohesion in the base and absorb all kinds of dirt. In some cases the primer must be a true adhesive when the coating itself does not stick well to the base. Because of all these reasons the primer has to meet some special specifications. In particular, it must penetrate and it must adhere both to the base and the top sealant coat. Furthermore, a top coat must never be harder than a lower layer. So the primer should not be softer than the sealant. This requirement is sometimes difficult to meet as the harder a primer is the less adhesive it is. A primer mostly has no sealing function itself, but it only prepares the substrate for the sealant.

The top coat or sealant has quite a different task to the primer. As its name implies, it coats or seals. That means it must possess all the properties needed to meet the requirements described in Section 1. To fulfil this there must be a satisfactory method to apply the sealant. In particular, bubble-holes, cracks or gaps in coating must be avoided. Furthermore it is necessary to ensure the thickness of the layer is at least as much as it is wanted all over the treated surface. A little instrument was developed which can measure thickness of wet films between 25 microns and 2 mm in small steps. The instrument is called a 'comb' (Fig. 1).

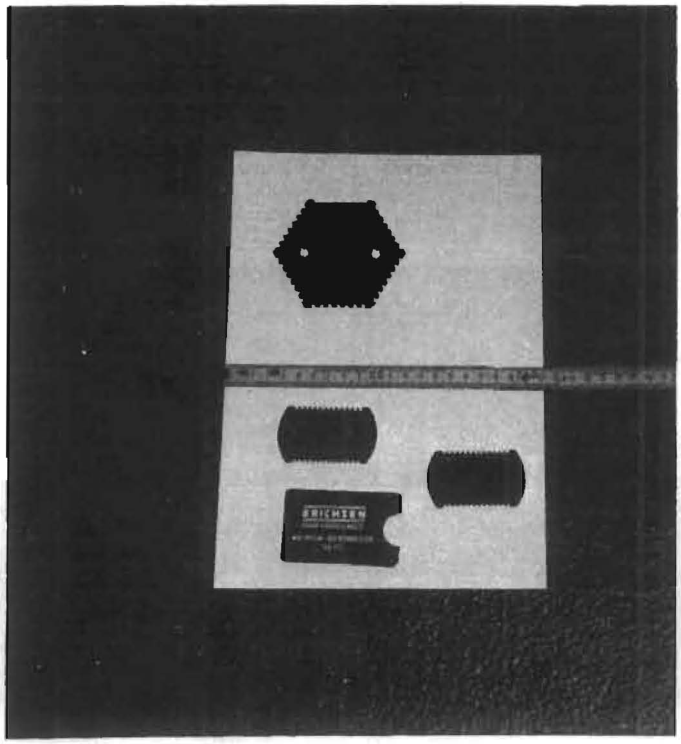

Fig. 1 Some 'combs' for measuring the thickness of wet layers.

The wet layer must usually be much thicker than the dry layer. If solvent or something else evaporates, there is a volume contraction and the layer thickness decreases. So the solids content by volume must be known when the material is applied so that allowance can be made for solvent loss when the expected dry film thickness is calculated.

If the material contracts during drying, there is a danger that cracking may occur. This danger differs from one product to the other and the producer must specify the upper limit for wet layer thickness that can be applied in one operation. Mostly two coats of material are necessary to reach the required dry layer thickness. If two coats are used it is more likely that the coverage will be uniform and gastight, as it is very improbable that two defective spots coincide. However, today more and more products are applied in one operation to save money. This needs operators who are very skilled in their jobs.

3. METHODS OF APPLICATION

There are three different kinds of application technique:

a) Manual, i.e. brushing, rolling or trowel application.
b) By sprayers either air-entrained or by airless spraying.
c) Bridging over cracks, joints and connections using a 'fleece' for reinforcement.

Fig. 2 A fleece being applied to a construction join precoated with Wastolan. The Wastolan penetrates the fleece giving a reinforced cornice over the join.

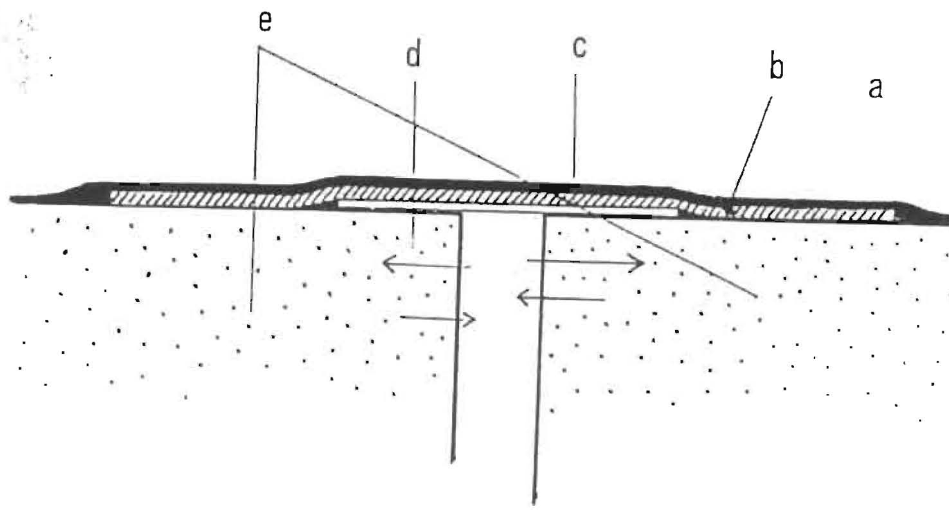

Fig. 3 A sealing of a joint a = sealant, b = penetrated fleece, c = loose foil, d = movement of the joint, e = concrete. The loose foil gives a much wider stretch area for the sealing.

Application of coating's by roller or brush are well known techniques. However, there is a great difference between the two methods. If a material is brushed on, it is really rubbed into all unevenesses of the treated surface (eg. holes, cracks, pores). The material gets a very good grip on the surface. Furthermore, residual dust and loose stuff are rubbed into the sealant material. On the other hand, if a roller is used for application, the material is only laid on the surface and it must adhere by its own power. The minimum requirement for this is good penetration of the substrate. This is dependant on the viscosity of the coating. In most cases, the roller technique needs a special primer for optimal adhesion. Trowel application can give good results, because the material is pressed into all unevennesses of the substrate. However, the traces of the trowel very often are obvious and the thickness of the layer can vary considerably.

Spraying techniques require specialised equipment. With simple air-assisted spraying, air is pumped at 4-6 bar into the material to be applied, forcing it out of a gun as a fine spray. This kind of spraying is sometimes called the 'low pressure technique'. With airless spraying, a much higher pressure is required at the gun (eg. 300 bar). The material is released from the spraying nozzle at high speed.

Fig. 4 Wastolan Red is sprayed with air in the low pressure method to penetrate a fleece for tightening a pit.

Both spraying methods are very economical. Large areas can be coated in a short time with very little manpower. However, a compressor must be available which not only gives enough pressure but also enough air volume. Furthermore, the airless method can be used only if the material is absolutely free of foreign bodies. If there are lumps in the liquid which plug the nozzle the spraying angle is altered and the thickness of the applied layer becomes very uneven. Thus liquid materials to be sprayed must be manufactured under very clean conditions.

The spraying techniques have other advantages over manual application. The material is sprayed into all unevenesses of the surface, particularly if the high pressure method is used. All dust and loose stuff on the surface is blown off. The airless method does not produce air pockets and foam in the applied layer because the drops are very small and meet the surface at a very high speed. If bubbles and holes do appear, they always result from pores in the substrate (Fig. 5).

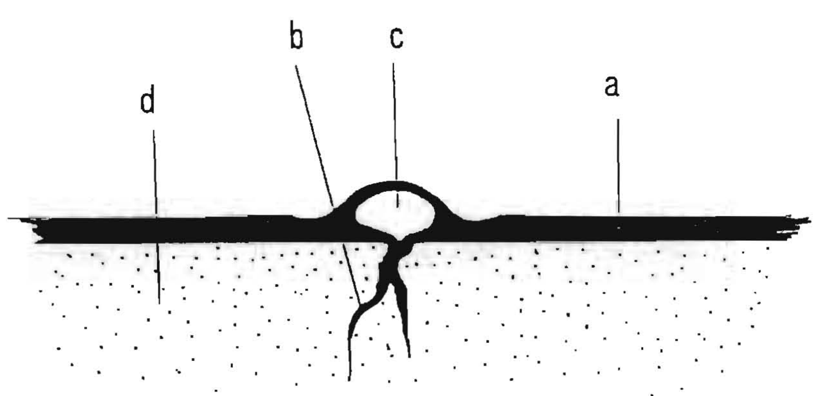

Fig. 5 A bubble develops in the sealant where it penetrates into a pore and removes the air, a = sealant, b = pore, c = bubble, d = concrete.

Using fleece as a membrane reinforcement is a special technique. It can only be applied manually, (Fig. 3) but is always necessary when the building to be sealed has special critical points, such as joints, connections or cracks that must be bridged over. The sealing material itself is normally not able to withstand the movement in these regions. There is a rule for bridging over joints or cracks: if a slit is to be bridged by an elastic or plastic material, the thickness of the layer must be at least three times the width of the slit (Fig. 6).

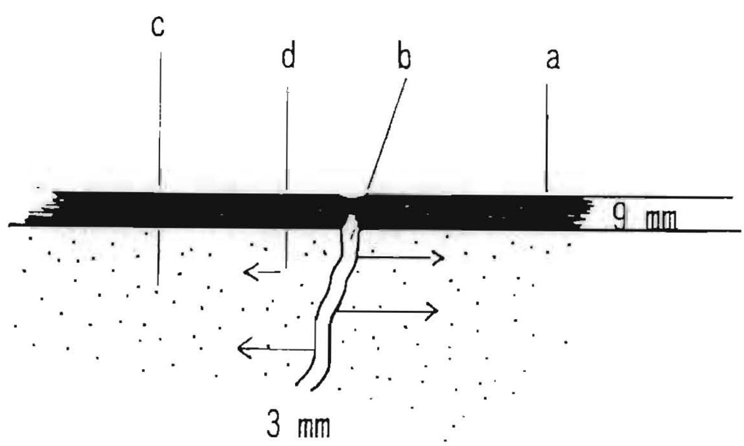

Fig. 6 A crack has formed suddenly and elongates the sealant. On the bottom it is streched from zero and begins to tear. a = sealant, b = crack, c = concrete, d = movement. The sealant thickness should be more than 3 x the crack width to ensure that the seal does not fail.

Fleeces for reinforcing membranes must have special characteristics: they must be elastic and as isotropic as possible, because they must withstand movement and stretch in all directions. The liquid sealing material must totally penetrate the fleece and it must be completely free of tension. The latter feature allows the fleece to be laid on all bends, hollows etc. without loosening as long as the sealant has not yet hardened.

When applying a fleece, the surface is first coated with material. Then the fleece is rolled into the wet sealant starting from one end to the other of the fleece, so that folds and turns are avoided. The roller enables the liquid to penetrate the fleece without tension. After the fleece has been penetrated totally, a new layer of sealant is applied by roller or spray.

If the surface to be treated is in poor condition, the fleece must be taken over the entire area. This gives much more stability in the sealant and is always advisable where a sealant has to withstand pressure. Furthermore use of a fleece guarantees a certain thickness of the sealant as the layer cannot be made thinner than the penetrated fleece itself.

When a large crack is to be sealed, it is useful to put a loose cover over it before applying the fleece and sealant (Fig. 6). This spreads the tension resulting from movement of the crack over a larger area of sealant, reducing the posibility of failure.

4. THE WASTOLAN SYSTEM

Some general aspects

In water-based media, insoluble binders can only be emulsified or dispersed. This means that a polymer forms very small drops in water that are prevented from agglomerating by giving them a charge or a protective coat. But as small as the drops may be, the whole mixture always remains a two phase system. The drop size of polymer dispersions normally is between 0.1 and 1.0 micron.

The following figures (Figs. 7, 8 and 9) show the setting mechanism of dispersions of various types:

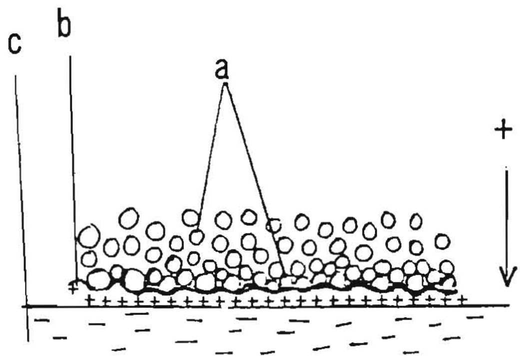

Fig. 7 Mechanism of setting in a cationic emulsion. The cationic emulsifier adheres to the anionic base. a = cationic drops of the emulsion, b = the first cationic film, c = concrete.

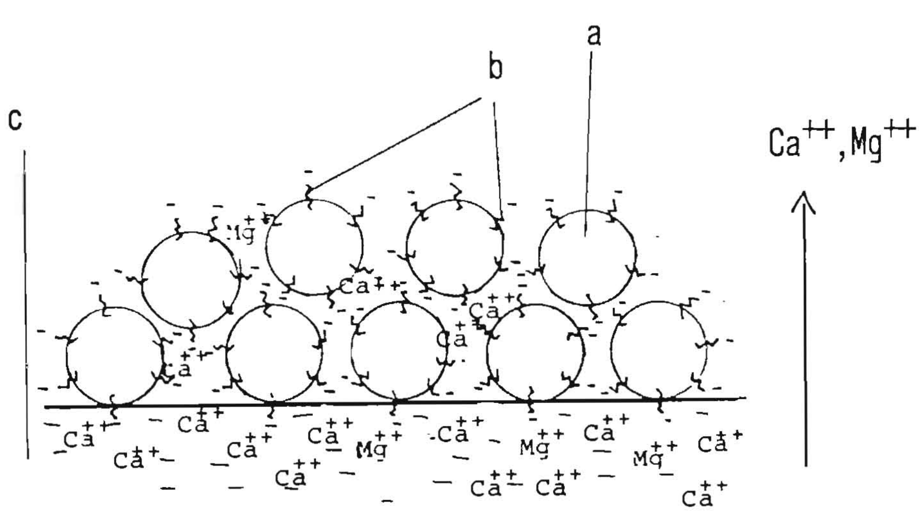

Fig. 8 Mechanism of setting in an anionic emulsion. Ca^{++} and Mg^{++} ions are dissolved out of the base and form an insoluble compound with the emulsifier. a = anionic drops of the emulsion, b = anionic emulsifier, c = concrete.

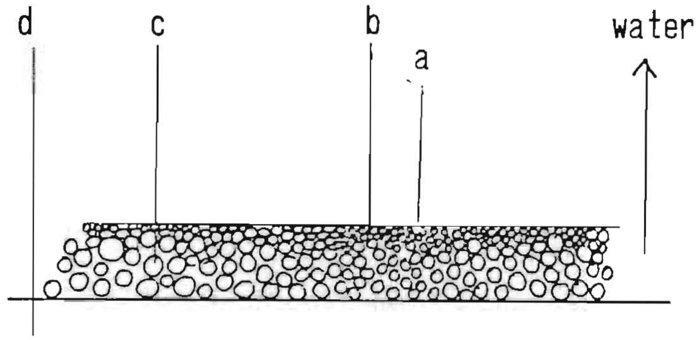

Fig. 9 Mechanism of setting in nonionic emulsion. Water evaporates and forms a skin after the protective layers around the drops have evaporated. a = skin, b = drops with dried protective layer, c = original nonionic drops of the emulsion, d = concrete.

Cationic emulsions normally adhere by exchanging their charge with the substrate which carries anionic charges. Anionic emulsions must react with the substrate. This can only happen if metal ions are set free that react with the emulsifier-soap to give water-insoluble compounds. both types form films and sealants which are highly resistant to water swelling because the emulsifiers are destroyed. Nonionic emulsions, which are stabilised by a protective nonionic coat around the polymer drops, are very stable indeed but they only dry and do not react to give a sealant. So the emulsifier stays unchanged in the sealant and can be reactivated if water treats it. Only very few emulsifiers of this type can be used without any risk, but nonionic emulsifiers are often preferred because they give more stability in manufacture and application of the sealant.

Wastolan Primers

In contrast to dissolved (single phase) primers, emulsified primers have difficulty in penetrating into the pores of the substrate. The surface forms a sort of sieve and the droplets of the emulsion are left on the surface, while the water is sucked into the substrate (Fig. 10). Therefore, an emulsion primer must contain emough water so that it does not stick to the brush or roller while being applied. If it dries too fast it does not have time to agglomerate to form a film.

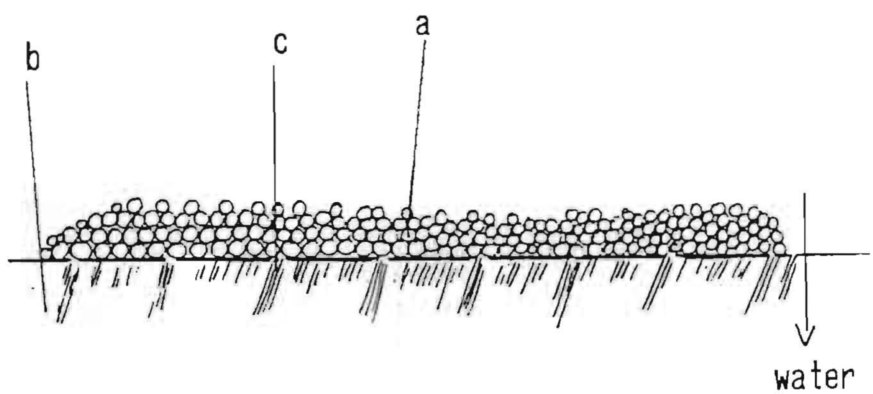

Fig. 10 Sieve-effect of concrete. The water is sucked out of an applied emulsion and the dispersed phase stays on top. a = emulsion drops, b = concrete, c = water sucked into the concrete.

Because of being left on the surface, an emulsion primer is well suited to seal pores and holes. This is especially important for a material used as a sealant. Of course the primer has to ensure good adhesion of the top coat as well. For these reasons, Wastolan Primer is made from very special materials: bitumen/neoprene/acrylic resin. Wastolan Primer shows the following properties: good pore sealing, very good adhesion, extremely gastight, good strength for carrying the top sealant and very tacky. It can be applied very easily by all methods described in Section 3, although best by brushing or spraying. The colour of the primer is black only because of the bitumen content. This black primer is rather thixotropic, so the consumption is about 0.5 kg/m^2, but depending on the roughness of the substrate.

The surface to be treated has to be very clean and solid enough to carry the whole sealant. If the surface is too weak the total sealant may loosen (Fig. 11). Technical data on Wastolan Primer is given in Table 1.

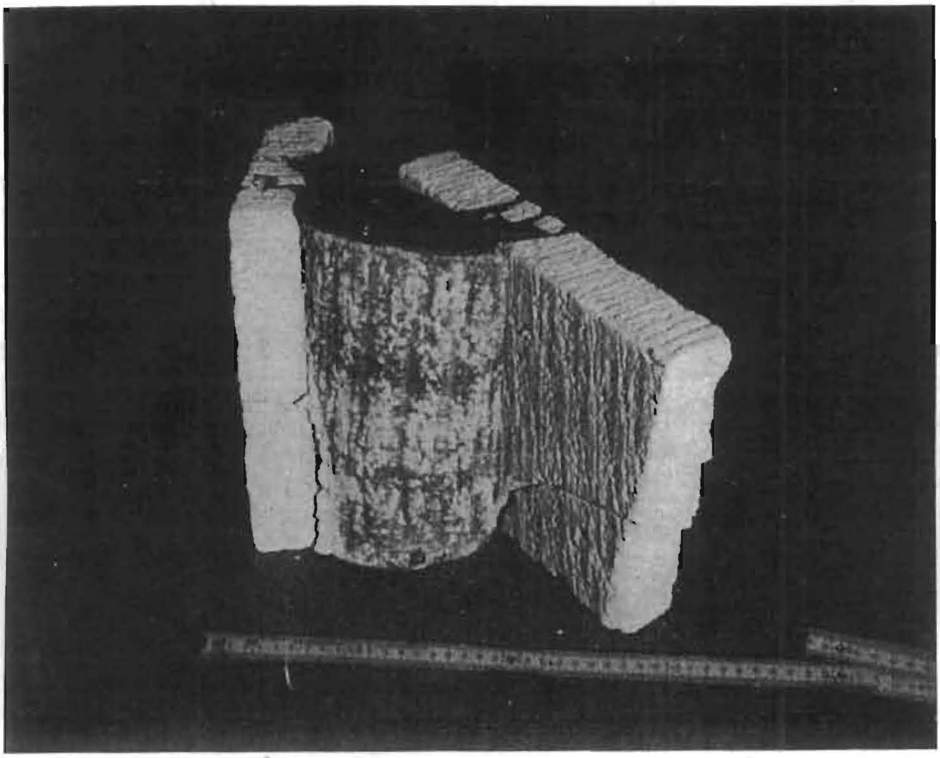

Fig. 11 The Wastolan sealant was removed from a weak surface. The loosened concrete still adheres well to the Wastolan.

Table 1. Technical data for Wastolan Primer.

Density:	1000 kg/m^3
Solids content by weigh:	60 %
Solids content by volume:	60 %
Charge of the emulsion:	anionic
Characteristic of dried material:	
Resistance to heat (DIN 52123)	100°C
Resistance to cold (DIN 52123)	-10°C
Waterproofness, 2.5 mm:	2 bar
Elongation at break depending on thickness:	300 - 400 %

There is also another primer available for Wastolan. It is also an emulsion but containing acrylic resin and being free from bitumen and pigments it dries rather colourless, is much harder than the other primer and is not thixotropic. It is thus an adhesive primer only without sealing properties itself (see Table 2).

Table 2. Technical data for the acrylic-based primer for Wastolan.

Density:	1000 kg/m^2
Solids content by weight:	38 %
Solids content by volume:	38 %
Charge of the emulsion	anionic
Cleaning agent, wet:	water
Cleaning agent, dry:	butyldiglycolacetate
Consumption:	200 - 300 g/m^2
Drying:	1 - 3 hours dependent on weather conditions

Wastolan Top Sealant

This material is based on an emulsion of acrylic resin. It is solvent-free and filled with china clay mineral powder and pigments. The acrylic resin forms a highly elastic sealant film which is able to bridge over cracks. The sealant has a very good resistance to UV-light and keeps its white colour for many years. Of course it may happen that dirt and dust darkens the surface, but this is not unusual.

Wastolan can be used to seal all kinds of buildings against water, humidity or escape of gases through porous walls. It is particularly suited for sealing grain storages for controlled atmospheres and fumigation.

Wastolan can also be used in sealing of pits (Fig. 12). The technique differs quite a lot from that used for sealing walls. Here a big sheet of fleece is put on the bottom of the pit in the soil as evenly as possible. Then the fleece is impregnated with the Wastolan material (thinned a little with water) to give a very strong, elastic membrane. After the impregnation, a second coating is usually applied to guarantee full sealing and give a smooth surface. The whole application can only be done by spray. Air-assisted spraying is most suitable.

Fig. 12 A pit sealed with Wastolan. It is supposed to be impervious to sewage to avoid seepage and pollution of the soil.

For sealing of pits and all other kinds of sealing there is also a second type of Wastolan based on polychloroprene rubber (Neoprene).

Wastolan is available in different colours: red, green, grey and white. Of course the white one reflects light best, so it should always be used where maximum reflection of light is required, especially if heating by the sun is to be avoided. The white colour is favoured in sealing of grain silos to avoid this heating.

Wastolan layers are very tough and elastic after full drying. But at critical points a fleece should be laid in to increase the strength of bridging over all sorts of slits. It also is able to bridge over cracks in *"Statu nascendi"*. In this situation a crack may appear very suddenly. Only if it is very tough and elastic can the sealant still bridge over the crack and only if it is thick enough (Fig. 6).

Technical data for Wastolan sealants are given in Table 3.

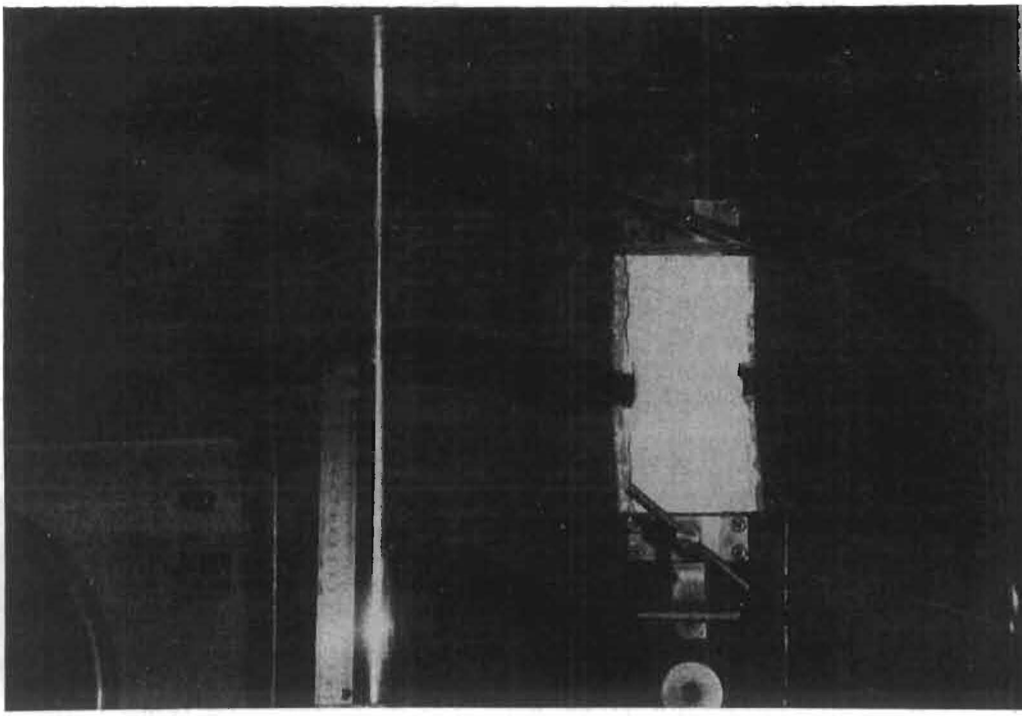

Fig. 13 Wastolan reinforced with fleece under tension. The crack has been opened from 0 to 40 mm without failure of the sealant. The sealant has lost adhesion to the substrate from 20 - 40 mm from the slit.

Table 3 Technical data for Wastolan sealants.

	Wastolan White acrylic resin	Wastolan polychloroprene
Number of components:	1	2 (100 : 30)
Potlife:	-	2 hours, 20°C
Density:	1300 kg/m^3	1250 kg/m^3
by weight: (DIN 53219)	70 %	
Solids content by volume:	60 %	50 %
pH-value:	8 - 9	10
Charge of the emulsion:	nonionic	anionic
Temperature of film-forming:	5°C	4°C
Resistance to heat (DIN 52123)	+ 130°C passed	+ 130°C passed
Resistance to cold (DIN 52123)	- 15°C passed	- 35°C passed
Elongation at break (DIN 53571) (without fleece, 1.5 mm thick film)	250 %	650 %
Maximum thickness of layer applied in one operation	1.5 kg/M^2 1150 µm wet 700 µm dry	1.0 kg/m^2 800 µm wet 400 µm dry
Conditions for airless spraying (without fleece)	5 bar, nozzle width 0.79 mm 1:45, initial pressure	5 bar, nozzle width 1.09 mm 1:60, initial pressure
Spraying with air compressor for 6 bar (1200 L air/min)	5 - 6 bar, nozzle width 1.02 mm	5 - 6 bar, nozzle width 1.02 mm
Resistance to UV-light	very good, no change of colour	good but change of colour
H_2O-vapor permeability	3.2 g/m^2 . d 1.5 mm film	appr. 0.4 g/m^2 . d 2.5 mm film
Cleaning agents		
Wet emulsion:	water	soft water
dry polymer:	butyldiglycolacetate, ethylglycolacetate	aromatic oils

Wastolan Fleeces

The main features of fleeces were described above (Section 3). It must be emphasized that the fleeces have to be totally isotropic and very elastic or otherwise they do not give the necessary elastic quality to the Wastolan to resist all forces caused by movement of the building in all directions. Furthermore the fleece must be very soft so that it fits in all unevenesses of the surface. The normal weight of the fleece is approximately 150 g/m^2 and it is available in rolls of 300 m length and 420 cm width.

Barument

Barument is a mortar made with acrylic resin despersion instead of water. The mortar is made by mixing 1 part by volume of cement and 2 parts by volume of dry sand. 1 part by volume of Barument emulsion is added and the ingredients are mixed until a pasty mortar is formed. Then water is added very carefully in small amounts. The viscosity of the mortar is very sensitive to the quantity of water added (Fig. 15) There is less danger of cracking the less water is added. The mortar gets stiff rather fast and dries quickly. A certain volume concentration is unavoidable. Though rather highly viscous, the mortar flows into all holes, joints and cracks. Before it is applied into cavities these must be treated with a primer (1 part by volume of Barument dispersion with 2 parts by volume of water). The adhesion is so good that under strain the material cracks in itself rather than loosen from concrete. After drying the mortar produces a very elastic (Fig. 14) and hydrophobic material.

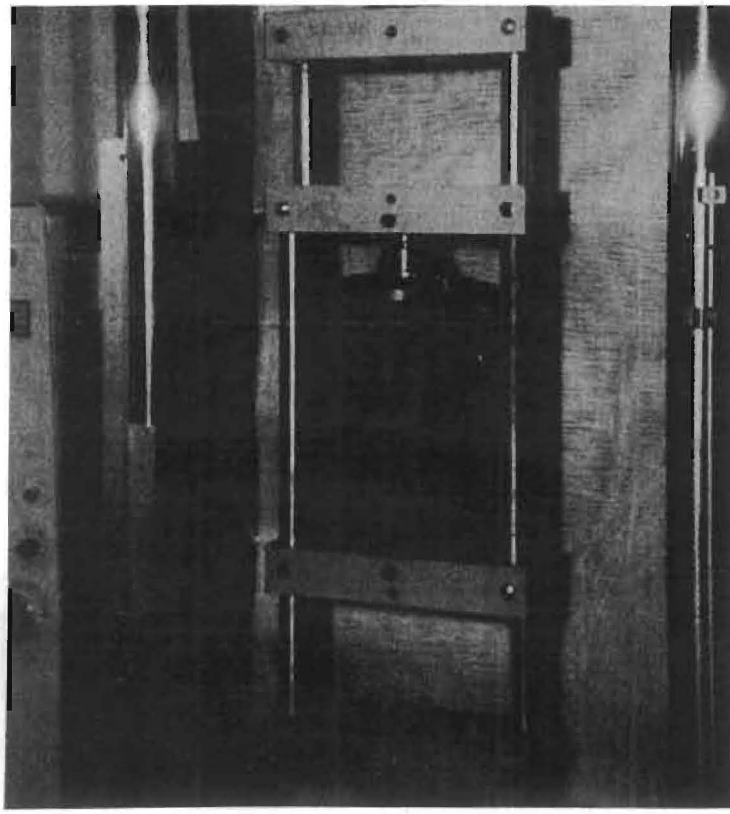

Fig. 14 A sample (4 x 4 x 16 cm) of Barument is tested under DIN 1164. It is obvious how much it can be bent without breaking.

Being an acrylic resin dispersion Barument is compatible with the Wastolan System. It is highly elastic, has good adhesion and is a very good sealant. Technical data for Barument is given in Table 4.

Table 4 Technical data for Barument.

Density:		appr. 1.04 kg/m^3
Charge:		nonionic
pH-value:		7.5 - 9.0
Solid content	by weight by volume	56 m% 54 %
Viscosity:		200 - 350 mPa's
Temperature of film-footing		0°C
Colour of film:		transparant, colourless
Density of dry mortar, 28 days old:		1900 - 2000 kg/m^3
for the following test 10 % water is added to the mortar		
	bending strength	after 7 days: 1.8 N/mm^2 after 28 days: 6.9 N/mm^2
	crushing strength	after 7 days: 3.6 N/mm^2 after 28 days: 13.0 N/mm^2

Samples were so elastic that the values are difficult to measure.

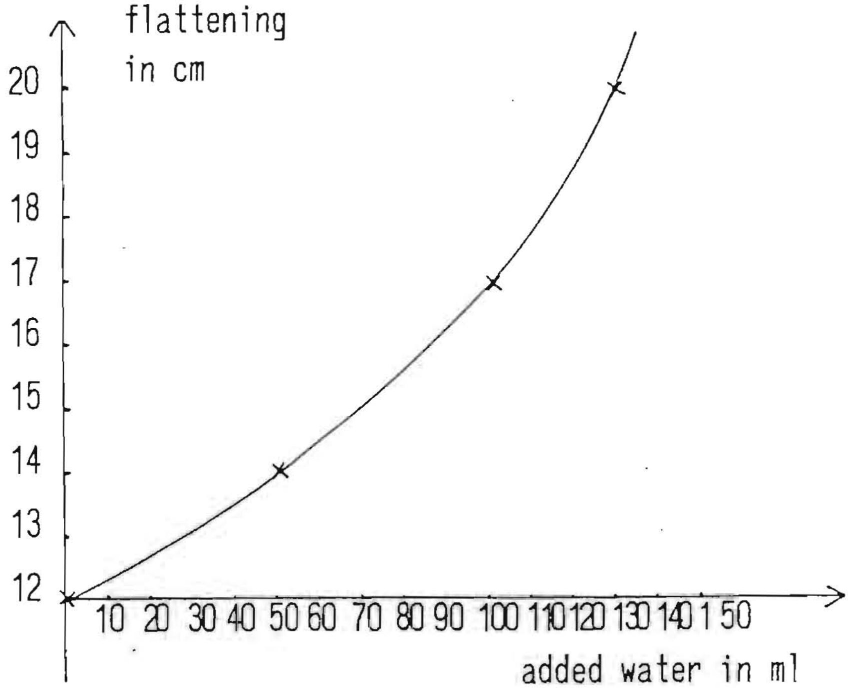

Fig. 15 Barument Mortar becomes the lower viscous the more water is added. The consistency is measured by a compression test (DIN 1060).

5. REQUIREMENTS AND TEST METHODS OF SEALANTS FOR GRAIN STORAGE

There are three different aspects of testing and proving sealant materials:

a) Conditions and features of application and storage
b) The physical properties relating to sealing and resistance against all sorts of influences and ageing.
c) The requirements that the sealants be safe for use in contact with foodstuffs.

For the first two items there are many national standard methods (eg. ASTM, AS, DIN, FTM, BS).

The test methods used here are mostly based on DIN standards because Wastolan is a German development. However, both types of Wastolan were tested by CBH in Australia on the basis of Australian Standards (AS). The standards used by many other nations are similar.

It is much more difficult to set proper standards for the use of polymers in sealing materials which have contact with foods. Of course there are several nations which have strict regulations and laws in this regard, but they often differ a lot and are more or less severe. In addition, copies of the regulations are difficult to obtain. Here only German requirements can be cited. These were published by the "Kunststoff-Kommission des Bundesgesundheitsamtes" (Plastics Committee of the German Federal Health Office). Polychloroprene and acrylic resins may be readily used if they are fully cured. Wastolan, moreover, does not contain any agents which are contrary to these German rules. So no influence is to be expected on the grain by the sealant Wastolan.

SILO SEALING WITH ENVELON

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ABSTRACT

The paper describes the development of ENVELON Polymeric Vinyl Membrane specifically for gas sealing bulk storages used for wheat storage and describes in practical terms the qualities required for sealing coats and the way in which they should be applied. Envelon was introduced to Australia in 1940 and was used extensively for mothballing of naval and army equipment after the Second World War. It was later used for sealing fruit storage cold rooms and investigations into its use for sealing grain stores was begun in 1977. It is now used extensively, on both very large and relatively small farm stores.

When using sealants it is important to select the most appropriate sealant system for each particular application and to prepare precise specifications for both the use and application of the coating and membrane system. Flexibility, strength, ability to bridge gaps, ease of application, weather resistance and impermeability are considered to be essential features of successful sealants. Guidance is given on applying sealant coats, with particular reference to Envelon, in particular the importance is stressed of not applying sealants under ambient conditions when condensation on the substrate is likely to occur.

Technical details of Envelon are given in the appendices, as are duties of inspectors during the application of sealant coatings and a glossary of terms used in describing coatings and corrosion.

INTRODUCTION

Thirty years ago the principal reason for industrial painting was to improve aesthetic appearance. Long term protection was considered as incidental. Today there is a change in this approach with long term sealing and protection being equal in importance with appearance and environmental considerations.

This change has been brought about by the customer being more conscientious and critical of the quality of the coating and the price he pays. A few years ago the appearance of a coal tar epoxy coating on wharf piling was not really important provided the film thickness was to specification; the coating on a bridge was intended primarily to prevent corrosion, not to enhance its appearance. Today the appearance of the bridge and the wharf is almost as important as the performance of the coating system. A storage structure must not only be well sealed and be aesthetically acceptable, but must also remain so for a reasonable period of time.

During the last few years membrane coating systems for use in controlled atmosphere stores have advanced dramatically. However, in despite of the development of modern sealing systems, there still remain four basic areas requiring careful attention when considering the sealing and protection of bulk storage structures:

1. Selection of the appropriate coating or sealant system for the particular situation and conditions.
2. Preparation of precise specifications both for the use and application of the coating and membrane system, and a clear understanding by all parties of what the specification entails.
3. Inspection to ensure the specification is correctly adhered to.
4. Protection of the membrane and its associated paint systems maintained during its entire service life by periodic inspection and spot repair.

A further important consideration is the proper preparation of the substrate for protective coatings. If this important step is ignored the whole operation is likely to be a waste of time and money. The measure of a coating's adhesion to the substrate is an important criterium in assessing its ultimate durability and performance. A protective coating, unless sufficiently thick to form a self-supporting structure, must be uniformly bonded to the substrate for the following reasons:

1. Sufficient attachment is necessary to prevent dislodgement under the gravitational and mechanical forces to which it is exposed.

2. Under immersion or conditions where condensation will occur, water vapour will penetrate between a barrier coating and an unbonded surface and condense on the unbonded surface. Progressive disbonding is most likely to occur.

THE DEVELOPMENT OF VINYLS AND THEIR USE IN SEALED STORAGES

There is some ambiguity in the literature and elsewhere as to precisely what is meant by the term vinyl; for example, vinyl house paints have a completely different composition and function to the vinyl Envelon used in sealant coatings. To simplify the nomenclature it is common practice in both the plastics and coatings industries to restrict the expression vinyl to the polymers and copolymers of vinyl chloride and vinyl acetate.

Polyvinyl chloride was first reported in 1872 and polyvinyl acetate in 1913. However, no serious commercial developments occurred until the mid

1920's. Polyvinyl acetate attracted interest as an adhesive, but polyvinyl chloride proved difficult to dissolve and was unstable in UV light or when heated. It was obvious that modifications were needed to improve these properties. In 1928 it was noted that copolymers of vinyl chloride and vinyl acetate produced resins of improved solubility in the then available solvents. Early developments are credited to German scientists, however Doolittle of Union Carbide in about 1936 patented a variety of solution vinyl resins using different monamer ratios. These were vinylite resins and have been marketed ever since for use in the coatings industry.

A copolymer of about 85-86% vinyl chloride with vinyl acetate was found to be almost impermeable in thin films to metallic ions and water. This polymer is completely tasteless and non toxic and is still used today as the final coat in beer and beverage can liners. Most of the basic coating formulations and systems were later developed during World War II. Solution vinyl coatings have proven over the years since then to perform in the most aggressive conditions. Their use in maintenance coating systems combines the properties of chemical resistance, toughness, water resistance and good exterior durability.

EARLY HISTORY OF SEALED STORAGES AND THE USE OF ENVELON

In the mid 1940's the US Department of Agriculture engaged in considerable research and development to find a suitable surface coating which could be applied by spray application to tobacco stores, where an air tight situation was required. Envelon membrane coating was selected because of its inherent properties and reliability.

During the 1940's, Envelon was brought to Australia and marketed under the name of Liquid Envelope, which is still a registered name held by Dominion Plastic Industries. Liquid Envelope was used extensively for the moth-balling of naval and army equipment after the Second World War and has continued in current use with various defence departments. Early in the 1960's, the name Liquid Envelope was changed to ENVELON, although even to the present time this range is still widely known by its original label.

As a result of research during the 1960's, it was found that by making fruit storage cool rooms completely air tight and by introducing certain selected gases, fruit could maintain its freshness up to and, in certain cases, in excess of one year. This process became known as controlled atmosphere (CA) storage. Once again a spray applied film was sought which would remain permanently flexible to accommodate substrate movement, even down to freezing temperatures, remain inert and stable to many gases while maintaining a completely impervious air seal. The Envelon membrane was found to fulfil all these requirements.

Dominion Plastic Industries became involved with wheat storage through State Agricultural Departments and bulk handling authorities, the Australian Wheat Board and the CSIRO in Canberra. Experience had shown that a spray applied membrane was needed for the permanent sealing of grain stores for fumigation and atmosphere control against insect infestation. Envelon appeared to possess what was needed to meet these stringent requirements.

After research by the CSIRO into the insect infestation of stored wheat, in 1977 it made contact with Dominion Plastic Industries to enquire after Liquid Envelope, which had been used by the Department of Entomology for a small experimental bin in Canberra many years previously. A small amount of work was carried out leading to further research into the technical and economic feasibility of sealing large wheat stores on a commercial basis. Work was subsequently carried out in nearly all the States in Australia by Dominion Plastic Industries and various State authorities. Interest was widely shown by scientists, engineers and managerial staff in this application of Envelon being carried out by Agricultural Departments, CSIRO, the State bulk handling authorities and the Australian Wheat Board. Extensive testing programmes were instigated by the CSIRO and the "Silo Sealing Committee" was appointed, which elected to carry out field tests in conjunction with laboratory testing. Although at the present time there is still much development work to do on sealed wheat stores in Australia, and, in fact, throughout the world, Envelon has already been used successfully. Structures which have been treated and sealed to a precise code of practice established by a Government-appointed scientific committee include horizontal concrete/steel stores, vertical concrete cylindrical cells, vertical steel bins and small, on-farm stores. The more noteable installations which have been sealed with Envelon include:

(a) A 300,000 tonne horizontal store constructed of concrete walls and aluminium roof cladding at Kwinana.

(b) A 22,000 tonne horizontal store constructed of concrete walls and galvanised steel roof cladding at Cunderdin.

(c) A similar store to (b) at Ongerup.

THE REQUIREMENTS OF A SEALANT COATING, WITH PARTICULAR REFERENCE TO ENVELON

Envelon is now being widely used on farm stores, particularly in Western Australia where the Agricultural Protection Board and Co-operative

Bulk Handling have vigorously advocated the concept of sealed storages as being the most appropriate for the highest standards of insect control now being demonstrated. Envelon has been developed over many years to include those characteristics and qualities most suitable for the sealing of grain stores for fumigation and controlled atmosphere storage. The more important features include:

I. Permanent Flexibility. Flexibility is of particular importance when considering expansion and contraction, e.g. steel or concrete, due to high and low temperatures or where two different types of substrate material are joined or overlap, with different coefficients of expansion. It is more important that the coating employed should not rupture or crack under such circumstances and over a long working life. This is an extreme test even for the best of membranes.

II. Strength and Abrasion Resistance. One of the essential properties of a film is its strength, often referred to as its toughness. Films of approximately 0.6 - 0.8 mm are commonly applied. Thickness will assist the material to last a long time but it is important that, at the same time, the sealant remains flexible and elastic. Envelon cures with a tough leathery feel, although unlike leather it will stretch and remain flexible. Films with these characteristics are resistant to abrasion and suffer minimal damage to the coating in the event of scuffing and abrasion.

III. A Seam-Free Surface. Sealants when applied by brush, roller or, as is most common, spraying, should leave a smooth seam-free surface, eliminating insect habitats in joints and gaps. Cleaning also is greatly facilitated once a bin is either being emptied or has been finally emptied. Brooming and dusting of areas are made considerably easier. During the application process, unless an area is specifically masked, which is normally not necessary, a fine smooth feather edge results which greatly reduces the event of a curl edge developing.

IV. Ability to Bridge Gaps. An essential property of successful sealants is the ability to bridge gaps. This means that, even though two surfaces may not meet or match perfectly, the sealant will bridge the gap to provide a continuous, even and seam-free surface. Envelon will bridge gaps up to 40mm wide. This is done purely by an application procedure. This bridging ability is of particular importance and assistance on older structures where movement has taken place, leaving gaps and spaces, or on concrete where cracks have developed or form work has left a rough or pitted surface. Extra filling of such cracks should be avoided by using sealants which can bridge gaps.

V. Weather Resistance and Impermeability. Surface coatings should be fully stabilised against changes from extreme heat to cold and solar radiation including particularly ultra violet radiation. This is of great importance when considering the resistance of coatings to weather, including high and low temperatures and frost. Some water based materials tend to crack when exposed to low temperatures and frosts, which are known to be critical times for the development of cracks. Envelon has been recently selected for use in the Antarctic after extensive testing of various coatings by the Department of Housing and Construction after being satisfied it remained flexible at sub-zero temperatures. Along with extreme weather resistance, also important is high impermeability, meaning the ability to withstand ingress of water from the outside and stop the escape of gases or atmosphere from the inside of any given sealed structure. When applied properly and with proper equipment, coatings should not have pin holes or a centre "honeycomb" texture. They should have a consistent smooth membrane providing a barrier to some of the most penetrating chemical gases. Impermeability figures for Envelon are given in the Technical Data Annex 1.

VI. Ease of Repair to Damaged Surfaces. Unfortunately, seals may be damaged by mechanical or other means, rendering a previously sealed storage either very expensive to use due to leakage and thus high costs of recharging or, in some cases, completely unuseable because of the amount of damage caused. With coatings that are very fast drying (touch dry in 5 minutes) an area can be repaired, sprayed and the store put back into service within a minimum of time. It should also be possible for damaged areas of the coating to be reinstated without disturbing the sound membrane around the repair area. Envelon will continue to cure in a sealed area. The repaired area will be immediately absorbed into the original coating without leaving any weakness in the repaired film. There will be a total molecular compatibility between the old and new coatings, regardless of age.

THE PRACTICAL APPLICATION OF SEALANT COATINGS

(a) Primers are formulated to adhere to metals, concrete, wood and masonry rather than to surface contaminants. If the surfaces to which primers are applied contain dirt, dust, scale, rust, oil or moisture, the bond of the protective system to the structure will be as good as the bond of the contaminant to the intrinsic surface of the structural component. The need for scrupulous surface cleaning prior to priming has become increasingly important, particularly since spraying has replaced brushing as a common method of application.

Applying specialist coatings is a skill learned by practice. However, many painters or applicators could improve the quality of their work and avoid costly mistakes by following the precise instructions of the coatings and equipment manufacturers. Frequently these require different techniques and practices to those used for conventional paints and if the manufacturers' instructions are not carefully followed there is a serious risk of failure of the coating.

(b) The ideal time for painting is when the weather is warm and dry with little wind. Obviously, many coating projects cannot be delayed until these ideal conditions prevail, therefore extra care will be needed (see (c)-(h) below).

(c) The substrate should be dry. Application should be avoided in rain and under conditions of high humidity when condensation of moisture is likely to occur on surfaces. Rain or condensation on the substrate interferes with bonding of the coating. Condensation on the surface of a freshly applied coating may alter its curing process.

(d) Extremely dry weather and low humidities can be a problem with water-based products. Rapid evaporation ("flash off") of the water may result in film cracking. It can also cause poor curing rates for solvent based coatings and also the ammonium types of inorganic coatings.

(e) At low temperatures the film thickness of high build or thixotropic coatings becomes more difficult to achieve. Curing reactions slow down or stop for many materials. Water based products may freeze. Solvents evaporate more slowly. Furthermore, when the relative humidity is over 70%, condensation is likely to develop.

(f) High ambient temperatures have some beneficial effects but this often increases overspray (dry fallout), trapped air or solvent bubbles and also reduce the pot life of catalysed materials.

(g) Wind is a nuisance, particularly when spray painting. The material, as it leaves the spray gun, can be deflected from the target. Solvent tends to evaporate quickly causing excessive dry spray at edges of the spray pattern. Lap marks become more evident. Dirt and other debris may become embedded in the wet film.

(h) Condensation becomes a problem when relative humidities exceed 70%. The solution is to avoid coating surfaces which are at temperatures below the ambient (surrounding) air. Unfortunately on large scale projects, primers are often applied late in the working day and sometimes at night. Contractors should establish that there is no likelihood of the structures falling below dew point temperatures during the night. Table 1 shows the conditions of ambient air temperature, uninsulated metal temperature and ambient relative humidity that will lead to condensation.

Table 1 - Relative humidity (%) above which moisture will condense on uninsulated metal surfaces.

METAL SURFACE TEMPERATURE °C	Surrounding Air Temperature °C																
	4	7	10	13	16	18	21	24	27	29	32	35	38	41	43	46	49
2	60	33	11														
4		69	39	20	8												
7			69	45	27	14											
10				71	49	32	20	11									
13					73	53	38	26	19	9							
16						75	56	41	30	21	14	9					
18							78	59	45	34	25	18	13				
21								79	61	48	37	29	22	16	13		
24									80	64	50	40	32	25	20	15	
27										81	55	53	43	35	29	22	16
29											81	68	55	46	37	30	25
32												82	69	58	49	40	32
35													83	70	58	50	40
38														84	70	61	50
41															85	71	61
43																85	72
46																	86

(i) Many of the application and dry or curing problems created by weather conditions can be reduced by lowering the viscosity of the material by the addition of the proper thinner. Thinning will improve flow and uniformity in the application of the material. It will also reduce overspray, lap marks, bubble entrapments and film "mud cracking" caused by rapid solvent flash off. However, the degree of thinning given in the product's application instructions should not be exceeded without checking with the manufacturer.

(j) Since thinning reduces the volume solids of a coating, film build may become difficult to obtain. In some situations reducing the thickness per coat and increasing the number of coats will result in a better job. This is often true in both cold and extremely hot weather. Thinner films permit easier escape of solvent under both conditions. Bubbles and pinholes in hot weather and extremely slow hardening rates of thick films in cold weather are the result of the solvent's difficulty in escaping at its ideal rate.

(k) To obtain a satisfactory coating it is essential that periodic inspections be made before, during and following the application. Proper inspection procedure begins with an inspection of the prepared surface. Specifications for the work should detail the quality of the prepared surface but often this is a decision left to the inspector and it is important that he be familiar with the individual tolerances of coatings. (For duties of inspectors, see Annex 2). Once the surface has been satisfactorily prepared as required by the specification it should be thoroughly dusted. Only then is it ready for paint. Surfaces which do not meet the specification should be referred for further preparatory work.

Each coat should be carefully inspected before the succeeding coat is applied. Oversprayer dust should be removed by light sanding or dusting. All loose contaminations in the primary coat should be removed and, together with any bare patches, be reprimed. The inspector must also ensure that sufficient drying time, as stated in the manufacturer's application instructions, be allowed between successive coats. To ensure proper coverage it is sometimes recommended that succeeding coats be of alternating colours or shades.

Specifications for the finished membrane and coating system usually call for a minimum film thickness. A variety of instruments would be used to make such measurements.

HUMIDITY AND DEW POINT CONDITIONS

The atmospheric conditions which prevail during the application of an organic coating should be such that the surface being coated is completely free of moisture. To ensure that no condensation occurs on the surface, the temperature of the substrate being coated should be at least $3^{\circ}C$ above the dew point for the prevailing conditions. Additionally, it is normally recommended that this moisture-free condition be maintained from one hour prior to coating through to one hour after coating. In accordance with good painting practice, no organic coating should be applied if the temperature is below $5^{\circ}C$ or the relative humidity is above 85%.

To check the suitability of conditions for painting, the relative humidity can be indicated by an hygrometer and the ambient air and substrate temperatures measured with a surface thermometer. It is recommended that the same instrument or sensor be used for measuring air and substrate temperatures since any minor error between two instruments may lead to significant errors.

From Figure 1 the difference between ambient air temperature and steel temperature for the observed relative humidity indicates if conditions are suitable for painting*.

COATINGS FOR CONCRETE

The reasons for painting steel are obvious but it is not so obvious why concrete should be protected. In general, concrete is considered to be a material of high durability, but it is still correct to speak about the "corrosion" of concrete. This deterioration is mainly caused by water, existing in different phases. Concrete surfaces, which are alkaline, are attacked by acid gases in the atmosphere, such as carbon dioxide and sulphur dioxide. By painting concrete it is possible to fabricate surfaces of high resistance to both mechanical and corrosive damage and wear.

When concrete cures, a water rich layer forms at the surface which, on drying, results in the mechanically weak material known as "laitance". Apart

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*The evaporation of volatile solvents from paint can result in the condensation of moisture when the relative humidity is high because when solvents evaporate there is a reduction in surface temperature which can cause moisture to condense. Because of this an upper limit of relative humidity of 85% is usually set for the applicationof paint containing volatile solvents. However, this limit should be varied if the temperature of the steel being coated is different from ambient temperature. Provided that the temperature of the substrate is at least $3^{\circ}C$ above ambient temperature, painting can be carried out up to at least 95% relative humidity. On the other hand, condensation is much more likely at high humidities, and it may be desirable to allow the temperature to stabilise.

from the poor mechanical properties, the chemical composition of this layer is different from the rest of the concrete. From reactions with carbon dioxide and a variety of oxides of sulphur, a voluminous calcium-sulphur-aluminate material is formed. This is a poor surface for painting and most coating failures on concrete are connected with the inefficient removal of this laitance layer.

Normal new or fresh concrete contains over 80kg of water per cubic metre and this evaporates until an equilibrium is reached with the surrounding air. For example, a concrete composed of water/cement/aggregate in the ratio of 0.6:1:5 contains about 9% water with 3.5% bound chemically to the cement. This concrete would contain 1.5% moisture when in equilibrium with air at 50% RH and the remainder must evaporate. If this moisture cannot escape before painting, or through the paint film, it sometimes forces off the paint film causing blistering and flaking. The ususal procedure is to allow the concrete to "cure" for about a month before laitance removal and subsequent paint application.

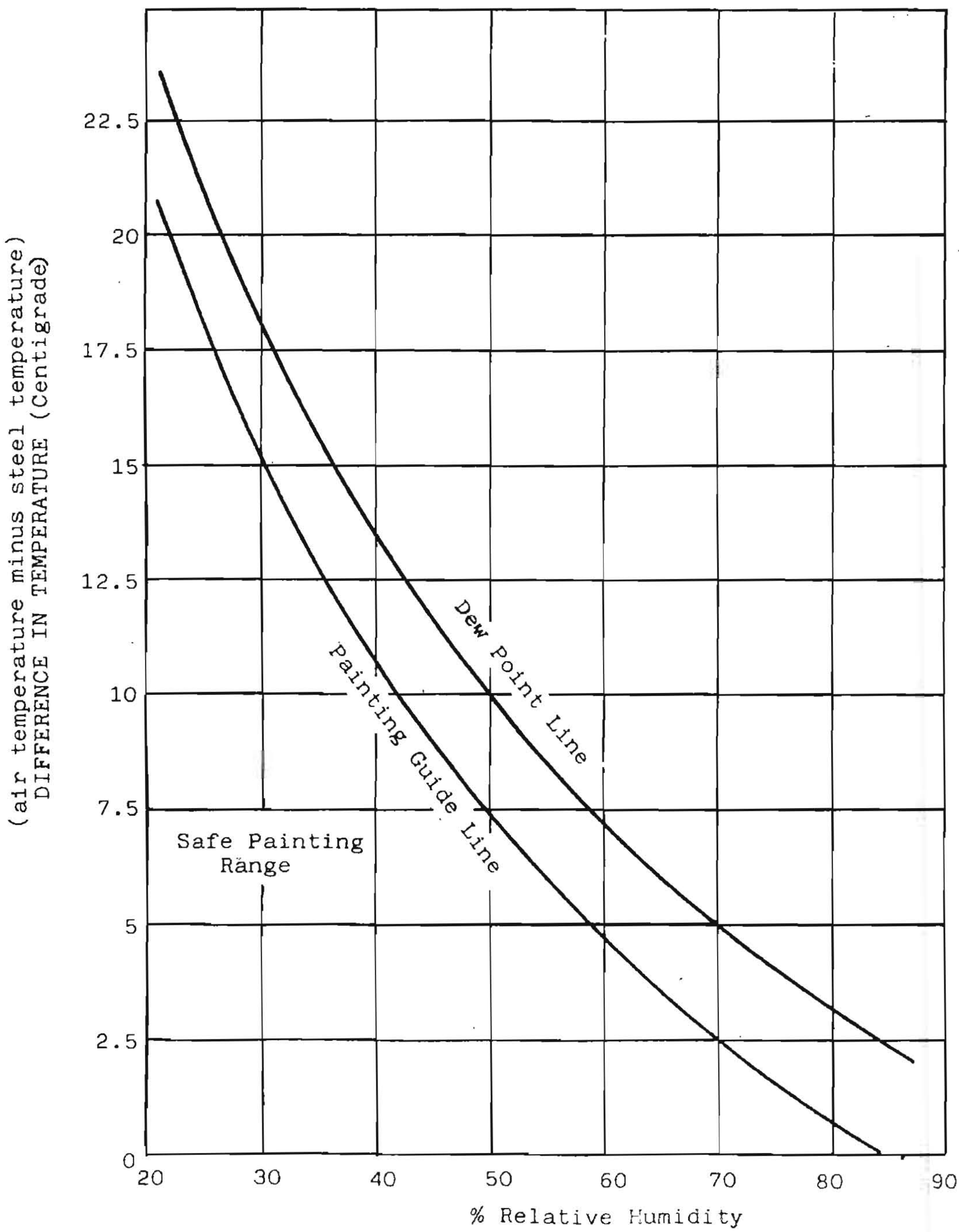

Fig. 1 Painting guide.

Annex 1

ENVELON TECHNICAL DATA

Colour: Aluminium Grey or White

Viscosity: 65 Krebs units at 25°C measured on a Stormer Viscosimeter using a 100g weight

Weight per litre: 0.758 Kg per litre

Solids Content: Non volatile - by weight 30-32%

Drying Time: Air dries to touch in 4 minutes, to handle in 20 minutes and cure in 12 to 24 hours

Spraying: Conventional air atomising or airless spray equipment is recommended. The following equipment has been used and found suitable. However, equivalents may be substituted:

	Gun	Air Cap, Fluid Tip/Needle
Binks-Bullows	Beta	68PB 2110
Arnold (C1G)	Beta	410553
Devilbis	J.G.A.	62AC

Moisture traps should be installed to avoid contamination of the coating. For airless spraying, use a machine of at least 16,000 kPa capacity and with adequate volume output for the specific application. A tip of 0.011" or 0.013" is normal; fan width should be chosen for particular work involved. For application to roof areas, a pole gun extension may be helpful. Continuous agitation is recommended during spraying operation.

Reduction Required Not normally required as ENVELON is supplied ready for use. Below 20°C viscosity increases considerably. Rapid agitation will bring material back to spraying consistency.

Shrinkage 40 μm film will not bridge over a concave surface after being subjected to accelerated weathering for six days.

Adhesion Measured on a Scott Tensilometer with jaws separating 7.5cm per minute. 30 μm film adhered to Alclad Aluminium (24-ST) with force of 5-10 kPa.

Chemical Resistance Excellent resistance to gasoline, oil, fat, grease, acids, alkalis, alcohol and salt spray.

Heat Resistant Shows no sign of plasticizer separation after being subjected to a constant temperature of 80°C.

Tensile Strength Measured on a Scott Tensilometer according to Federal Specification 22-R-601, produces a tensile strength higher than 3790 kPa.

Elongation Fresh films 280%. Aged film 270% when measured in a Scott Tensilometer according to Federal Specification 22-R-601.

Water Vapour Measured in modified Southwick cup in General Foods Cabinet at 100% RH and 38°C. Grams per 645cm^2 in 48 hours, 0.6 grams.

Incombustibility Dry film will not continue to burn or glow after source of ignition has been removed.

Storage Should be stored at 20°C for at least 24 hours before using. If body is still too heavy, rapid agitation will bring viscosity back to spraying consistency.

Cleaning Spray ENVELON thinner 5 is recommended for cleaning spray equipment after using ENVELON.

DUTIES OF INSPECTORS

General

The prime function of an inspector is to ensure that the agreed painting specification is being followed correctly without unnecessary delay or hindrance to the contract.

The inspector should not become the work supervisor accordingly, all communications should be made through the contractor's on-site foreman and should not be given directly to the painter.

The contractor should advise the inspector in sufficient time to enable the inspector to be present at the time he is required. The inspector should try to anticipate when he is to be required.

It is usual for the painting foreman to initially inspect all painting and carry out any necessary rectification before calling in the inspector.

Surface Preparation

The inspector should ensure that the following matters associated with surface preparation are observed:

1. That surface preparation complies with the specified Australian or other standard and that it is free from contamination.

2. That no area of prepared surface is primed before approval to proceed has been granted. Note: Inspectors should ensure that unnecessary delays are avoided.

3. That the first coat of primer or membrane is applied within the specified time period.

4. That coated surfaces are not contaminated by other cleaning processes.

In carrying out these duties, the inspector should note whether on-site ambient conditions are satisfactory for painting, as set out before.

Paint Application

The inspector should ensure that the following matters associated with the application of the coatings are carried out:

1. The membrane and associated products to be used comply with the relevant specifications.

2. The material containers are sound and not damaged in any way which may have caused, or will cause, the contents to deteriorate.

3. The condition of the membrane or paint in the container is satisfactory, i.e. the paint is free from undue settling, thick surface skin, gelling, foreign matter and the like.

4. No unauthorised thinning (which can affect the membrane or paint consistency, its hiding power, elasticity and application properties) has been allowed to occur.

5. No adulteration of the paint has taken place.

6. Multi-component paints are correctly mixed in the correct proportions and the paint is not used after the expiry of the pot life stated by the manufacturer.

7. The membrane or paint is applied by the correct method and that the specified drying times have elapsed between coats.

8. Any defective work is corrected immediately and any recoating follows precisely the coating system specified. Note: Irregularities or unsatisfactory areas should be clearly marked with chalk. Grease pencils or other such marking materials, which can affect paint adhesion, should not be used.

Final Inspection

Final inspection of the painted steelwork should include the following:

1. Measurement of the total dry film thickness of paint.

2. Checking for holidays in the paint film, if applicable.

GLOSSARY OF TERMS USED IN DESCRIBING CORROSION AND COATINGS

Acid — Compound producing hydrogen ions in water. See "pH".

Acrylic Resin — Synethetic resin that has excellent water resistance and hardness.

Activator — Component which, when added to a coating, speeds up a desired rection.

Aerobic — Micro-organism (bacteria) contributing to corrosion. Needs oxygen for growth.

Air Dried — Coatings that normally reach a desired hardness without external heat.

Airless Spray — Method of applying coatings using high fluid pressures - no air for atomisation. See "Hydraulic".

Alkali — Term applied to caustic and strong bases. See "pH".

Alkyd — Resin used in many types of industrial enamels. The reaction products of polyhydric alcohols and polybasic acids. Properties vary widely.

Alligatoring — Pronounced wide cracking over the entire surface of a coating. Resembles alligator skin,

Alloy — Mixtures of metals that are blended to provide various physical characteristics, e.g. brass, stainless steel, bronze.

Anaerobic — Micro-organism growth contributing to corrosion where no oxygen is present.

Anode — Positive terminal of an electrolytic cell; metal that corrodes.

Anodizing Generally pertains to an oxide deposited on aluminium electrolytically.

Anti-Fouling Coating applied to ship bottoms to prevent marine growth. May contain toxic ingredients.

Aromatic Hydrocarbon Volatile solvent such as benzene, toluene, xylene. Usually extracted from coal tar, but can be made from petroleum. See "Solvent". Term refers to chemical structure - not smell.

Asphalt Black resinous material of petroleum origin.

Binder The resin portion of coatings whose function is to hold pigments together, and provide a cohesive film.

Bleeding The diffusion of coloured matter through a coating from the substrate, also the discoloration arising from such diffusion.

Cathode Negative terminal of an electrolytic cell; Hydroxyl ions usually produced here.

Cavitation Force exerted by a high-velocity liquid forming a gas filled space. Usually a combination of corrosion and erosion.

Chalking Form of paint degradation in weather resulting in loose pigment on surface.

Checking Breaks or cracks in the film that may or may not penetrate to the underlying surface.

Coal Tar Black resinous material derived from coal. A resin used in coatings.

Coat (of paint) One application. Can include several passes if no drying time is allowed between.

Cold Flow Characteristic of many plastics to take a permanent set after pressure is applied.

Cold Rolled Steel	Produced from hot rolled; has little, if any, mill scale. Typical use - auto industry.
Conductive Coating	Accomplished by the addition of pigment which will conduct (static) electricity.
Corrosion	Adverse reaction of any material with its immediate environment.
Cross Spraying	Pertains to making two passess with a spray gun over the same surface at right angles to each other.
Craters	Circular domes in dried film with a thin spot in centre. Can be minute up to 3 mm in diameter.
Curtains	Long horizontal runs in film; occurs on vertical surfaces.
Curing Agent	Activator or hardener added to a synethetic resin to increase chemical resistance and hardness.
Degrease	Removal of grease, petroleum products, fish oil, etc. Generally by the use of solvents, e.g. trichlorethylene, methyl ethyl ketone.
Deionized Water	See "Demineralised".
Delamination	Separation between coats and very poor adhesion.
Demineralised Water	Purified water from which metallic cations are removed by a hydrogen cation exchanger. Anions are removed by an anion exchange.
Diluent	Product introduced as a portion of the solvent to reduce cost.
Dispersion	Suspension of minute particules in a suitable medium.
Distilled Water	Water that has been vaporised and then condensed to remove impurities.

Dry Spray	Result of over-atomization. Produces dull powdery or pebbly finish. Some of the surface dust can be wiped off with the palm of the hand. Also called overspray or spray dust.
Drying Oil	A fatty oil capable of conversion from a liquid to solid by slow reaction with oxygen in the air. The "drying" thus refers to a change of physical state and not an evaporation of solvent. Paints can be made from drying oils due to this hardening ability.
Elastic	Springiness, rubbery quality - can be stretched.
Electrolyte	A solution of ions in water capable of conducting current.
Electro-chemical	Chemical changes produced by an electrical current or the production of electricity from a chemical reaction.
Enamel	Type of oil-base paint with high gloss.
Epoxy	Synthetic resin derived from petroleum products that can be cured by a catalyst or formulated to upgrade other synthetic resins to form protective coatings.
Epoxy-Baked	Epoxy formulation that requires higher than normal temperature to cure or react. Requires another resin or catalyst.
Epoxy-Catalyzed	Epoxy formulation that cures by the addition of a catalyst, generally at room temperature.
Epoxy-Modified	Predominantly epoxy but mixed with other resin or resins, e.g. epoxy-phenolic.
Ester	Compounds formed by the reaction of alcohols with organic acids, e.g. butyl acetate.
Etching	Generally pertains to the result of the treatment of a surface with acid.
External Atomization	When air is used to break up the coating material after it has left the spray gun nozzle.

Film	Coating or paint thickness. A wet film is one that has just been applied.
Flexible	Ability of a film to bend without breaking.
Flooding	Pigment that floats to the surface of a film, usually in streaks.
Force Dried	Application of heat above room temperature to hasten drying. May be up to 80°C but below that of baking temperature.
Foreign Thinner	A thinner that is not specifically recommended by the coating manufacturer.
Fouling	Marine growth, weeds, barnacles; growth of attachments to hulls of ships or marine structures.
Galvanized	Coated with molten metallic zinc by dipping.
Gelled	Coating which has coagulated and formed a jelly-like body.
Glossy	Mirror-like finish.
Grit	Angular abrasive particles made from by-product steel or iron slag. Frequently used instead of silica sand for blast cleaning.
Heavy Scale	Iron oxide rust formed in layers from 3 mm to 12 mm thick.
Holiday	Any discontinuity or bare spot in a painted area.
Hot Rolled Steel	Normally used in structures and tankage; has mill scale on surface. See "Cold Rolled".
Hydraspray	Trade name meaning hydraulic spray. See "Hydraulic Spray".
Hydraulic Spray	Material at 10,000 kPa or higher is forced through an aperture. No air is used for atomization.
Hydrocarbon	Extracts from petroleum, e.g. gasoline, lube oil.

Immersion	Refers to an environment which is submerged continuously.
Inhibitors	Compounds added in small concentrations to form protective films or which, combined with corrosion products, form less active compounds.
Inhibitive Pigment	A pigment added to coatings capable of passivating or retarding the corrosion of the metal over which the coating is applied. Examples: red lead, chromate salts.
Inorganic	Chemicals such as salts, acids, alkalis, etc. Based on all chemical elements except carbon.
Inorganic Zinc	Coating containing a zinc powder pigment in an inorganic vehicle, e.g. zinc silicate.
Internal Mix	Refers to a spray gun in which the material is mixed with air before being discharged through the tip.
Ion	An electrically charged particle derived from soluble mineral chemicals on dissolving in water. Ions are both positively and negatively charged and equal amounts of each are formed by splitting of the original chemical when it dissolves.
Iron Oxide	Ferric oxide - rust. Reddish in colour. Also used as paint pigment.
Ketones	Class of organic compounds, e.g. acetone, MEK.
Lacquer	Type of coating that dries from evaporation of solvent.
Lacquer Thinner	Used to describe such solvent as ethyl alcohol, ethyl acetate and toluene.
Latex	Milk-like fluid made up of particles of rubber or synthetic resin suspended in water.
MEK	Ketone solvent, methyl ethyl ketone.
MIBK	Ketone solvent, methyl isobutyl ketone.

Mastic	Term used to describe a heavy bodied paste-like coating.
Metalizing or Metal Spray	Method of applying atomized molten metal to a surface, e.g. zinc, aluminium.
Micron	Unit of film thickness = 0.001mm.
Mild Solvent	Solvent that has a limited range of dissolving ability.
Mill Scale	Layer of iron oxide formed on the surface of steel plates and sheet during manufacture. May be 50 - 125 micron thickness.
MILS	Unit used to designate film thickness. 1 mil = 0.001 (one thousandth) of an inch. Equivalent to 25 micron (μm).
Modified	Pertaining to a mixture of resins - one of which is predominant.
Monomer	Molecule of low molecular weight capable of conversion into polymers, plastics and synthetic resins.
Mottled	Descriptive of coatings of spotted appearance, blotches of a different colour or shades of a colour.
Mud Cracking	Phenomenon that occurs to films as they dry, appearing like mud drying in hot weather.
Natural Rubber	Sap or gum taken from the rubber tree.
Neutral	Term used to describe an environment which is neither acid nor alkaline.
Non-Ferrous	Term used to designate metals and alloys that do not contain iron or steel, e.g. brass, aluminium, magnesium.
Non-Immersion	Refers to an environment which is not continously submerged.
Oil Cleaner	Solvent and detergent often used to remove heavy deposits of asphalts and oils from ship hulls.

Orange Peel	Appearance of a coated surface resembling the skin of the orange.
Organic	Chemicals based on carbon, as contrasted to mineral chemical compounds. Carbohydrates, synthetic resins, solvents and an enormous variety of chemicals are organic.
Organic Zinc	Coating containing zinc powder pigment and an organic resin, e.g.. zinc and epoxy.
Organosol	Colloidal dispersion of an insoluable material (synthetic resin) in a plasticizer with a solvent added. See "Plastisol".
Osmotic Blistering	Blistering of film due to salt deposits beneath the coating. Wet blisters, filled with salt solution, are formed.
Overspray	Sprayed coating that is dry when it hits the surface, resulting in dusty, granular adhering particules.
Oxidized Film	Coating that has lost its gloss and/or its surface has become powdery.
pH	Value indicating the acidity of a solution. Pure water has a pH value of 7 or neutral. Acidity ranges down to pH 0 and alkalinity up to pH 14.
Passive	Surface that has shown no active corrosion or an atmosphere that is not corrosive.
Pattern (Spray)	Shape of stream of material coming from the spray gun.
Pebbly	Rough, irregular surface having a coarse grainy texture.
Peeling	Poor adhesion but implies cohesion of film.
Phenolic	Synthetic thermosetting resin. The most important type historically is phenol-formaldehyde (Bakelite). These resins can be formulated to produce coatings, varnishes, moulding materials and adhesives.

Phosphatizing	Use of phosphoric compounds as a surface treatment to combat corrosion, e.g. parkerizing, bonderizing.
Pickling	Chemical treatment of iron or steel, usually with sulphuric acid to remove mill scale and/or rust.
Pigment	Insoluble finely divided material whose function is to provide obscuring value, colour and protection.
Pinholes	Formation of tiny circular holes in the film up to a few millimeters in diameter, usually having a residual solid in their centre.
Pitted	Result of local corrosive attack forming holes in a metal surface. May be described as shallow or deep, small or large in diameter, and quantity or number per unit area.
Plastic	Resinous material that may be moulded into desired shapes or dissolved to form a coating.
Plasticizer	Materials added to improve flexibility and assist in compounding a coating.
Plastisol	Similar to organosol but is packaged at 100% solids content.
Polymer	Substance composed of giant molecules formed by the union of a group of simple molecules (monomers).
Polymerization	Uniting of monomers to form polymers.
Polyurethane Resins	Class of resins obtained by the reaction of diisocyanate with organic compounds, e.g. phenols, amines, to form polymers.
Potable Water	Water fit for human consumption - drinking water.
Primer	First coat applied to a surface. See "Coating System". Formulated to have good bonding and wetting characteristics and may or may not contain inhibitive pigments.
Profile	Term used to describe anchor pattern on a surface, produced by sandblasting or acid treatment.

Reducer — See "Thinner".

Relative Humidity — Ratio of the quantity of vapour actually present in air to the greatest amount possible at the given temperature.

Resin — Any of a group of amorphous organic materials; usually can be moulded or dissolved. Can be natural or synthetic.

Retarder — Liquid added to a coating usually used to modify the drying rate.

Runs — Synomous with sagging and curtaining caused by improper thinning or poor application.

Rust — Formation of visible iron oxide, as a result of corrosion of iron or steel. May be described in order of severity: scattered pinpoints, blush or powdery, freckled or streaked, light scale, paper thin, flaked, medium scale (layers up to 3 mm thick), heavy scale (over 3 mm thick).

Sacked — Treatment of concrete with grout to fill voids. A burlap sack may be used.

Salt Solution — Commonly known as any combination of cation and acid radical as distinguished from acids and bases.

Satin Finish — Descriptive term generally with reference to decorative paint, usually semi-gloss.

Silica Sand — Clean sand made up of sharp silica particles. Does not contain dirt or clays. Usually recommended for blast cleaning.

Silicone Resin — Resin formulated into coatings to withstand high temperatures.

Slush Coat — Coating that is applied to the interior of a container by revolving the container and then draining out the excess coating.

Solvent — Liquid whose function is to put the actual film composition in a physical condition so that it may be applied conveniently.

Surfacer	Pigmented composition for filling depressions to obtain a smooth uniform surface before applying finish coats; usually applied over a primer.
Synthetic	Manufactured, as opposed to 'made by nature'.
System Coating	See "Coating System".
Tensile Strength	Longitudinal resistance of a material to resist elongation. Expressed in pounds per square inch at the failure point.
Thermosetting Resin	Resin having the property of becoming insoluble upon the application of heat.
Thermoplastic Resin	Resin having the property of becoming soft upon the application of heat. Regains hardness on cooling.
Thinner	A liquid (solvent) added to a coating to adjust viscosity.
Thixotropy	A specific type of variable consistency behaviour. A thixotropic coating formulation shows a "false body" or apparent high viscosity, but on stirring or other agitation readily flows and shows low viscosity. Thixotropic paints are easily brushed or sprayed in thick films, but after application quickly "set" and do not run or wrinkle. Example: ketchup, mayonnaise, whipped cream.
Thousandths	Measurements of film thickness in decimal inches. See "MILS".
Tubercles	Round protuberances having the appearance of a plant-root segment, usually rust growth.
Varnish	A binder for enamels. The resins are chemically combined with the oil at high temperature to give a product of increased hardness and much faster drying time. Chemical resistance is also improved.
Vehicle	Liquid portion of a coating; a fluid or mixed solution made up of the binder and solvent.

Viscosity	Resistance to flow. The internal friction of a fluid.
Vinyl Resin	Synthetic resin having a wide range of chemical resistance. Can be formulated to produce adhesives, sheets, textile coatings, etc.
Volatile	Thinner component of the vehicle. The non-volatile components are known as the film formers.
Weld Spatter	Round particles of extraneous metal that adjoin a welded joint.
Wet Film	Designates the coating after application but before the thinner volatizes.
Wet Sand-Blasting	Use of wet sand in blast cleaning; has the effect of reducing dust particles in surrounding areas.

THE PROPERTIES OF VARIOUS SEALING MEMBRANES AND COATINGS USED FOR CONTROLLED ATMOSPHERE GRAIN STORES

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ABSTRACT

This paper describes the work undertaken by the Gardner Perrott Group in investigating consistent types of failure of sealants and coatings used in sealing operational grain stores. Failures mostly occurred at lap joints between cladding sheets, or similar joints and overlaps where mechanical forces were imposed on the sealants through movement of the structural components. The ideal sealant properties are described; in particular the ability of the sealant or film to peel off the substrate in preference to breaking so that the tensile strength should be greater than the adhesive strength. A series of tests were carried out on common sealants, coatings and foams to determine their suitability in practice. It is concluded that PVC coatings used on their own will peel, but have poor elongation properties and need top coatings to give protection from UV-radiation. Acrylic coatings, if used alone, need reinforcement to increase tensile strength. A combination of the two systems is possible provided that the tensile strength of the films is greater than the peel strength and that PVC films in a combination should always be weaker than the associated acrylic film. If carefully chosen all the films under test were considered suitable for sealing grain stores.

INTRODUCTION

The Gardner Perrott Group has been involved in silo sealing technology since the first attempts were made to seal operational grain stores. Many of the techniques were developed in the earlier programmes, for example the sealing of the horizontal storage silo at Harden in New South Wales. During this period the CSIRO and various state grain storage and handling authorities have developed criteria which they believe sealants should meet under test conditions before they can be considered for use as sealants in the field. The test criteria were most relevant to the vertical concrete silo.

In 1982 a series of contracts for the sealing of grain stores owned by Co-operative Bulk Handling were put out to tender and the Gardner Perrott Group was awarded the contract for sealing nine stores, located at Beacon, Nembudding, Bencubbin, Kodj Kodjin, Bodallin, Doodlakine, Shackleton and Hyden. All the stores except Kodj Kodjin were "A" type stores, while the Kodj Kodjin was a "G" type store *.

* "A" type stores are framed buildings with concrete walls, concrete floor, concrete or steel portal frames, or concrete steel columns with warren or similar trusses, corrugated galvanised sheet steel roof cladding.

* "G" type stores are generally steel framed buildings with walls of A shaped timber frames sheeted internally with corrugated sheet steel, the corrugations running horizontally. The floor is compacted hardcore with an asphalt surface. Roof cladding is corrugated galvanised sheet steel.

There was some significant differences between the specifications for the Harden and CBH stores, for example in Western Australia external foaming was not allowed and this necessitated a change in the method of sealing end laps of sheets. New techniques were required and these were rapidly developed and applied without difficulty. In the case of the sheet end laps the techniques developed and tested were:

double taped putty films
sand acrylic grout
foam rubber saturated with acrylic

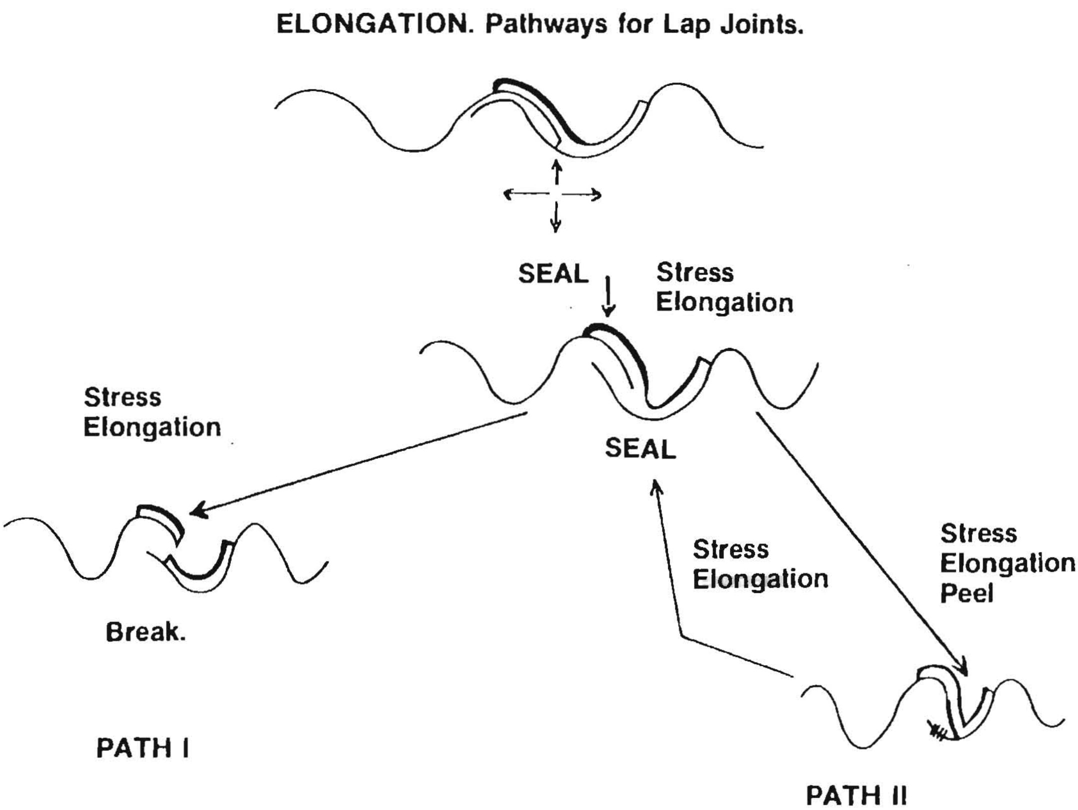
The most uniformly suitable technique was the use of foam rubber with acrylic.

There were however some apparently consistent types of failures associated with corrugated sheet steel lap joints on roofs and walls. We embarked on a failure analysis study of stores which showed these failures.

It became apparent that the failure on lap seals is a common problem. One way in which the Gardner Bros. & Perrott specification overcame this was to reinforce the film with glass fibre or synthetic fibre mesh sealant. This has proved successful particularly in such difficult locations as sealing the flashing bridging the gap between the wall and the roof where movement due to mechanical forces is a common phenomenon. In some cases the reinforced film peels away from the wall in attempting to accommodate wall movement.

An analysis of the lap failure mechanism is presented in Figure 1 and indicates that two possible pathways can occur; one which will certainly lead to failure or seal breakdown and the other which will probably retain the integrity of the seal. The sequence of events of Pathway I is the elongation of the bridging sealant beyond its tensile and elastic limits until breaking of the film or sealant occurs. Pathway II is a cyclical series of events in which peeling of the sealant adjacent to the lap takes place before the tensile strength of the film is exceeded. This can theoretically continue until peeling has reached the edge of the sealant furthest from the lap.

FIGURE 1


ELONGATION. Pathways for Lap Joints.
SEAL
Stress
Elongation
SEAL
Stress
Elongation
Break.
PATH I
Stress
Elongation
Stress
Elongation
Peel
PATH II

ANALYSIS OF SEALANT PROPERTIES

In our analysis of the sealant films and foams, we felt a clear statement of objective and terms of reference needed to be made:

1. No sealant, film or foam used in normal commercial thicknesses can withstand the forces which cause the movement of the silo structure. They must yield or break.

2. The ideal material should have:

   I. Maximum elasticity
   II. Excellent gap bridging properties
   III. Tensile strength greater than adhesive strength, that is peel in preference to break
   IV. Maximum tear resistance
   V. For external use, UV and weather resistance.

The physical properties which we then defined as essential to measure under controlled conditions were:

(a) Tensile strength
(b) Percentage elongation at break
(c) Tear strength
(d) Adhesive or peel strength.

The method of measuring tensile strength and per cent elongation is described in ASTM D882. We carried out each measurement only in duplicate whereas ASTM D882 requires ten replicates. However, we feel our abbreviated tests were satisfactory for the purposes of this study.

For the tear strength tests, films were prepared as required in ASTM D882 by drawing out wet films on glass, curing, cutting into 25mm strips, and peeling from the glass. A variable which we need to acknowledge is that we cannot be absolutely sure all films were totally cured. We were reasonably confident they were cured by allowing films to cure for several days on the glass plate and then allowed several more days cure after peeling from the glass. We believe that, since we did not observe large differences in tensile strength versus thickness, that we achieved total cure. Tests were carried out at 350, 700 and 1,500 microns dry film thickenss at $0^{\circ}C$, $25^{\circ}C$ and $50^{\circ}C$.

The adhesive strength of films to new and old galvanised sheet steel plate was measured by drawing out a 1,000 micron thick film over the plate, half of which was covered by plastic coated paper. The films were cured and 180^{o} peel tests were carried out using the tensile testing apparatus. Several of the films broke rather than peeled and so the procedure was repeated with reinforcing fabric so that a peeling mode was achieved.

Tensile and per cent elongation tests on foams were carried out on the same apparatus as for films. Tear strength was considered irrelevant. Adhesive strengths were measured in a compressive, shear mode for a 25mm wide strip compressed at a constant speed and the force needed to cause peeling on the 25mm face was measured.

Per Cent Elongation of Films

Maximum elasticity before break is a prime requisite of a sealant film. The results of elongation tests are depicted in Figures 2, 3 and 4. it will be seen that the polychloroprene type film gave the best performance followed by the acrylic coatings and then the PVC coatings. No judgement can be made as to what is a satisfactory elongation. Our belief, which is made evident later, is that if properly specified, all the coating systems tested will perform satisfactorily. However, elongation cannot be considered in isolation. In Figure 5 a graph of elongation versus temperature shows that most coatings lose elasticity with increasing temperature. The variant on this was the Envelon which becomes more elastic. Two possible explanations for decreased elasticity with increasing temperature are a more complete cure or loss of volatiles from the film.

Tensile Strength of Films

To the extent that no sealant will resist the forces applied to them by movement of the structure, its tensile strength cannot be considered in comparison to the strength of structural components. The ideal characteristic would be for there to be no significant change in tensile strength with variations in thickness of film or temperature.

Tests of tensile strength with respect to thickness of film at different temperatures are illustrated in Figures 6, 7 and 8. It is readily apparent that the acrylic and polychloroprene films possess the ideal characteristic while the PVC coatings less so. The Elascote at $50^{o}C$ has a dramatically increased tensile strength shown in Figure 9. The increase in tensile strength of the Elascote correlates with the reduced elongation at higher temperatures shown in Figure 5.

Tear Properties of Films

The most important aspect of tear susceptibility of films is the reduction in desirable properties such as elongation and tensile strength when the film is cut. Table 1 shows the elongation (ER) and tensile strength (SR) ratios for the cut and uncut test strips. The PVC films are similar to each other, while the acrylics are different to the pVC;s but similar to each other also. The PVC films have less reduction in elongation, but greater reduction in tensile strength. The polychloroprene shows different characteristics to both in that its strength shows neglible sensitivity to cutting, but the elongation sensitivity is markedly affected by temperature.

Table 1 - Elongation and tensile strength (tear/tensile ratios)

Material	Thickness mm	Temperature °C 0		25		50	
		ER	SR	ER	SR	ER	SR
Gaseal	300	0.31	0.61	0.26	0.60	0.24	0.65
	600	0.34	0.46	0.31	0.48	0.30	0.50
	1,500	0.45	0.64	0.45	0.69	0.43	0.45
Flexacryl	330	0.27	0.95	0.28	0.95	0.31	0.94
	650	0.27	0.85	0.27	0.93	0.29	0.88
	1,300	0.31	1.13	0.35	1.05	0.42	0.87
Siloseal	300	0.32	0.95	0.20	0.59	0.32	0.68
	750	0.32	0.90	0.32	0.81	0.44	0.86
	1,500	0.45	1.30	0.35	1.08	0.41	0.88
Envelon	350	0.45	0.70	0.44	0.69	0.50	0.52
	700	0.47	0.56	0.46	0.47	0.43	0.55
	1,100	0.52	0.64	0.53	0.62	----	----
Elascote	350	0.55	0.59	0.55	0.75	0.35	0.85
	1,000	0.51	0.74	0.57	0.73	0.45	0.79
Polychloroprene	300	0.88	1.33	----	----	0.34	1.15
	450	----	----	0.45	1.29	----	----
	850	0.73	1.25	0.30	1.17	0.21	1.24
	1,350	0.73	1.16	----	----	----	----
	1,500	----	----	0.25	1.14	0.20	1.07

The Wastolan acrylic is not included in this table because it shows anomalous behaviour. It was the only one which possessed a yield point, that is there was a critical point beyond which elongation continued with a reduced or reducing tensile strength below the maximum tensile strength of the critical point. The decision as to whether the yield or break point should be used for assessment is clouded by the fact that the yield point gives 50 to 80% higher tensile strength, but the elongation is only 20% of break point elongation. The choice would depend on integrity of the seal. Since we have not measured this, we make no choice as to which is suitable. All previous strength and elongation figures were based on figures for the break point on the assumption that there appears to be an integral film until that point.

Adhesive (Peel) Strength of Films

This is considered as a critical property of a film. The elongation pathways illustrated in Figure 1 indicate that the ability of the sealant to peel from the substrate is an essential property to accommodate excessive structural movement. The adhesive strengths of the films to new and old galvanised sheet steel is presented in Table 2, along with the tensile strengths for a 700 micron film. The figures in brackets beside the tensile strengths are the tensile strengths converted to a linear force (kgf/cm) for comparison to the peel strength.

There is one feature of the results which should be noted; the films gave an initial peak strength which was slightly higher than the residual peel strength. Gaseal was the only film with a significantly larger initial peel strength.

The observation was that only the Envelon and Elascote peeled from the surface in an unreinforced state. This is consistent with the data in the table. Figures with an asterisk indicate cohesive peeling rather than adhesive peeling.

SPECIFICATION DESIGN

The principles established earlier are quite clear. Good elongation and the ability to peel from the substrate are the important characteristics. The PVC coatings on their own will peel, but they have lower percentage elongations. They need a surface coating to give protection from UV radiation. Acrylic coatings, if used alone, need reinforcement, which increases labour costs at application. Combination of the two systems is possible, and can take two extremes:

I. Thick PVC and thin acrylic, to give protection from UV radiation.

II. Thin PVC and thick acrylic. This offers the potential for acrylic peel with reinforcement. The combination gives maximum elongation characteristics and has relatively inexpensive application costs.

Table 2 - Adhesive (Peel) strength of films

	Peel Strength at 25°C kgf/cm^2		Tensile Strength at 25°C 700 m kgf/cm^2 (kgf/cm)
	OLD	NEW	
Wastolan P	1.61	0.14	3.0 (0.2)
Wastolan	3.13	2.81	7.5 (0.5)
Flexacryl	*2.00	*2.44	17.5 (1.2)
Siloseal	*1.64	*0.85	19.5 (1.4)
Gaseal	*1.12	1.16	24.0 (1.6)
Envelon	0.89	1.05	32.0 (2.2)
Elascote	*1.10	1.20	46.0 (3.2)

* Failed cohesively

Table 3 Tensile Strength and Elongation of foams at 25°C

FOAM	TENSILE STRENGTH	% ELONGATION
Formafill	0.94 kgf/cm^2	approx 10%
Aerofroth	1.56 kgf/cm^2	approx 20%
ICI Rigid	3.39 kgf/cm^2	approx 6%
ICI Flexible 1	1.00 kgf/cm^2	90%
ICI Flexible 2	0.46 kgf/cm^2	140%

We believe, that with proper consideration of the physical properties of the films, any combination of the acrylic and PVC films in Table 2 will be able to seal grain storage silos satisfactorily. To illustrate how to select materials to meet a particular specification from data derived from this study:

It is essential that the tensile strength of the film must be greater than the peel strength, and that the PVC film should be weaker than the acrylic film. Consider a proposed combination of a 100 micron film of Envelon and a

500 micron film of Gaseal. Figure 7 gives details of tensile strength for varying thicknesses of film. Extrapolating the data for Envelon, a 100 micron film is shown to have a tensile strength of 47 kgf per cm^2 yielding a strength of 0.47 kgf per 1 cm wide strip. A 500 micron Gaseal film has a strength of 28 kgf per cm^2 yielding a strength of 1.35 kfg for a 1 cm wide strip. It is therefore three times as strong as the Envelon film.

From Table 2, a 700 micron film of Gaseal is shown to have a linear resistance equal to a force of 1.6 kgf per cm; a 500 micron film will be expected to have a value of 1.2 kgf per cm. The adhesive or peel strength of Envelon is of the order of 0.9 to 1 kgf per cm.

On this basis, the combination of Envelon at 100 micron dried film thickness (DFT) and Gaseal at 500 microns DFT would be satisfactory.

FOAM SEALANTS

The same consideration of physical properties can be applied to the foam sealants. They are not structural materials that can be expected to resist the forces applied by a silo when it is being loaded. Therefore, the ability to yield is a critical property.

The forces applied can be tensile, shear or compressive. In Table 3, the tensile strength and percentage elongation of five commercial foams are presented. Three are rigid foams, while two are flexible. The rigid foams do not actually elongate under stress. They begin breaking immediately a stress is applied.

In adhesive or peel strength tests on the foams, a force was applied in a shear mode at 90° to the adhesive bond. Four foams steadily sheared from the surface failing cohesively, while the fifth failed suddenly over the entire surface area of the bond. Foams which will partially fail resemble our peel under stress philosophy for films and would be technically preferred.

We believe that the flexible foams are the most appropriate for sealing as they will yield under strain and the elongation is easily accommodated by the sealant membrane applied to the surface of the foam. A rigid foam which snaps can be expected to cause immediate breakdown of that sealant membrane.

CONCLUSION

We believe that the ability of sealants to give under load is critical. The study of film and foam properties presented here has enabled us to develop a way to maximise this characteristic by proper specification design.

FIG. 2 ELONGATION VS THICKNESS at 0°C

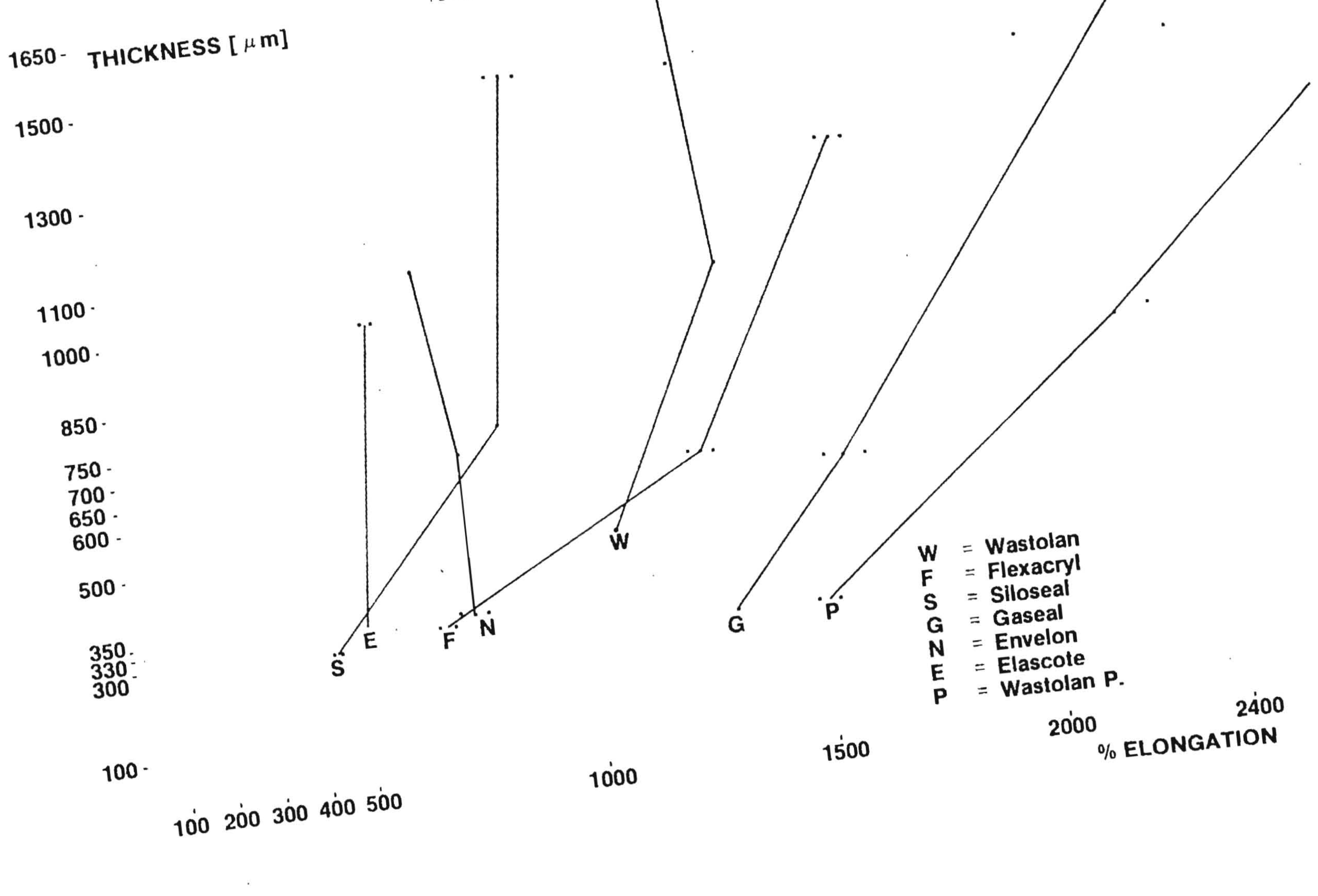


FIG. 3. ELONGATION VS THICKNESS at 25°C

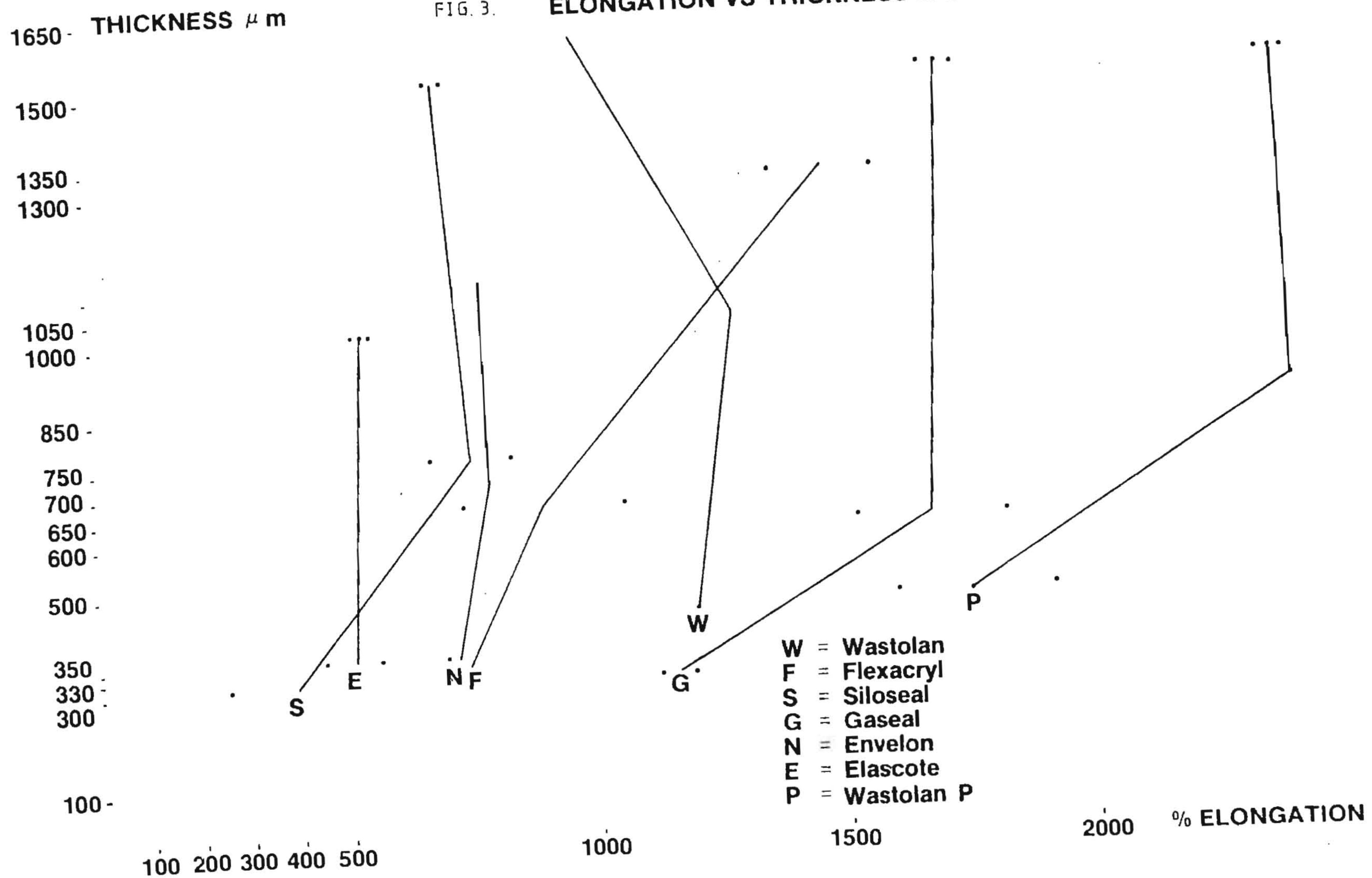


FIG. 4. ELONGATION VS THICKNESS at 50°C

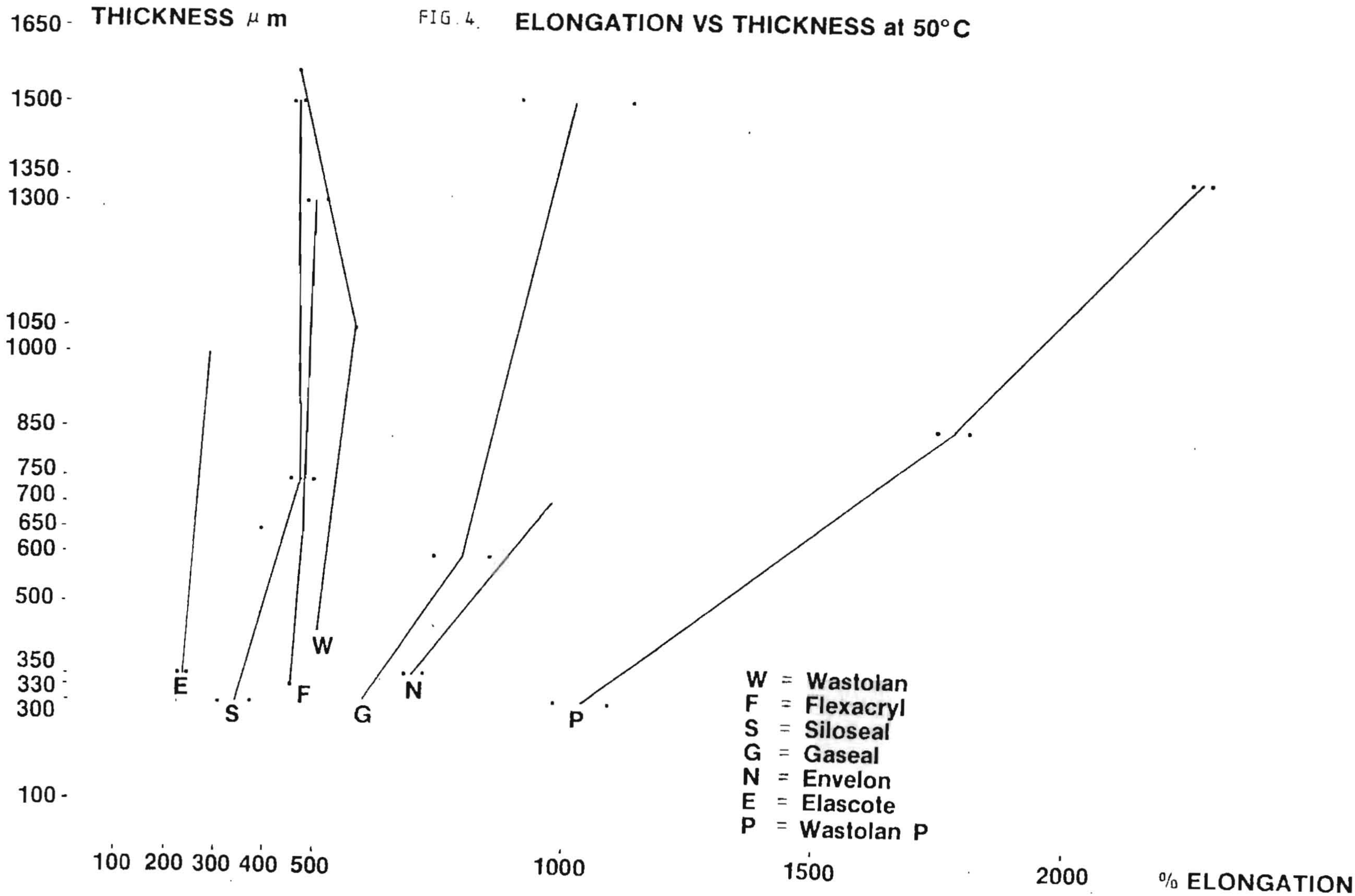
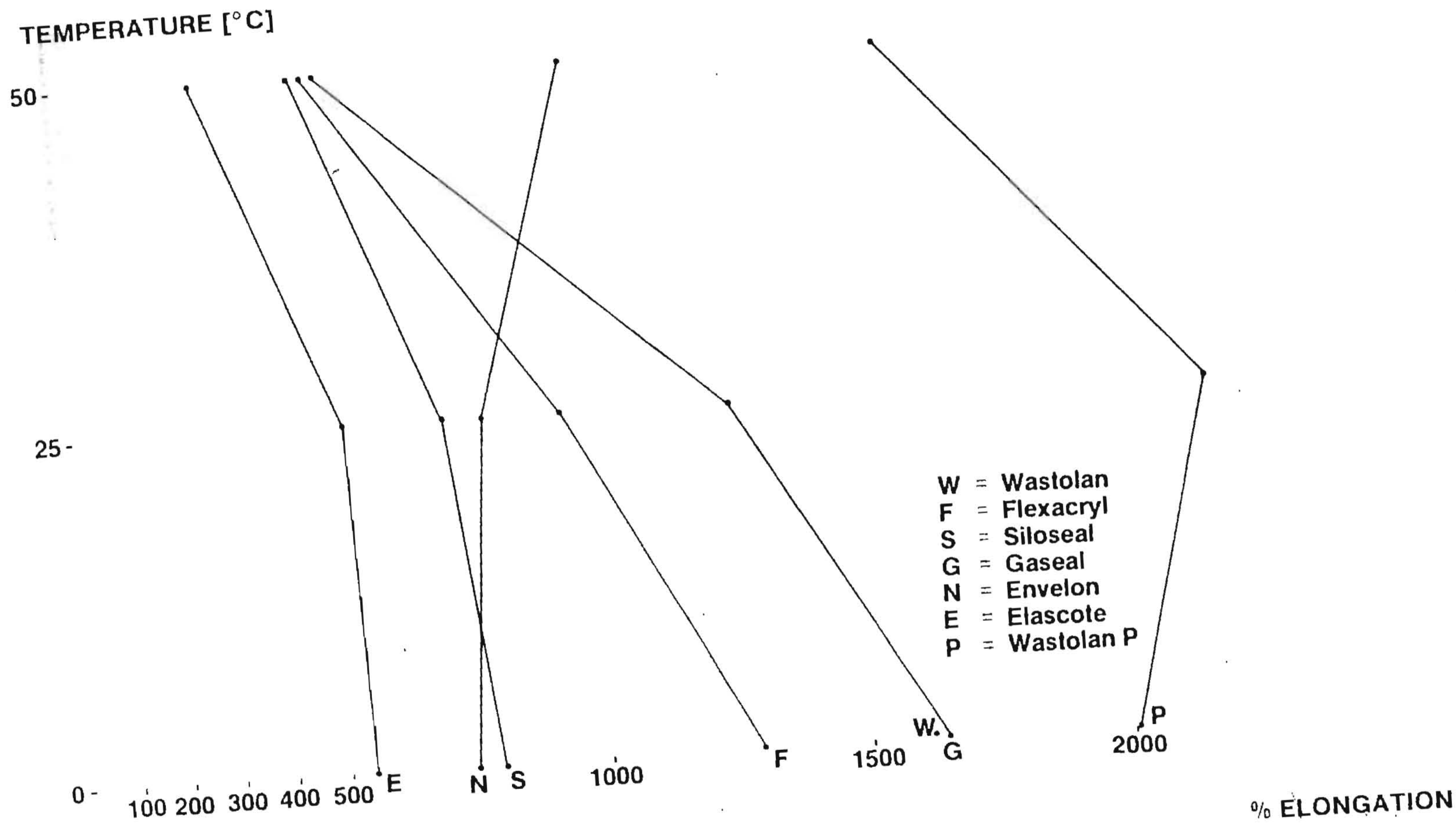


FIG.5. **ELONGATION VS TEMPERATURE at 700 microns D.F.T.**



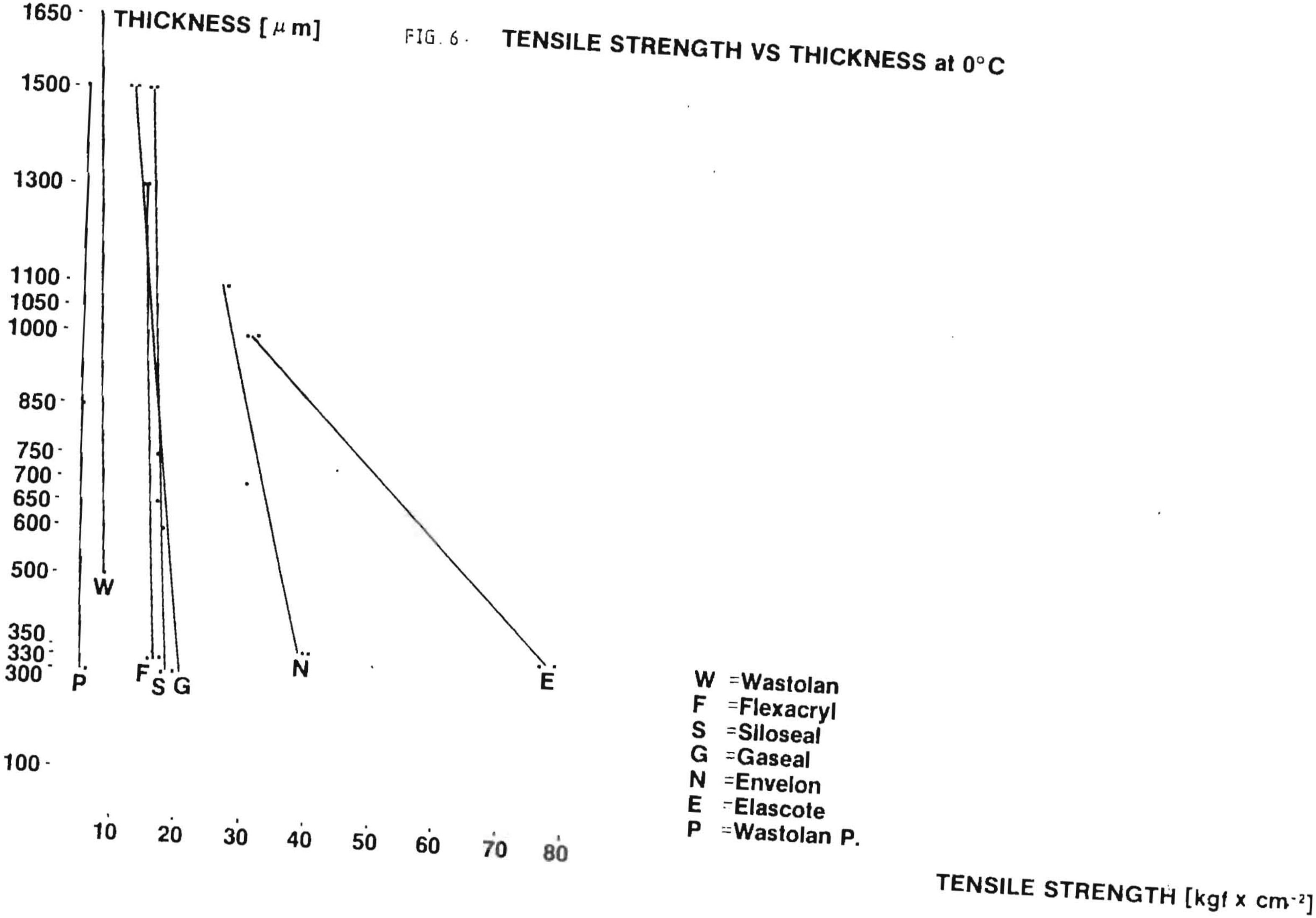


FIG. 6 · TENSILE STRENGTH VS THICKNESS at 0°C

FIG. 7. TENSILE STRENGTH VS THICKNESS at 25°C

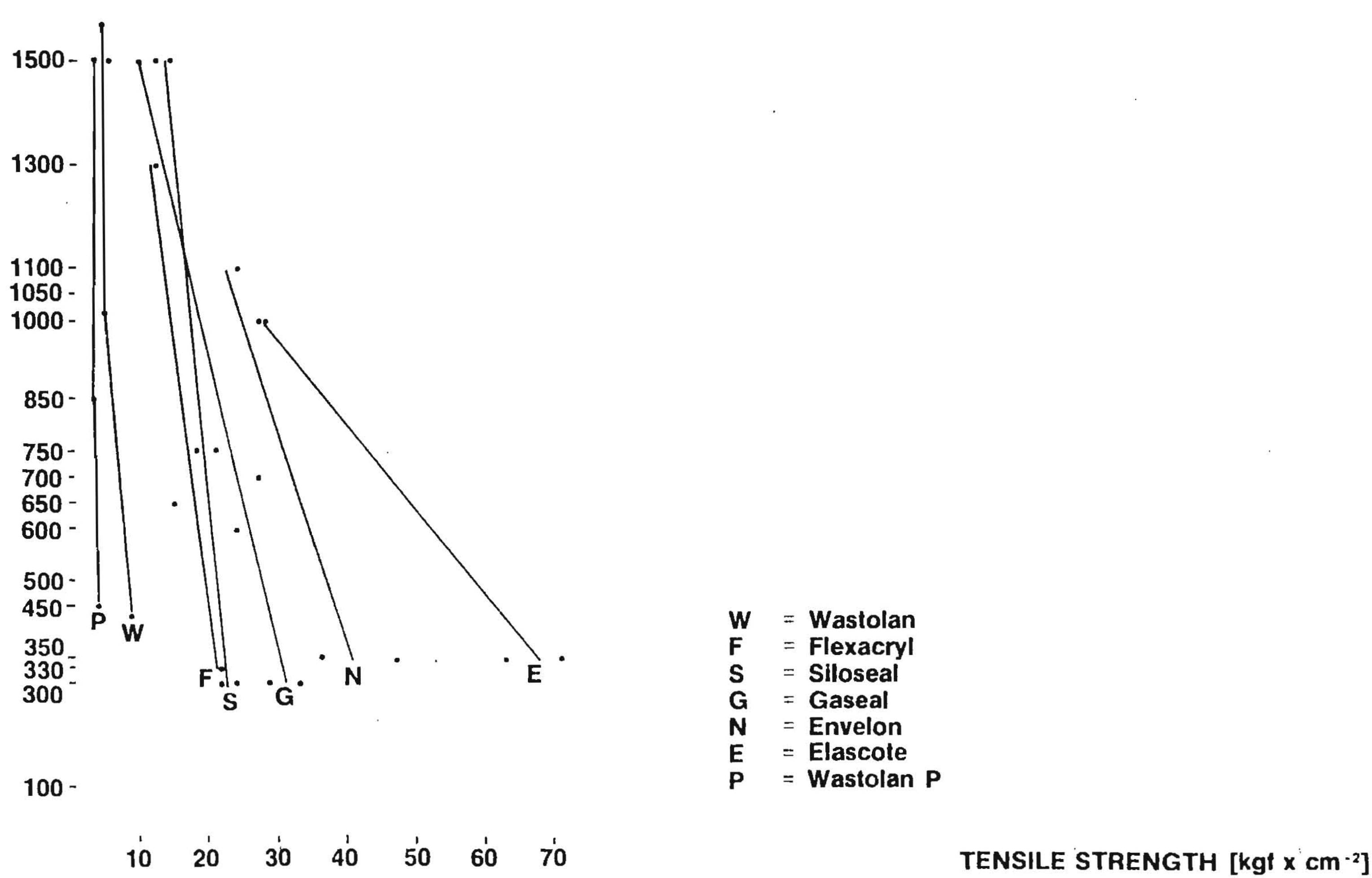


FIG.8. TENSILE STRENGTH VS THICKNESS at 50°C

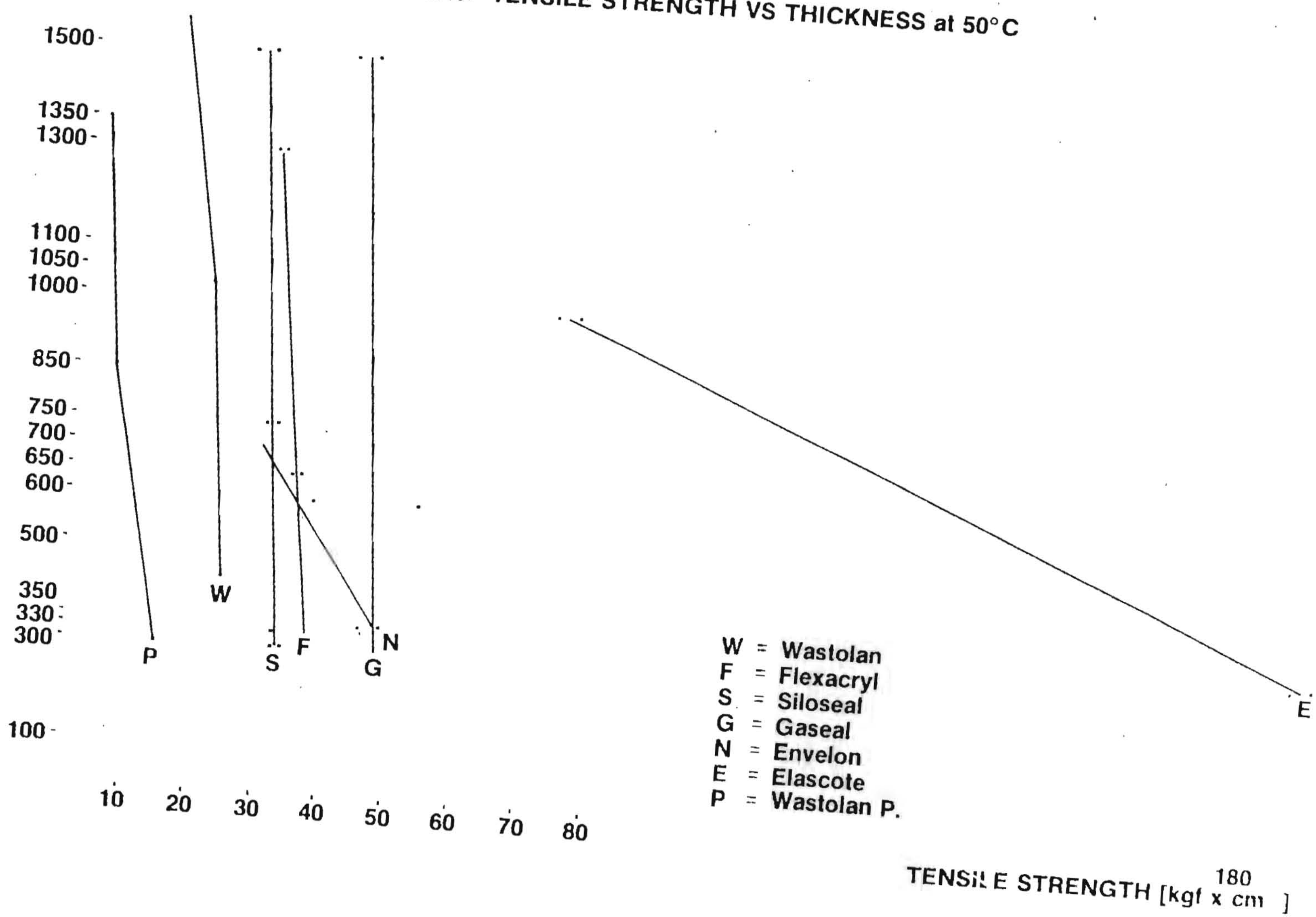
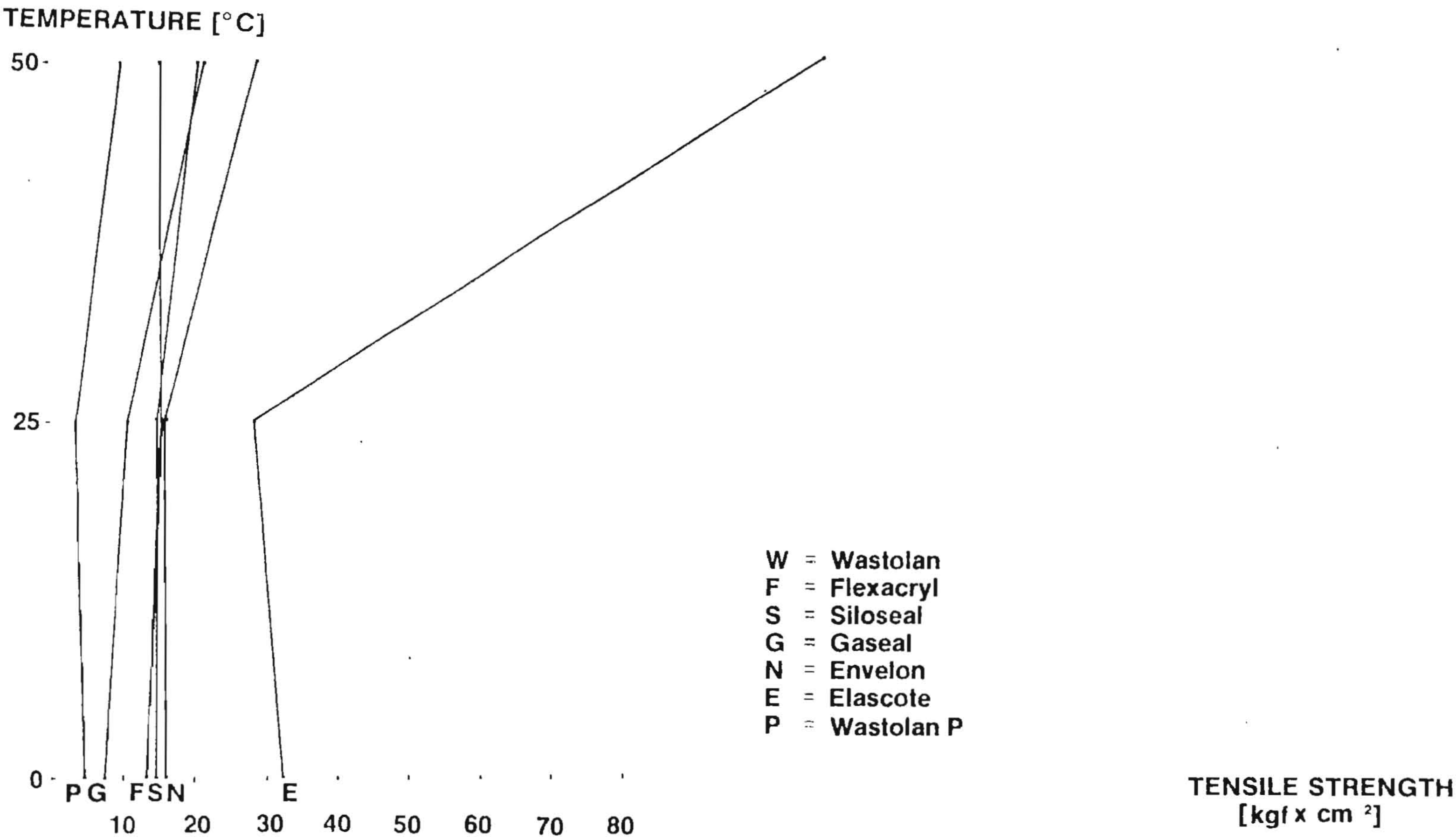


FIG. 9. **TENSILE STRENGTH VS TEMPERATURE**
at 700 microns D.F.T.



CONSTRUCTION AND OPERATIONAL PROBLEMS ARISING IN SEALING LARGE CAPACITY HORIZONTAL STORAGES

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ABSTRACT

This paper describes development work undertaken by New England Industries Pty. Ltd., in the design and construction of large horizontal grain stores incorporating controlled atmosphere or fumigation techniques, automatic outloading and aeration. It has been accepted as a precept that storages must be capable of controlled atmosphere storage and total store fumigation, but it is considered pointless or futile having this capability unless all other essential functions of a modern grain storage facility can be undertaken without the need to regularly break the gastight integrity of the store to permit these operations to be carried out. The developments so far undertaken have culminated in a low cost "total concept" 60,000 tonne capacity storage at Moura in the state of Queensland due for completion in 1983. This storage is thought to represent a world "first" in its design concept and it is expected to be completed for less than 50% of the cost per tonne of more conventional vertical storages with the same functional capabilities.

Some of the problems associated with inloading, outloading and aerating grain in the sealed and gas charged situation have not been finally solved, as the store is not yet completed; this paper identifies the main problem areas remaining.

INTRODUCTION

The costs of vertical steel or concrete silos with a range of operational capabilities are now increasing to uneconomic and unacceptable levels. Since 1979, New England Industries Pty. Ltd. have attempted to develop low cost storage systems capable of providing the range of capabilities required in vertical silos. The functions required of grain storage systems are not static, but are subject to change as new materials and technologies become available and as market demands and statutory requirements change also. The functional capabilities of storage systems must therefore be both comprehensive and capable of modification. Taking this into consideration, it was found that the functional parameters used in this development were not necessarily compatible, they could appear to be contradictory. For this reason, as will be shown later, not all the functional requirements have so far been incorporated into the development.

The operational functions included:

(a) Mechanical handling for inloading and outloading at rates between 400 tph and 2,000 tph.

(b) A capability to control internal store atmospheres or for total store fumigation to meet the most stringent requirements for minimal or nil levels of insect infestation.

(c) Remotely controlled operations of inloading and outloading of grain, if necessary simultaneously, whilst the storage is sealed or charged with gases for fumigation. It is assumed that personnel could not/would not enter the storage to control the handling operations.

(d) Provision of inbuilt aeration systems to simultaneously cool and dry high moisture grain and to be able to carry out cooling and drying at the same time as fumigating and handling into and out of the store.

(e) Segregation of grain using free-standing dividing walls, capable of being loaded to a full height one side and with no supporting grain on the other side. It is necessary also to design free-standing walls and reclaim equipment in order that it can move past the walls from one area to another.

(f) Remote controlled monitoring of temperature; moisture levels; gas density and infestation levels.

(g) Safe working access to the most important mechanical equipment whilst the storage is charged for fumigation.

(h) The highest levels of safety and environmental protection to staff and others in the immediate surroundings of the store through control measures and safety systems.

THE PHASES OF DEVELOPMENT

The design and development has been a staged process with the various problems being identified and solved in discrete operations over a four year period.

During initial studies in 1979, a wide range of structural designs was analysed in order to arrive at the cheapest possible structure to provide permanent storage. An A-frame building design with low (2.8 metre) walls was selected. The concept is that an A-frame is the most economical covering of grain subject to its natural angle of repose. During the latter part of 1979, a 60,000 tonne storage was constructed at Trangie, N.S.W., for the very low cost of A $16.00/tonne of capacity. This storage has a 300 tph capacity inloading system, but it is not sealed and it does not have automated outloading.

However, the signficance of this first stage is that it highlighted the very substantial margin of capital cost available for sealing, automated outloading, segregation walls, and aeration when compared to the cost of the same capacity of vertical storage at A $150.00 per tonne of capacity but including the additional functions of outloading, aeration, etc. From this base of A $16.00/tonne, it therefore seemed feasible to develop a new type of permanent storage with considerable savings in capital cost.

In the years 1980-81, a 110,000 tonne capacity storage facility was constructed at Moree, N.S.W., and this became the first large horizontal storage in Australia in which special consideration was given to the structural design in order to maximise the benefits of controlled atmosphere storage and fumigation.

The store dimensions are 205 metres x 75 metres with walls 5 metres high. Grain is held to a height of 2.5 metres on the wall panels. Ridge height is 26 metres. Structural engineers for the project were D T Cohen & Associates. The basic sealing specification and test standards were provided by Banks and his colleagues from CSIRO and the sealing contract was undertaken by Programmed Maintenance Pty. Limited, on a guaranteed performance basis.

The following features were included in the design, some only minor in operational importance.

(a) Design pressures:

(i) Internal negative pressure of 2500 Pa. This is identical to the design live load, and it is considered they do not act simultaneously.

(ii) Internal positive pressure of 3000 Pa which is added on to the design external wind pressure.

Excess pressure is vented using "Protectoseal" two-way valves which actuate at $\pm$ 400 Pa.

(b) End wall and side wall cladding is placed inside the girts and finished flush to the top of the wall panels. Consideration was given to fixing roof cladding to the underside of the purlins in order to prevent dust trapping, but was ruled out due to excessive cost.

(c) All internal structural steel members and purlins are fabricated with downturn flanges so as not to collect dust.

(d) The floor finish is 50mm thick asphalt placed to highway standard. This finish has proved durable to oilseeds over a seven year period in other storages. It is found less prone to cracking and cheaper than concrete.

(e) Wall panels are pre-cast concrete slabs with the lower edge extending 300mm below floor level for protection from vermin.

(f) As far as possible all dust collection areas such as ledges were eliminated in the design.

(g) Gas venting is by natural air flow from a series of end wall and roof vents designed by the CSIRO and intended to ensure the structure will undergo several air changes in 24 hours. The vents are 14 metres above ground level. If natural venting is not successful, extraction fans will be installed.

(h) All inloading can be remotely controlled.

(i) The internal electrical system was designed to prevent corrosion from phosphine gas.

The integrity of the sealing of the store has been tested by CSIRO and found satisfactory under empty conditions. Unfortunately it has not been tested with grain because of the serious and continuing drought in that region. It has been loaded once only for a short period with 46,000 tonnes of wheat, sufficient only to commission the mechanical handling equipment.

Once again this store design did not include automated outloading equipment or provision for segregation. These two omissions led to considerable, and quite valid, criticism of the design and this has provided a further incentive to continue the design development.

Nevertheless, a number of the design features included in the Moree store, particularly relating to sealing and grain hygiene, have led to important lessons being learned from this development:

(a) As with Trangie (1979) the floor space is too large and the walls are too low for optimum economy when providing for automated outloading.

(b) No dust extraction equipment is provided, but it is interesting to note that under normal light wind conditions the building is kept sufficiently free from dust during inloading only using the end wall vents. If no vents are open, the dust becomes dense in a very short time.

(c) The purlins attach to cleats fixed to the top chord of roof trusses which create dust collection areas. This problem can be eliminated by placing the cleats to the side of the chord member.

(d) Pre-cast concrete wall panels are not entirely satisfactory as they may require sealing to prevent possible corrosion of the reinforcing steel, this is a matter still under study.

FULLY AUTOMATED OUTLOADING SYSTEMS FOR HORIZONTAL STORAGES

Studies in 1980 suggested that no efficient or proven system existed in Australia and enquiries were then extended to North America, Eastern and Western Europe. Three different systems were subsequently inspected and of these, the Nordon T.M.S. equipment in France was judged to best suit the requirements of unloading from a sealed storage. In May 1982 an inspection of this equipment in operation was carried out by engineers from Queensland State Wheat Board; mechanical engineering consultants, H Platt & Associates; and the structural engineering consultants, McWilliam & Partners. This inspection confirmed the suitability of the equipment and subsequently an order was placed after completing the design of a suitable structure; details of which are described below.

Several ideas for free standing segregation walls have been investigated by McWilliam & Partners. The principal limiting factors being foundation requirements and required wall heights of 10-15 metres. There are two possible solutions, both in reinforced in situ concrete. In general terms they can be described as:

(a) An inverted T tied into bored pier foundations.

(b) A profile of large vertical curves - this method is in use in France.

It is clear that portable pre-cast segregation wall units can be constructed where only low wall heights are required.

During 1982-83, designs were started for a 60,000 tonne capacity store at Moura, Queensland. The problem areas for the design of this facility will fall into two distinct categories:

(a) Separate operations of:

i. Controlled atmosphere storage or total store fumigation, without any grain movement.

ii. Aeration in an unsealed situation.

iii. Inloading and outloading in an unsealed situation.

(b) The ability to accommodate simultaneously, in a sealed situation, any two or all three of the following operations:

i. Phosphine fumigation or controlled atmosphere storage.

ii. Inloading and/or outloading.

iii. Aeration.

Design problems associated with the separate operations of the store are now largely solved. Nine proposed designs were analysed and costed before a final decision was taken on the Moura structural design. The great difficulties were in selecting wall height, internal width, foundation and column design. The selected design was intended for a new storage facility, but it now seems certainly possible that a range of existing stores could be modified to provide the same facility and accommodate the same or similar equipment.

For controlled atmosphere fumigation only, design problems have been reduced by changing to a new load bearing wall material "Siloclad" developed especially by Lysaght Brownbuilt Industries for the project. This is a deep rib vertical profile in 1.6mm high tensile galvanised steel. Purlin cleats have been reduced and the floor area reduced by 22% per unit of storage volume compared to Trangie.

To accommodate the unloading equipment, all aeration ducting (for use in an "unsealed" situation) is underfloor and the inlet ducts are below the concrete perimeter beam. Reversible fans are to be installed so that they may be used also for downward purging of fumigant gases.

The aeration ducting is also designed to provide drainage to the outside if washing down of the store is required.

The major changes in store design relate to the automatic outloading system provided by Nordon TMS of France. This equipment was chosen because:

(a) It is already proven;

(b) It can deal with level or inclined grain surfaces;

(c) The equipment can be operated remotely by Programmed Logic Control;

(d) It is accessible in the event of breakdown;

(e) It is programmable for different loading rates and has inbuilt sensors for following grain levels;

(f) It can be used for spreading grain as well as reclaiming.

The design changes required include:

(a) Sufficient headspace to allow longitudinal travel;

(b) Structural members to carry the equipment (which weighs 20 tonnes);

(c) Gantry rails for support of unloaders;

(d) The joint between wall and floor to be self cleaning;

(e) Construction of a series of continuous in-floor gravity hoppers and valves feeding to the underground reclaim conveyor. Initial design studies were directed towards an above ground fabricated steel walk-though tunnel with side valves feeding a drag conveyor but this idea was later abandoned.

The problems of integrating controlled atmosphere storage, fumigation, inloading/outloading and aeration still largely remain. There is no evidence that the simultaneous carrying out of these operations has been attempted previously in horizontal stores. The major operational problem is the control and containment of toxic substances such as phosphine. The safety aspects are vital considerations and control must be to such a degree that there is no possibility of leakage that will endanger employees. It is anticipated that control and containment of carbon dioxide and nitrogen will present far less hazard than phosphine, accordingly the development work and the gas removal specification will be based on phosphine.

The problem areas for containment and control that are presently being investigated are:

(a) Inloading - Transfer Chute to Ridge Conveyor

(b) Inloading - Wall Grain Levels Coupled to Trippers

(c) Placement or Retrieval of Fumigant Strips or Pellets

(d) Testing for Infestations

(e) Monitoring of Temperature, Moisture Levels and Gas Density

(f) Remote Control Operation of Reclaim Conveyors

(g) Design of Gas Tight Valves to Underground Reclaim Conveyor

(h) Remote Control Valve Operation

(i) Positive Fresh Air Pressure in Tunnel to Permit Maintenance

(j) Fresh Air Venting of Outloading Elevator Shaft

(k) Purging Gas of Outloading Grain Stream and Prevention of Excess Gas Escaping from Storage

(l) Monitoring Equipment for (k)

(m) Aeration

Some possible solutions have already been identified and it is hoped they will all be successfully implemented by the end of 1983.

ENGINEERING ASPECTS TO BE INCORPORATED INTO DESIGN OF NEW STORAGES AND MODIFICATION OF EXISTING STORAGES FOR CONTROLLED ATMOSPHERE

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ABSTRACT

This paper sets out various modifications to a standard design structure which should be carried out if a programme of sealing is to be implemented in the near future. The costs of these modifications when carried out during construction are much less than the costs associated with altering and modifying an existing building. All of the modifications keep in mind good storage practice and are in no way detrimental during an interim period. The items listed hereunder are to serve as guidelines and cover structural, ventilation and interior treatment.

INTRODUCTION

The great majority of storages operated by Co-operative Bulk Handling Limited (CBH) are of the flat, or 'horizontal' type. Figure 1 shows the typical 'A' Type storage, having concrete walls, concrete floor and truss or portal frame roof structure and corrugated, galvanised iron roof sheeting. Figure 2 shows a typical buttress wall type storage, known as 'E' or 'G' Type. In this instance the floor is of compacted form with bitumen topping. Walls are inclined timber 'A' frames, sheeted in horizontal running, corrugated iron. The building structure for roof and floors is independent of the wall system. Both types of construction have been utilised throughout the C.B.H. system for a number of years and, in essence, are very similar to many storages constructed around the world for handling grain, either in bag or bulk.

DESIGN CHANGES TO ASSIST SEALING

It is easy to make minor changes in the design of these storages so that they can be more easily sealed after construction than the current storages. This paper details some changes made to CBH designs to assist sealing after construction.

Changes to Ventilator Design

Existing storages were ventilated throught the roof with a number of convection-type ventilators. When the storage is to be sealed, opening and closing these ventilators would be a difficult and expensive undertaking. The design of ventilators was, therefore, modified to allow for the later addition of a sealing support plate (Fig. 3). At the time when sealing is undertaken, the plates would be attached to the underside of the ventilator and sealant material applied. They may be removed for ventilation at a later date if so required.

Changes to entry into storage from inloading Conveyor

The overhead belt conveyor from elevator or from distant pit area generally passes through the wall into a penthouse from which the grain flow is either split or distributed on to overhead conveyors. A readily sealable panel around the belt was considered difficult to achieve. Therefore the conveyor is now elevated above the roof line and spouting passes through the roof to the distributing belts. a simple manually operated slide plate is provided in the spouting and the outside edges of the spout sealed against a support platform. Access by personnel is through a lockable, sealable man-hatch (Figs. 4 and 5).

Elimination of Bird Netting

In previous designs of storages ventilation openings were fitted around the structure under the eaves. This area is sealed off with sealed storages. It has been the practice to fit moulded nylon netting there to prevent entry of birds. This has now been replaced with inclined galvanised sheeting which may be readily sprayed with sealant to give a gastight seal at the eaves.

Modified Sealing of Main Doors

The main doors of the storage are used for access of mobile grain handling equipment during outloading. As the storage is filled and the grain heap reaches the floors, they are closed and sealed from the inside. Previously, flaps of rubber belt had been provided as a rough seal at the door to prevent grain escape at the hinges. These are now eliminated to make the work of sealing the hinge line easier. The doors are also fitted with a 'last man out hatch' so that whichever is the last access door, sealing can be fully carried out from the inside. The operator crawls then through the sealable manhole hatch and bolts it into position (Fig 6).

Installation of Lighting

Previous storages were found to have sufficient lighting from the natural light coming through the eaves, doors, ventilators and translucent sheeting (skylights). With advent of the design changes, as described in this paper, it was found necessary to install artificial lighting along the conveyor gallery. The lighting is D.I.P. (Dust Ignition Proof) and suspended from the main portal frames. Lights are used during operation and maintenance of the conveyors and as general lighting over the area during outloading by front end loaders. Alternatively, installations in remote areas without power can utilise light panels with sealed edges to be closed and sealed whenever sealing is carried out. (Light Specification:- D.I.P. HAZLUX No 3.400W High Pressure Sodium DS25C-225B).

Addition of Girts to Allow for Installation of Fans

When the additional sealing preparations described here are made, it is found that the storages are part way to being fully sealed and as such, air movement within the storage is reduced. Loose fines and dust from the grain remains in suspension in the air, particularly during full sealing stage. It has been found that the installation and use of reversible fans in each of the gable ends of the storage disperses these fines. Fan direction is operated in accordance with prevailing wind direction. For a storage capacity of 27 200 tonnes (approximately 35 000 m^3), fan capacity of 170 m^3/min^{-1} to 180 m^3/min^{-1} each is satisfactory i.e. air 'in' equals 340 to 360 m^3/min^{-1} with equivalent air exhausted at the other end of the storage. Some strengthening of the area where the fans are installed is required to provide adequate structural support. After storage has been fully sealed, changes in fan capacity are not required. In addition to the fans, ventilation doors are provided in the apex of each gable end (Fig 7). In Co-operative Bulk Handling installations the ventilation door is fitted behind a retractable monorail utilised when servicing motors and other equipment.

Additional Purlins for Strength on Roof Sheeting

Normally purlins are installed on our recommended pitching consistent with roof loading and type of sheeting. It is found, however, that with the additional loads of sealing material plus the requirement for operators and sealing personnel to move about the roof it is necessary to decrease the purlin spacing slightly. To make the roof safer to walk on and to reduce the headspace volume in the storage the pitch has been reduced by 10% from the normal design specification. Safety eye-bolts are provided at ridge line for maintenance workers to attach safety lines when working on roof (Fig 8).

Elimination of Translucent Sheeting

Many of the old storages had translucent sheeting at approximately five metres staggered pitch with one section of roof sheeting being replaced with translucent sheeting. The light input was sufficient for work within the storage. However, the heat input was found to be high. Translucent sheeting on existing storage has to be sprayed over with a clear material for sealing. With new storages, the sheeting is simply eliminated.

Provision of Colourbond Roofing

To lower the heat input to the air within the storage, white colourbond roofing material has been selected. Colourbond is a corrugated iron sheeting with a factory-applied coloured coating on one side. Tests have shown that substantially lower day temperatures ($45^{\circ}C$ compared to $60^{\circ}C$) are experienced in the headspace of storages so constructed. Gas losses due to the daily temperature changes are therefore considerably less. (For additional data on tests carried out by Co-operative Bulk Handling Limited for heat reflective materials, see Paper No. 20 (C. Barry) - this Symposium).

Gallery Conveyor Painting

With the loss of natural light in the storages from the elimination of skylights, attention was given to the colours for equipment and walkways within the storage. Originally, Co-operative Bulk Handling Limited conveyors were painted glade green with grey structure and galvanised walkways and black grids. This has now been modified to galvanised equipment including walkways and grid so as to form a better reflector for what light is available. Machinery such as trippers, conveyors, head etcetera have been painted white enamel. Pulley shells and rotating items have been retained at Industrial yellow and electrical equipment remains orange.

CONCLUSION

It can be seen that the design changes facilitating sealing are minor and simple to carry out. Incorporation of such changes in the design stage makes the work of sealing subsequently both cheaper and more reliable.

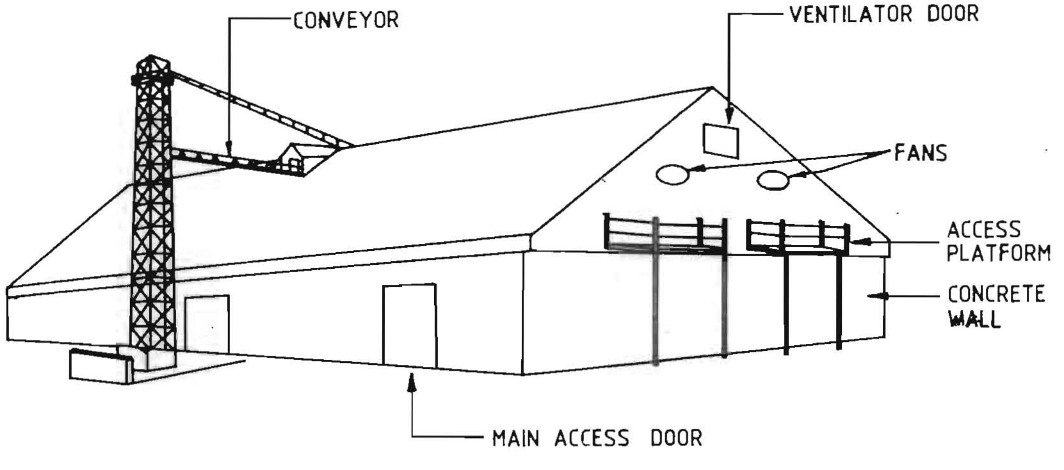

FIG. 1. A TYPICAL 'A' TYPE HORIZONTAL STORAGE AS USED BY C.B.H. IN WESTERN AUSTRALIA. DESIGN CHANGES ASSISTING SEALING ARE INDICATED.

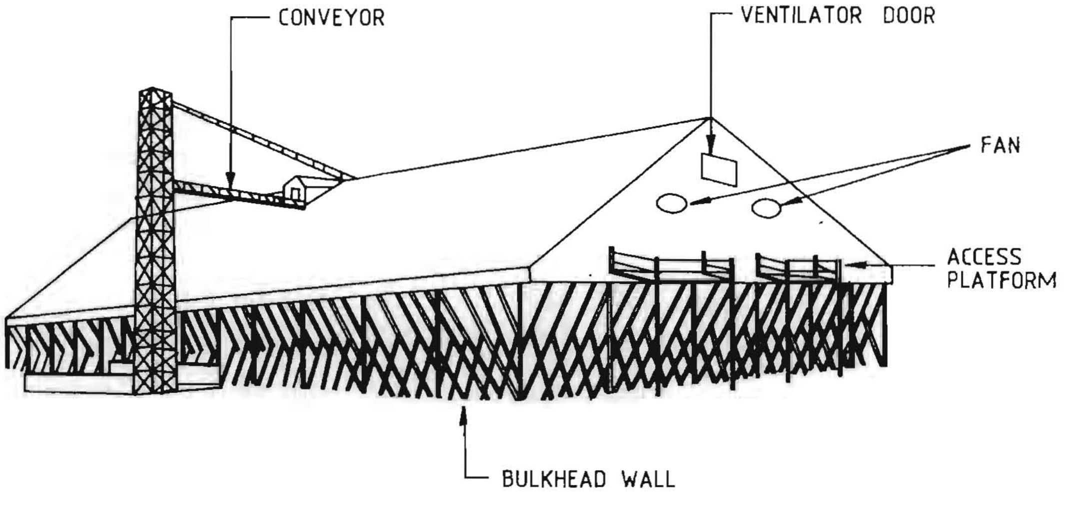

FIG. 2. A TYPICAL 'G' TYPE STORAGE AS USED BY C.B.H. IN WESTERN AUSTRALIA. DESIGN CHANGES ASSISTING SEALING ARE INDICATED.

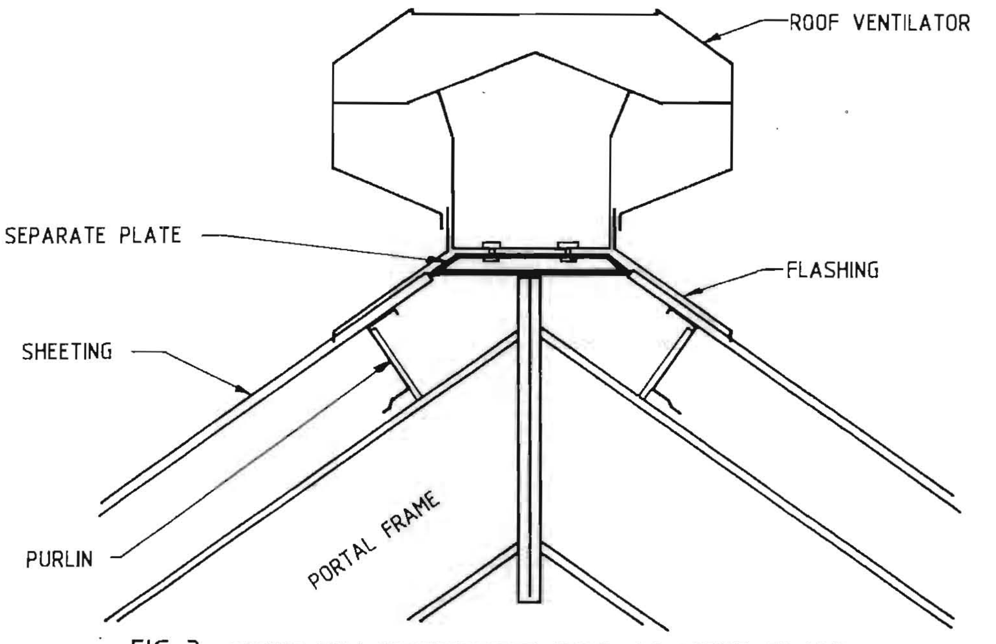

FIG. 3. MODIFICATION OF VENTILATOR DESIGN TO ASSIST SEALING

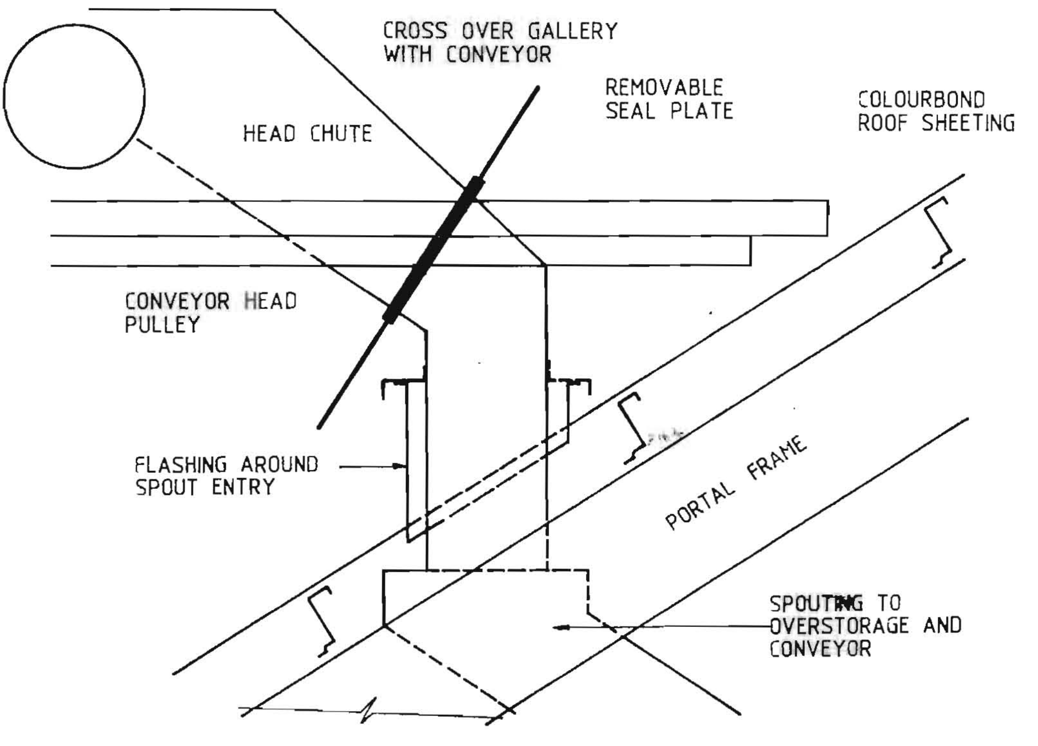

FIG. 4 REDESIGNED GRAIN ENTRY SPOUT

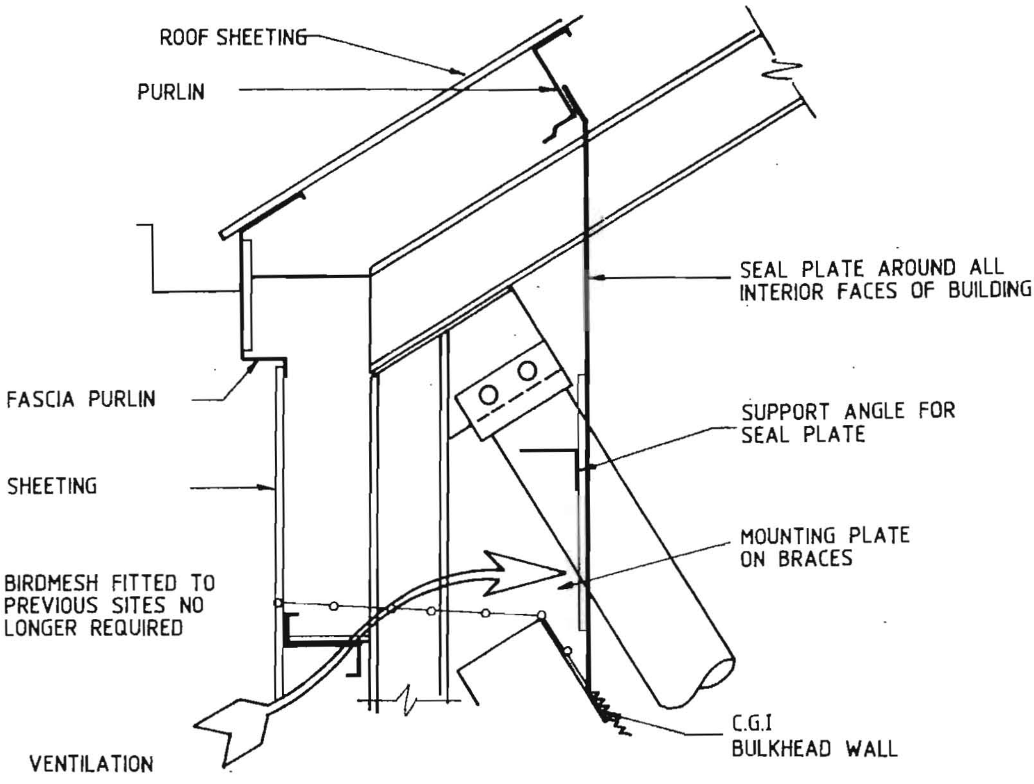

FIG. 5. DETAIL OF TOP OF WALL

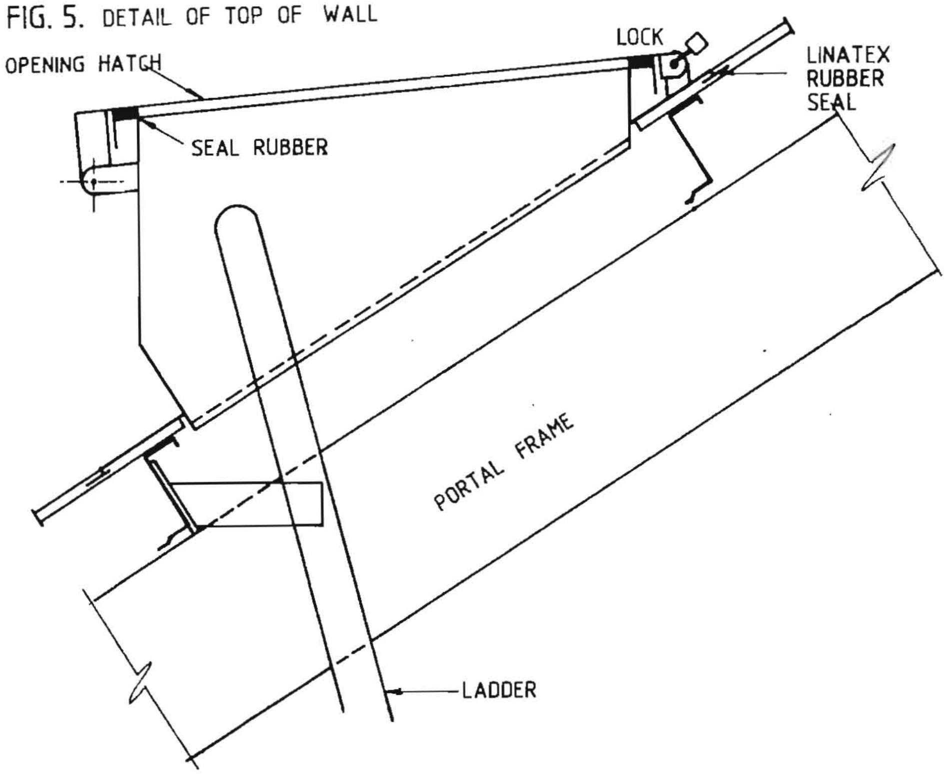

FIG. 6. DETAIL OF FLASHING AROUND CONVEYOR ACCESS HATCH.

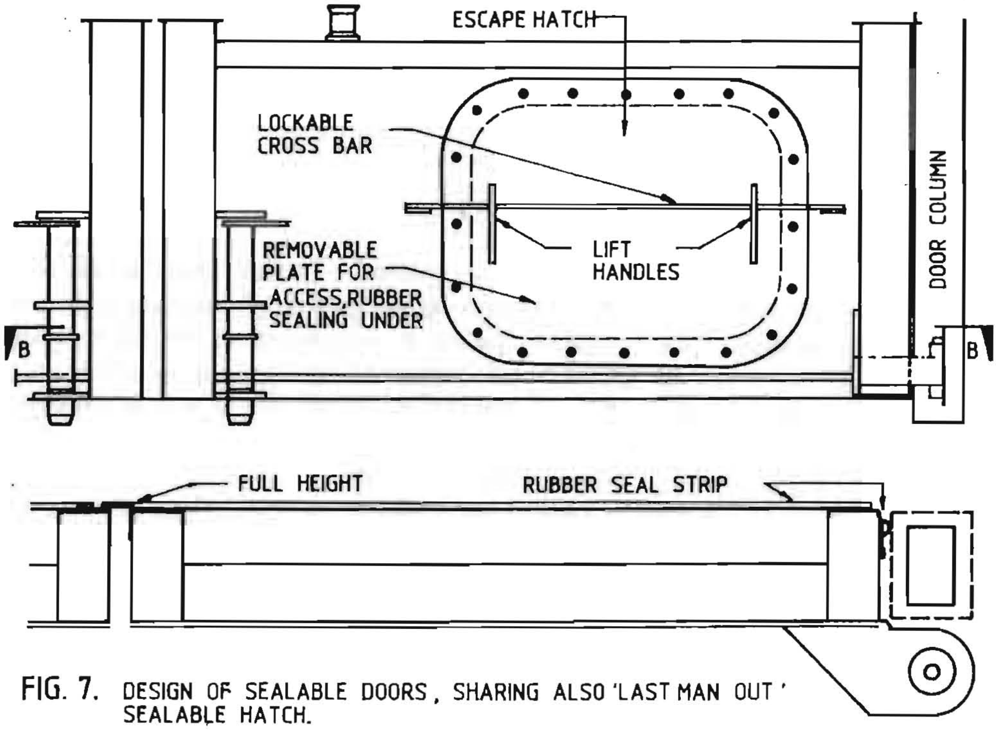

FIG. 7. DESIGN OF SEALABLE DOORS, SHARING ALSO 'LAST MAN OUT' SEALABLE HATCH.

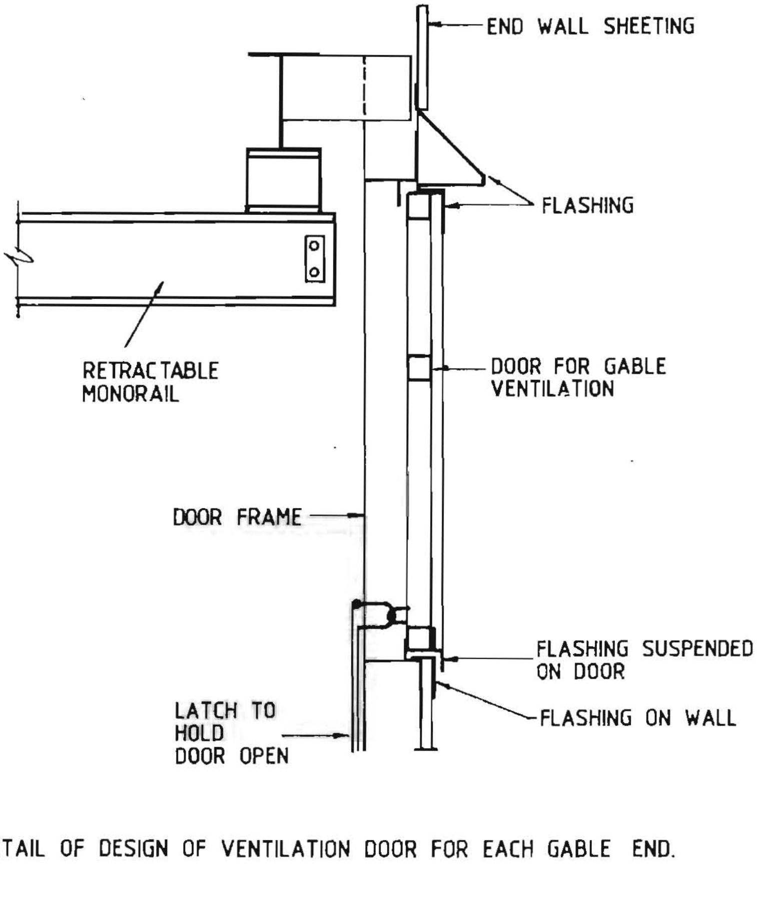

FIG. 8 DETAIL OF DESIGN OF VENTILATION DOOR FOR EACH GABLE END.

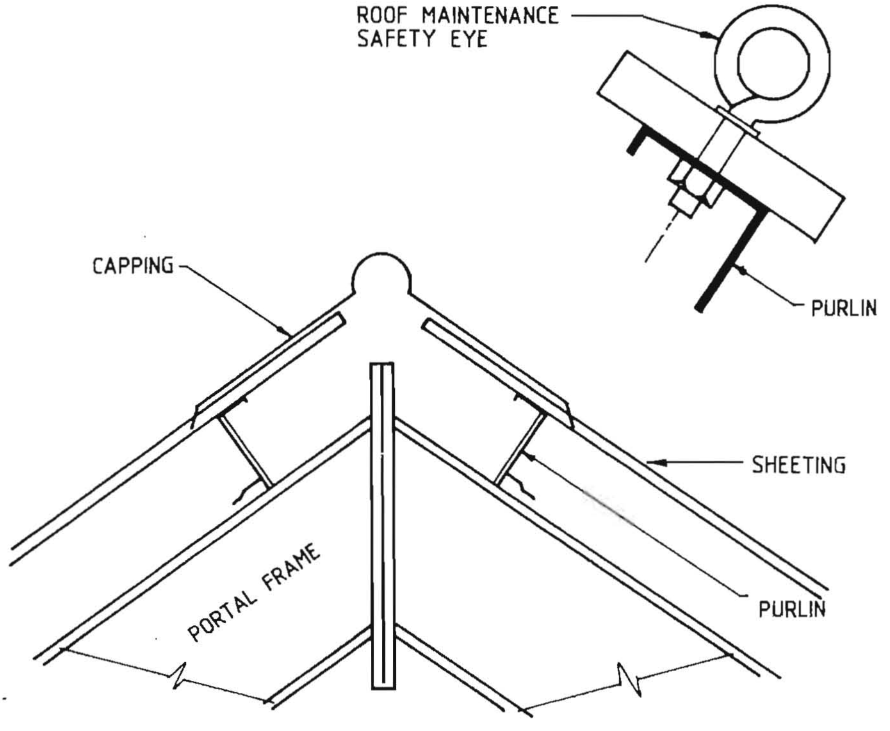

FIG. 9 . DETAIL OF SEALED RIDGE LINE SHARING FITTING OF SAFETY EYE FOR MAINTENANCE AND SEALING WORK.

DESIGN OF FUMIGABLE STORES IN THE TROPICS

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ABSTRACT

The design of grain storage buildings is related to function, location and available resources. Whilst resources in developed countries are relatively unlimited leading to high standards of design and construction, many developing countries lack both material resources and experience of design and construction. Basic design requirements for fumigable stores in the tropics are considered, together with standards for assessing quality and workmanship. Standards attainable are discussed in relation to who pays for, constructs and supervises the building. The difficulty of achieving acceptable standards is emphasised. Some difficulties are caused by non-availability of materials, but local materials have been successfully used as substitutes. It is concluded that caution is needed in laying down precise design criteria for fumigable stores when resources are limited. However the design of such stores needs urgent attention because of reports of total store fumigation in unsuitable buildings causing resistance to fumigation by phosphine by a variety of pest species.

INTRODUCTION

There are three important aspects to be considered in the design of buildings for the storage of grain. First, it is necessary to decide on the precise requirements of the building in terms of its function and the operations to be carried out within it. Second, whether the location of the building is likely to cause problems including the possible effects of climate on the design. Third, the availability of resources to construct and operate the buildings must be assessed, with special reference to materials, capital and the managerial and technical resources needed both for construction and operation.

It is particularly important that the resources available for construction and the subsequent operation and maintenance of stores be taken into account when buildings are designed. It is clearly quite impractical to provide design specifications which, for one reason or another, are unlikely to be met and, for this reason some designs would not be appropriate for many developing countries of the tropics. For example, the design proposed by O'Neil (1983) at this Conference is clearly based on a situation where resources are relatively unlimited and the major constraint is technical development.

In a large number of tropical countries the grain storage system is for bags. Bulk systems are often confined to port or mill installations where they form part of a complex that frequently can afford experienced and skilled management. Many of these port or mill units are linked to imported grain, whereas the main food grains produced and stored in the tropical zone are rice and maize, both of which are handled throughout the marketing chain in bags. Because of this, bag warehouses are ubiquitous throughout the tropics and likely to remain so for some time. However, it may be advisable to maintain a design that is flexible enough to handle both bagged and bulk grains. In these cases the problems of sealing entry doors, ventilation openings and grain handling systems are both more numerous and complex than in the case of large scale single grain type bulk stores.

A further factor is the significantly lower space utilisation of bagged commodities compared with the flat bulk stores or vertical silos. The designer of the fumigable stores for the tropics is therefore caught in a situation where the building volume per tonne of commodity is considerable larger than those current, for example, in Australia for wheat. Essential, but unused, space for passage ways, limitations on stack heights and general unsuitability of peaked bag stacks, result in increased building costs and a greater number of entry points. These factors alone indicate that a simple rescaling of storage designs based on those developed in Western Australia would be an unsuitable base for the tropical situation. To meet the basic design requirements it is advantageous to start at the first principles of a store design for bagged commodities in a developing country in the tropics.

Design Parameters for a Bagged Produce Store

(a) It will be used for in-sack storage. There is no clear justisfication in most developing countries to convert from bagged grain handling to bulk grain handling except in very special circumstances, which require individual consideration, but dual purpose bag/bulk stores may be an advantageous investment to cover future requirements.

(b) It will either be a primary or a secondary collecting point, therefore the delivery into the store will be slow, it may also be in an isolated situation.

(c) In order to effect adequate control of infestation, and possibly for other reasons, the size should be as small as economically practical. Economies of scale in store construction depend upon circumstances and generally extend up to 4,000 to 8,000 tonnes capacity. Therefore, there should be a tendency to build smaller units to achieve the desired flexibility.

(d) The store may be located in an urban area and used for seasonal or long term storage to maintain reserves for the urban population.

(e) It may operate as a transit store or a long term store wherever its location according to the seasonal supply from home grown production and from imported grains.

The most demanding requirements are for a transit store and these are:

(a) A length:width ratio of not less than 1:2.

(b) Doors to be provided one per 15m of wall length, preferably on each long wall of the building.

(c) Monolithic concrete floor with a vapour proof barrier.

(d) Ventilators in humid areas, or aeration where long term storage, with high moisture content grain, is contemplated. Ventilators should be at the eaves and the gables.

(e) Insulation should be provided by an eaves overhang, an orientation East-West and the outside painted white (the important part is to keep the outside white; therefore expensive, durable finishes which rapidly become stained or marked with dust, are less likely to be effective than simple whitewashing which can easily and cheaply be renewed as the reflective surface deteriorates).

(f) Masonry, concrete block or brick walls.

Where there are no constraints on the resources available, design parameters should be to the highest standards of engineering and building design and practice. Under these circumstances, the design of a fumigable store would be essentially the same wherever it was built. However, where resources are severely constrained, the design must take account of the limited facilities and resources that are available. However, there is no typical developing country; there is instead a range of conditions from those akin to industrialised countries where all the required resources are available in a descending progression to the very poorest country where few, if any, essential resources are available.

Generally the training of storekeepers and operators has been directed towards the use of phosphine for fumigation. If there is to be any substitution with inert gases for controlled atmosphere storage, carbon dioxide is likely to be the most commonly available. Both gases require a treatment time of 4-10 days as a minimum and therefore this defines the sealing standard of a fumigable store. However this indication is not a practical measure for site inspection during construction and commissioning, or a suitable method for management to check the maintenance of the sealing standard, both of which require the addition of a simple test that has been verified against the leakage rates of the gases employed and infiltration of atmosheric oxygen. The pressure delay time has the advantage of simplicity but as Banks and Annis (1980) have noted, it is as yet incompletely verified. It is however likely to remain the most practical method for design and more important the standard of inspection and final acceptance of the building as well as a management tool to indicate a need for building maintenance.

During modifications required to a conventional building for grain storge, any particular aspects needed for fumigable stores could be incorporated simply and cheaply into the building at the design stage. Sealing design and materials should also be included in any package for the supply and erection of fumigable stores offered by donors. Caution, however, should be exercised in relation to specialised application machinery and techniques especially for construction at isolated sites. Adhesives and mastics, silicone rubbers with extra lap fastenings for wall and roof sheets should, as far as possible, be the norm. The temporary closure of doors and ventilators should be with PVC tape, butyl or epoxy mastics which, whilst unsuitable for long-term storage may well be quite satisfactory for short periods.

The attainable standard of construction often depends upon who pays for the building construction and for the operation; i.e. whether this is a donor agency or a local organisation. Most aid donors operate on the principle of tied aid in so far that a high percentage of materials and equipment shall come from the donor's own country. In this case, it is not difficult to arrange a supply of good quality steel frame type industrial buildings. Basically these are perfectly adequate and require only minor modifications to design to make them suitable for in-sack storage. Indeed, donors should be encouraged at all times to supply the highest quality buildings in order to reduce, as far as possible, local requirements and costs for maintenance, although care is needed to ensure that the main items needing maintenance and replacement are ones that are available locally, choice of cladding pattern is one such example. Some donors also pay for the building and provide supervision for construction. This does not necessarily mean the buildings will be constructed to the same requirement and standards as in the donor's own country, but at least their presence on site is conducive to a higher standard of construction. Where a local building contractor is to construct the store then building standards can be extremely variable. Whilst there appears to be little difficulty in the construction of a Hilton Hotel or a convention centre, it is often very difficult to ensure the building of simple grain stores to basic construction standards.

Basic Requirements for Design of Fumigable Stores

Fumigable stores generally require most of the design features for general purpose stores. This means that in addition to providing adequate protection from rain and ambient humidity produce within the store must, as far as possible, be protected from fluctuations in temperature. These not only cause negative and positive pressures which will result in air being pumped into and out of the building, grain deterioration through high temperatures are related to the highest temperatures within a cycle, not to the median. If effective fumigations are to be carried out then losses of gas through ventilators, doors, the walls and roof must also be minimised so that adequate concentrations of fumigant gas can be maintained for sufficient periods of time. This is of particular importance for fumigations involving the use of phosphine where the duration of exposure is of more importance than the achievement of high concentrations (Bell *et al*, 1977).

Where the local organisation is responsible for the design and construction of buildings it becomes a major problem to maintain adequate building standards. Historical evidence in developing countries indicates that local design and construction often do not meet even the most basic of good building standards, both in quality of materials and in the placing and fixing of materials. In general this leads to a high and costly maintenance requirement. In the poorer countries where standards are lowest, planned maintenance and maintenance budgets for grain stores are an extremely rare occurrence. With inadequate maintenance, stores may deteriorate and still continue to be satisfactory for conventional storage but under this situation the ability to maintain adequate sealing for fumigation is extremely problematical and often not recognised by management. Under such circumstances, the danger of inadequate sealing become very real. Banks (1980) suggested that the success of sealing depends on the skill of the man doing it and his awareness of how the sealing system must be applied, rather than the particular method and materials used.

Some of the difficulties are due to the availability of supplies. Building material, including cement and reinforcing steel, are generally recognised as an economic necessity and, although sometimes difficult to obtain, are generally available. The requirement for particular sealing material may require a special import licence. Under present conditions where foreign exchange is desperately short in many developing countries, this is a very major problem leading to delays which make the local builder obliged to use what ever materials are locally available. In such a case in Senegal, bitumen became a universal sealant being applied liberally to cracks and joints. The eaves joint between masonary wall and corrugated sheeted roof was filled with a rigid packing of concrete or plaster and the ensuing cracks filled also with bitumen. Walls were painted with bitumen mixed with gypsum powder to eliminate the stickiness and finally painted with emulsion paint to relieve the drab colour. The treatment in this particular store is considered to be adequate although as yet untested. It was arrived at after simple laboratory tests but unfortunately this example is not common and frequently unsuitable decisions are taken without adequate advice.

Webley and Harris (1979) found in Mali that the traditional "banco" stores built entirely of mud with a flat roof, also of mud support by palm trunks, could be satisfactorily fumigated providing that cracks on the inside and around doors and windows were effectively sealed with fresh mud. Purpose built stores for fumigation have been satisfactorily constructed. Cocoa stores at Ikeja in Nigeria were constructed in 1966 and were reported to be satisfactory in operation (Riley and Simmons, 1967). Semi-underground hermetic stores, the so-called Cyprus Bins, described by de Lima (1980) have been used satisfactorily for the long term storage of wheat and maize under hermetic and fumigated conditions. However, neither of these stores could be said to meet the requirement of a general purpose transit store that can be fumigated and they represent isolated cases.

It must therefore be concluded that caution is needed in laying down precise design criteria for fumigable stores where resources for construction, management, operation and maintenance are severely limited. What limited field experience there is suggests that whilst building standards may be different from those employed in industrialised countries, they may be satisfactory for local requirements and that local materials may provide adequate sealing even though these materials would not be accepted as sealants in industrialised countries. The design of fumigable stores where materials and an understanding of the technology is very limited, has been seriously neglected and requires urgent attention being paid to it. Even the minimum standards of gas retention and sealing are not widely known or applied. The increasing number of reports that local organisations of developing countries are carrying out "total store" fumigation in what are clearly unsuitable buildings is causing serious concern about what the possible consequences might be. The recent work by Taylor (1982) and by Tyler *et al* (1983) in Bangladesh showed that a high degree of resistance to fumigation by phosphine by a number of pest species was due solely to regular and frequent fumigations by phosphine in unsuitable stores, lends weight to this concern and emphasises the need for urgent action to provide adequate store design criteria. The current increasing costs of carrying out under sheet fumigation compared to the apparent simpler operational procedures of total store fumigation is leading to its wider use in buildings, with insufficient attention being given to their suitability.

CONCLUSIONS

The need for very rigorous phytosanitary standards for export grain in Australia has clearly led to design criteria for flat grain stores that are suitable for controlled atmosphere storage and fumigation, and also the perfection of techniques for sealing already built conventional warehouses. There are, however, very few developing countries with the material resources or standards of technology to emulate the Australian methods. In the general case it may also be impossible to maintain a standard of insect free grain and it is arguable that it is not economically feasible or necessary to attempt to do so. Furthermore the long established tradition of handling grain in bags in developing countries and the unlikelihood of this practice changing rapidly in the near future, means that ubiquitous godown or bag warehouse provides a basic and fundamental design for a grain store. A design for fumigable stores must therefore be based very largely on these godown designs and on the minimum efforts and standards needed to make existing godowns suitable for fumigation and controlled atmosphere storage. Whilst many of the materials and techniques currently in use in Australia and elsewhere are relatively simple in principle, it must be recognised this technology is generally not available in developing countries and the materials may be largely unavailable because of scarcity of foreign exchange. Design standards and sealing techniques need to be based as far as possible on local standards and locally available materials.

The increasingly wide adoption of total store fumigation in developing countries in quite unsuitable buildings, with incontrovertible evidence that this is leading to increased resistance to fumigation by common insect pests, adds urgency to the development of appropriate designs and techniques for sealing.

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SESSION 5.

STORAGE SEALING TECHNIQUES (2)

Papers by:

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C. Barry	–	Australia
B.E. Ripp (2)	–	Australia
H.J. Banks and P.C. Annis	–	Australia

THE USE IN AND RECIRCULATION OF CARBON DIOXIDE IN WELDED STEEL BINS IN VICTORIA

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ABSTRACT

Different techniques are used to attain and maintain a high carbon dioxide atmosphere in stored grain in well sealed grain structures. This paper presents details of sealing, pressure testing, gas application and maintenance for typical Victorian welded steel bins. Costs for bin conversion and carbon dioxide applications are summarized. Lack of attention to any detail can reduce the CO_2 levels to a point below the level lethal for insects. Details are also given on a purge/recirculation pipe and on a simply manufactured pressure safety valve.

INTRODUCTION

The insecticidal properties of carbon dioxide (CO_2) in a controlled atmosphere storage situation have been realized in Australia for over sixty years. Since 1975, over 250,000 t of wheat have been treated with carbon dioxide. Currently, over 2.5 million t of storage space in the Australian bulk handling system requires little effort to seal to a standard which permits economical treatment of grain with CO_2. This paper stresses the need for extra care in all aspects of sealing and recirculatory layout in storage similar to typical Victorian Ascom type welded steel bins.

TYPE OF STORAGE

The bins used in Victoria for commercial usage of CO_2 are silver in colour, and are being progressively painted white as recommended by Banks and Annis, 1980. These bins vary in capacity between 1,500 t and 2,000 t and the measurements of a typical 1,900 t bin are height to eaves: 14.8 m; height of cone: 3.4 m; diameter: 13.9 m; and volume: 2,418 m^3.

The storages are flat bottomed with a central opening at the roof apex and a central outlet at the base. All bin openings and areas around the bin perimeter have been sealed.

SEALING

Wall to floor joint:

Because of slight movement of both steel and concrete, the base perimeter area of these bins is prone to gas leaks. This internal perimeter area is raked and cleaned, as are any floor cracks. Filling is done with a bitumen emulsion sealer. (Mightyplate plastic cement, Provence & Co., North Brighton, Vic.) This emulsion, a patented phosphorus stabilised material, was chosen because of its stability in weather extremes and its ability to fulfil requirements set out by the Co-ordinating Committee on Silo Sealants (Banks & Annis, 1980). Grain may stick to this compound so a 12 hour hardening period is desirable before treating the area with a polymetic vinyl plastic protective coating (Envelon, Dominion Plastics Industries, Shepparton, Vic.). Two coats of this flexible membrane are used and they provide a semi-hard cover upon which grains cannot attach. One coat is not sufficient to prevent the grain from sticking.

Doors:

The perimeter area of the door and cell opening are lined with 40mm width by 8mm thickness silicone rubber. The two areas are tightly joined by fastening the door with bolt pressure. However, for complete sealing, it is necessary to spray the outside perimeter with Envelon or cover with a mastic type seal.

Walls:

Thorough examination of all welds must be made. Signs of seepage (green discoloration) can be readily detected. 'Slag' holes and porous sections can be sealed successfully with 2-3 coatings of Envelon. Special care must be taken when spraying over these protruding slag sections or even bolts since gravity can cause a weaker upper area on the protrusion or bolts, and this small area of plastic coating can become perforated.

Roof hatch:

A sealed hinged disc hatch has been designed with rubber to rubber seals held under pressure to make this area gas tight (Fig. 1). This figure shows that a disc-shaped door folds diagonally for placement below the opening, and returns back to the original disc shape for holding fast against the base of the circular grain entry. The disc door is held secure by a bolt action across a 'T' piece at the bin apex. For a perfect seal, it is necessary to cover the hinged and perimeter areas with a mastic type seal.

PRESSURE RELIEF VALVE DESIGN

Pressure variations in a sealed storage may fluctuate because of wind velocity and diurnal temperature changes or during grain outloading or gas purging operations (Banks and Annis, 1980). A simple pressure relief valve is shown in Fig. 2. The 200mm diameter recirculation and vent pipe leads from the bin apex area downward and over the 75mm pipe which is fitted into the tank structure of the pressure relief valve. Water is added to the tank for sealing to cover the end of the 200mm pipe by exactly 75mm. A thin oil layer on the water surface can prevent evaporation. This device does not allow the internal bin pressure to go over 750Pa (75mm water guage). If the pressure in the bin exceeds this limit, the seal is broken allowing the bin to come to equilibrium with the atmosphere.

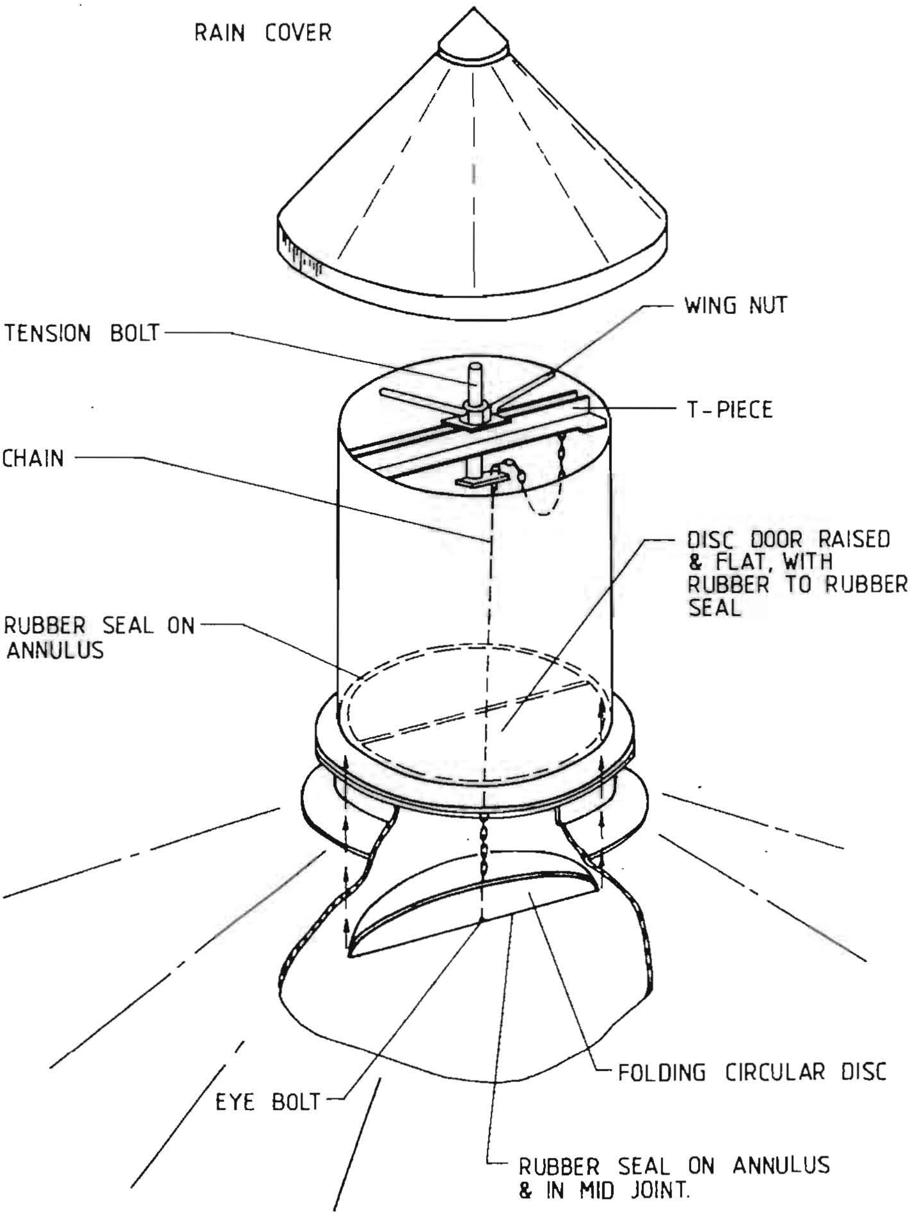

FIG. 1

DESIGN OF A TYPICAL BIN ROOF HATCH SEAL

GAS INTRODUCTION

Carbon dioxide is supplied in liquid form from road tankers of aproximately 20 t capacity. The tankers normal head pressure of 2000 kPa is used to force the liquid CO_2 through a vapourizer which converts it to gas at a temperature from 25 to 35^{o} C. This introduction temperature prevents condensation and subsequent moisture migration in the grain bulk.

Tankers and vapourizers are now designed to inject CO_2 at 3.5 t/h. This flow rate would create too great a pressure build up for a single bin so several bins are normally purged simultaneously (V. Guiffre, 1979, pers. comm.). The gas distribution piping is designed for minimum pressure drop so that uniform bin purging is obtained no matter which group of bins are being purged simultaneously. At a total CO_2 flow rate of 3.5 t/h the pressure difference in the CO_2 injection tubes is only a few kPa.

Victorian cells are purged in pairs using an 80% CO_2 - 20% air mixture. Gas introduced into the base of the bin is forced upwards and out through the 200mm recirculation pipe until measurement of the outflow stream at the base of the pipe indicates a level of 80% CO_2 (Fig. 3). The normal dose rate is approximately 1 t/CO_2 per 1000t of grain. Bin sealing is then completed by filling the pressure relief valve with water at the base of the overflow pipe, and closing the slide valve on the gas inlet pipe. Measurements of the CO_2 concentration are taken during purging and throughout balance of the treatment at the test point in the 200mm recirculation pipe (Fig. 2) using a Drager tube and pump.

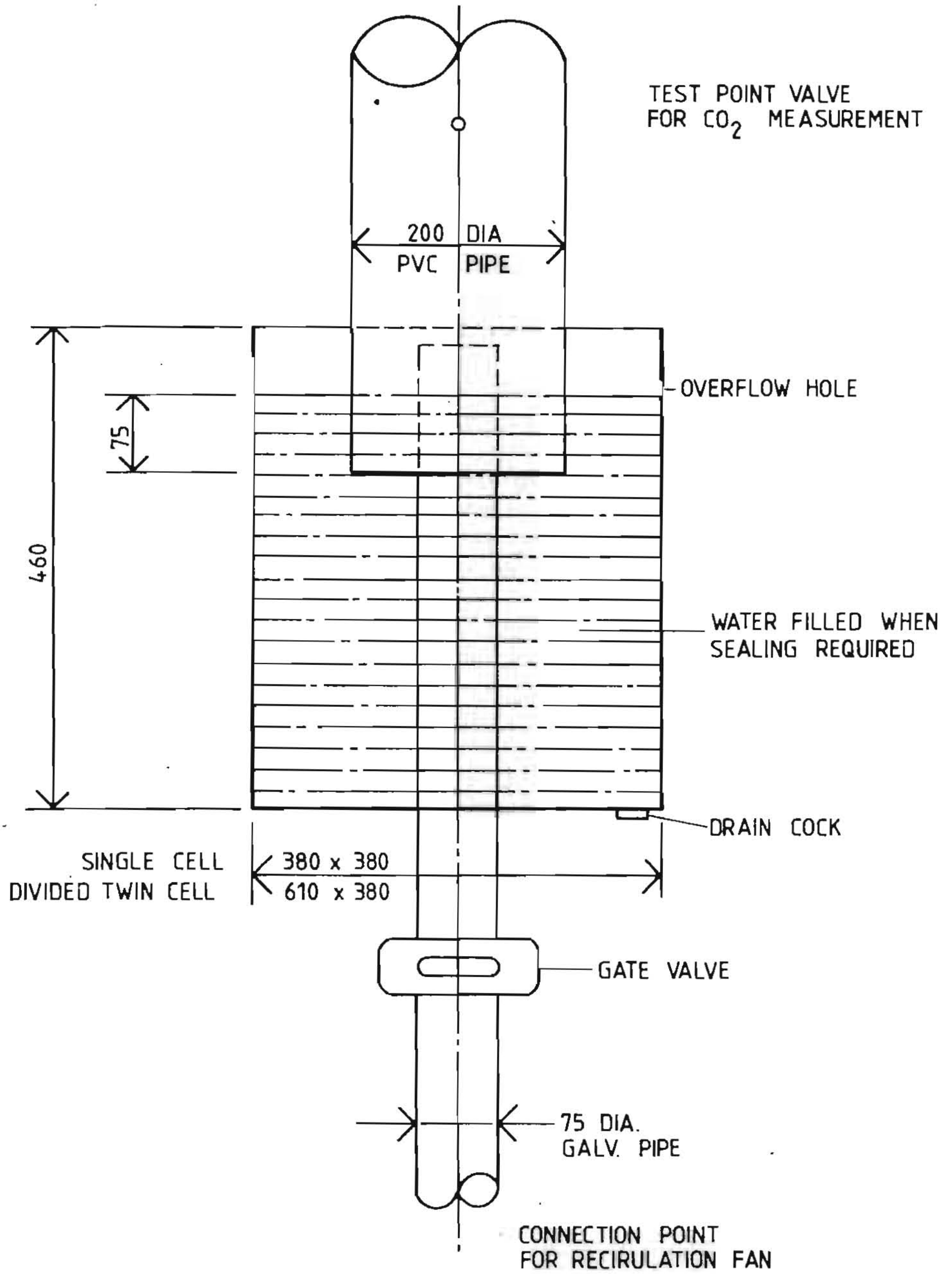

FIG. 2
CORRESPONDING PRESSURES FOR VARYING OUTLET PIPE DIAMETERS.

Only one inlet point is necessary for CO_2 introduction because of the nature of the gas (Banks 1978). The inlet and piping from the CO_2 vapourizer are both 75mm dia. The purge exit/recirculation pipework consists of a 200mm PVC pipe extending from near the bin apex down to the pressure relief valve, which is located approximately 1.5m from ground level. When the gas introduction technique is performed as described, a 200mm dia. outlet limits the pressure build-up in the bin to approximately 0.17 kPa. Outlets smaller than this cause excessive pressures in the bins, unless the gas is applied at a slower rate. Slower rates of application are more costly since the road tanker and driver are on site for longer periods. Smaller purge main diameters and corresponding bin pressures are shown in Table 1. (A. Segal, 1978, per. comm.).

RECIRCULATION OF CARBON DIOXIDE

Since CO_2 is 1-½ times as heavy as air it is necessary to recirculate the atmosphere gently within the storage to ensure that an even gas concentration is maintained for the duration of treatment (Wilson et al, 1980; Banks and Annis, 1980). This is achieved by installing a duct from the top to base of the bin, and utilizing a small fan for recirculation (Fig. 3).

Table 1.
Corresponding Pressures for Varying Outlet Pipe Diameters

Internal Diameter of Purge Main (mm)	Pressure in Bin (kPa)
100	3.45
150	.58
200	.17

The purge exhaust and recirculation pipe must be placed as near to the apex of the bin as possible. Experience has shown that the grain peak must clear the pipe opening by approximately 1m so that the high point of the grain does not prevent an even distribution of the recirculated CO_2 in the conical area. The elbow at the roof-to-wall joint consists of .5m flexible PVC hose (200mm dia.). A rigid joint can leak because of structural movement and climatic variation.

A fan (0.4KW, 75mm bore suction) is attached via a 75mm flexible hose to the base of the pressure relief valve and to the inlet port to complete the recirculation system. The fan achieves the rate of at least 0.1 atmosphere change per day (i.e. 10% of storage), which is stated to be adequate by Banks (1978). Experience has shown that under normal working pressure, hair-line fractures can form on the enamel along the seal-line at the side of the fan flanges which allows air entry during operation. These areas must be unbolted, retightened and sealed with covering layers of a mastic compound. The fan must have a tight fitting gland bearing with a grease cup around the shaft to prevent air ingress at this point.

PRESSURE TESTING

Prior to purging with CO_2, a standard pressure test as described by Banks and annis, (1977) is carried out on the bin. These standards state that it should take more than 5 minutes for pressure generated by a blower in a 90–95% full structure to decay from 500 to 250 Pa for storages of 300–10,000 tonnes capacity. Recent Victorian results using this pressure test resulted in decay times from 13 to 25 min which is substantially in excess of the required gastightness specification. The high levels of CO_2 maintained after two weeks recirculation, can be correlated with the standard of sealing as shown in Fig. 4 where longer decay times resulted in higher CO_2 concentrations at the end of this treatment period.

FURTHER CONSIDERATIONS

Moisture Migration:

In some studies with welded steel bins moisture migration to the hot upper areas has taken place, particularly if the grain received is at the Australian limit of 12% m.c. Areas of excess moisture are formed because the grain cannot reverse the moisture movement during the cooler night hours. Mould growth and bin scalding have been experienced under these circumstances.

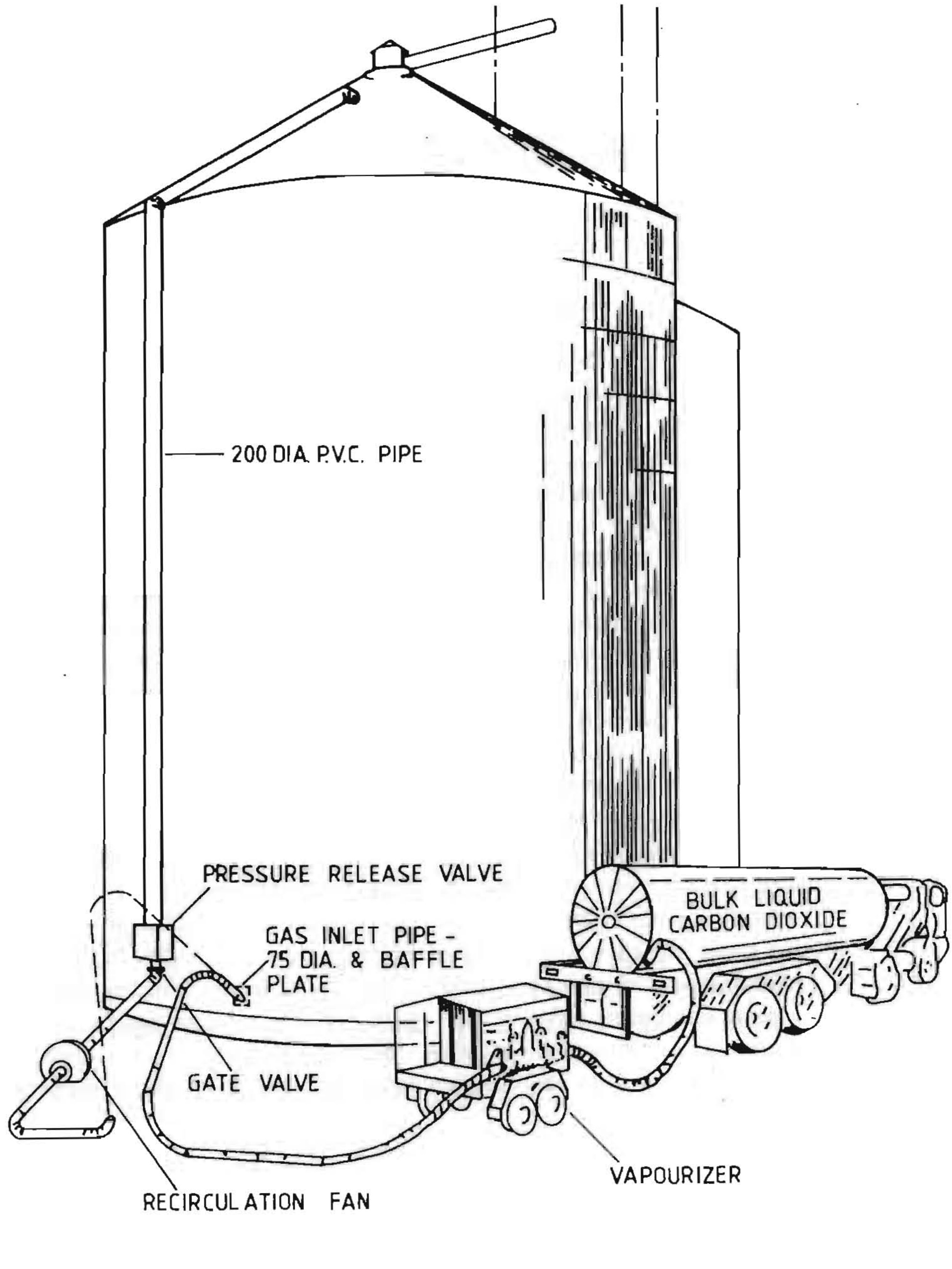

FIG. 3

A VICTORIAN TYPE STEEL BIN EQUIPED FOR USE OF CO_2 WITH TANKER AND VAPOURIZER ONSITE. ALL DIMENSIONS ARE IN mm.

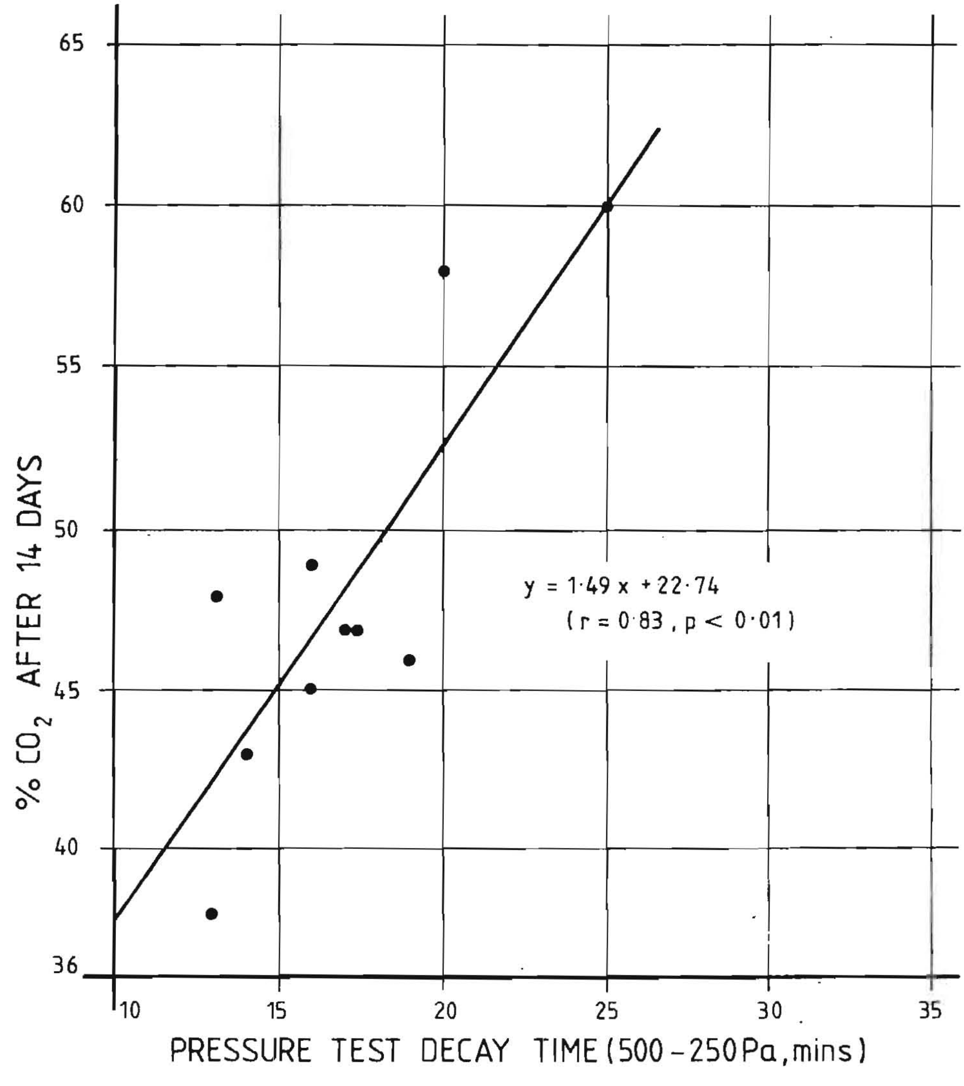

FIG. 4

PRESSURE DECAY AND CO_2 DECLINE 14 DAYS AFTER PURGING. STUDIES WERE CONDUCTED AT YARRAWONGA AND RENNIE, VICTORIA IN 1981 AND 1982.

To overcome this problem, care must be taken to ensure that overfilling does not take place and that the grain surface is 1m below the roof apex. Grain should be turned from base to top prior to CO_2 purging to ensure redistribution of any hot spots, and to remove grain compaction pressures from the baffle plate area of the CO_2 entry pipe. During the turning process the centre core of grain goes through a lot of movement and the grain at the base and sides of the bin moves marginally. This process results in less compacted grain and allows for more successful penetration.

Roof Painting:

A white acrylic matt reflectant finish is desirable to help reduce moisture migration and gas loss due to diurnal temperature changes in the headspace (Banks and Annis, 1979).

Outloading and Reinfestation:

Reinfestation can occur quickly if there are delays in the outloading processes of CO_2 treated grain. If delays are inevitable openings should be covered with insect proof mesh screen and the surrounding area sprayed with a residual insecticide. A suitable insect proof mesh measures $0.6mm^2$. A residual insecticide, azamethiphos, has proven successful in field experience. This spray has remained effective at 0.5% on metal surfaces, and at 1.0% on porous surfaces for more than 26 weeks (B. E. Wallbank, R. J. Hart, 1979, pers. comm.).

Back Pressure:

A Baffle plate, 600m x 600mm installed at the end of the inlet pipe will ensure that at a CO_2 injection rate of 1 to 1.5 t/h, the back pressure will be less than 10 kPa. Back pressure will vary only between 30/40 kPa. This plate is similar in design to those described by Banks and Annis (1977). Back pressure does vary between wide limits, depending on gas flow rate, grain condition, voidage and dust content.

Costing:

Total sealing, plumbing, labour and fan installation costs for the welded steel bins discussed here were carried out after works completion in 1978. The total cost per bin was Aus. $1,289.00 or 64¢/t grain capacity. This is equivalent to an amortized capital cost of 5.6¢/t calculated from a 6% real discount rate, an assumed average fill rate of 100% per annum, and an estimated sealing life of 20 years, with negligible maintenance cost per annum. Connell and Johnston (1981) by contrast, estimated capital costs of sealing similar bins to be 87¢/t, (or 13¢/t as an annual amortized figure calculated from a 6% real discount rate, and assumed average fill rate of 90% per annum, an estimated sealing life of 10 years and a life of engineering modifications of 15 years) and 4¢/t annual maintenance, giving a total annual equipment cost of 17¢/t. From 1978 the total carbon dioxide input costs have been approximately 25¢/t of grain treated as estimated by Connell and Johnston (1981). This gives a total annual cost of 30.6¢/t as compared with the estimate given by Connell and Johnston of 42¢/t. These figures show that total costs for carbon dioxide treatments are less expensive than the cost of the current chemicals in use, fenitrothion and synergized bioresmethrin, at application. These chemical costs have averaged 53¢/t of grain treated during 1978-83.

Safety:

Conveyor tunnelling beneath bins can be a danger area because small leaks from one or more bins could lead to a toxic accummulation of CO_2 in this area. Gas sampling pipes should be installed and access to the tunnel locked so that before unlocking after treatment gas samples can be drawn from the lowest area of the tunnelling. Grain must be thoroughly aired before outloading, with fans operating in confined areas where grain is moving. After purging the work areas of excess CO_2, the content of the air at any point must not exceed 1% CO_2. This is based on a time weighted average for up to a 10 hour shift in a 40 hour week with a maximum exposure of 3% CO_2 (V. Guiffre, 1978, pers. comm.).

CONCLUSIONS

The commercial work and details given are the cumulative effort of seven years work, and can be considered as a meaningful part of the general development of various modified atmosphere techniques of grain storage and disinfestation for Australian conditions. Attention to every structural detail and precise control of the use of CO_2 are all important in ensuring an economic outturn of the grain, especially when it is received at or near 12% m.c.

With the strategies described, and with continued attention to detail, CO_2 treatment of grain can be regarded as a viable and economic ally to the current chemical methods of grain protection. Further studies may indicate that it would be profitable to have stationery on-site CO_2 vessels to be refilled as needed at sites where large quantities are used. Certainly methods of insect detection and techniques for maintaining the grain insect free to its final destination need to be expanded.

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WESTERN AUSTRALIAN STUDIES OF SEALING HORIZONTAL STORAGES IN 1980-1982

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ABSTRACT

Sealing studies on three 25,000 tonne (t) horizontal storages were conducted in Western Australia in 1980. These studies included the evaluation of three different sealing materials, pressure testing of empty and loaded storages, and tests on the feasibility of using phosphine or carbon dioxide in these storages for insect control. Additional research was conducted in dust extraction during inloading and outloading and on the effectiveness of heat reflective coatings to prevent pressure buildup.

These studies were considered successful, so 20 additional storages ranging in size from 19,100 to 34,000 t were sealed in 1982. Many of these storages exhibited a higher degree of sealing than those sealed in 1980 and this programme was considered a success.

INTRODUCTION

Interest in the use of controlled atmospheres for insect control in stored grain in Australia prompted field trials on the use of nitrogen in several types of grain storages in the early 1970's (Banks and Annis, 1977). In the early portion of these studies it became obvious that rigorous sealing of these structures was necessary to prevent excessive gas loss. In the late 1970's and early 1980's studies on the use of controlled atmospheres shifted from the use of nitrogen to the use of carbon dioxide and research continued on sealing and pressure testing to determine the degree of gastightness of these storage structures (Banks and Annis, 1980). During this period it was also discovered that conventional fumigants could be used more efficiently and economically in sealed storages.

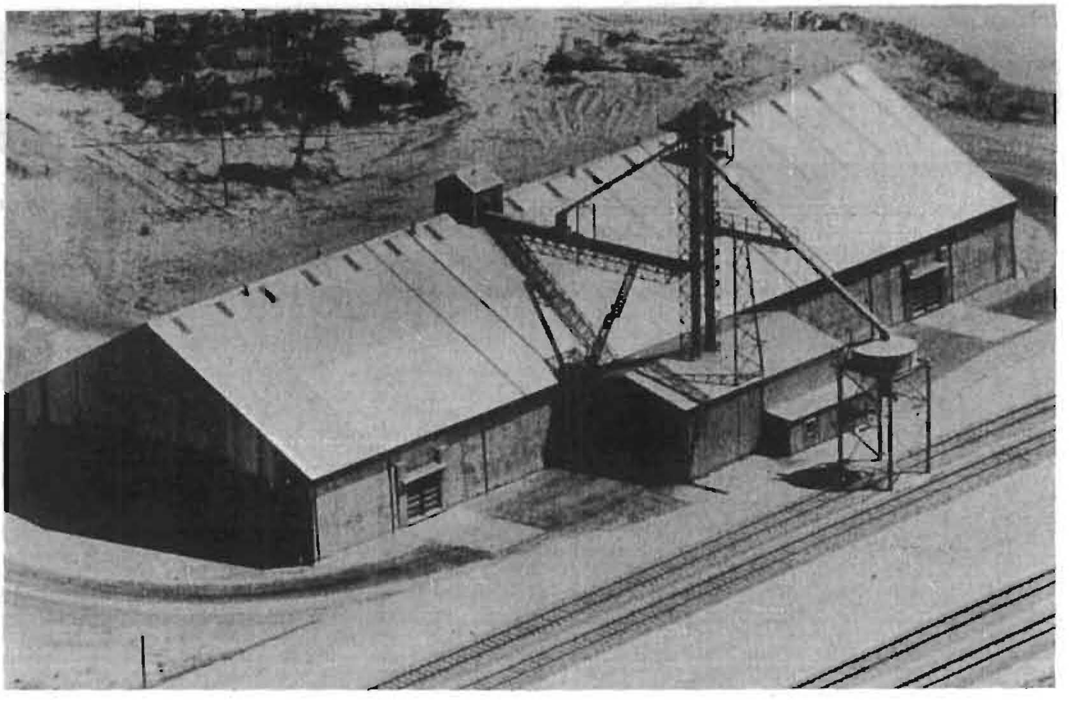
Co-operative Bulk Handling Limited, Perth, Western Australia, became interested in the sealing programme, particularly in horizontal storages. This paper describes some of the sealing techniques, pressure testings and other studies conducted in three horizontal storages. These studies eventually lead to the sealing of 20 additional storages having a total capacity of 458,400 tonnes by 1982.

METHOD AND MATERIALS

1980 Trials - Site Selection and Sealing Materials

In 1980 Co-operative Bulk Handling Ltd of Western Australia embarked on a trial programme to seal three horizontal grain storages to determine the suitability of this technique for improved insect control and quality preservation of grain. Three sites selected were located at Southern Cross, Burracoppin and Cunderdin. A 25,000 tonne (t) storage at each of these sites was sealed using Wastolan (a chloroprene latex based emulsion), Envelon (P.V.C.) and Polyurethane foam.

These horizontal storages measured 133m long by 30m wide with concrete walls 5m high. The side walls are extended a further 1.4m by non load-bearing curtain walls of corrugated steel sheeting and each end wall is topped by a gable-end reaching to the apex of the roof. The roof of overlapped corrugated iron sheeting is supported by steel trusses bolted into the concrete walls and pinned at the apex. The height from floor to roof apex is 14.7m and from floor to the peak of the grain when filled was ca. 11.3m. Each storage has three ground level double access doors each measuring ca. $3m^2$.



"A" type horizontal silo. The gable end and curtain walls above the concrete walls are of corrugated galvanised iron. Three main access doors are provided.

The total volume of each of these sites is $45,300m^3$ and the grain mass takes up ca. $28,300m^3$. The intergranular space of the grain is estimated to be $11,300m^3$ and the head space above the grain is ca. $16,990m^3$. Therefore, the total free air space in these storages is ca. 28,300m^3 when filled with grain.

These storages are located in an area of Western Australia which has a mean Summer temperature of from ca. 34 to 17°C (day-night) and a mean Winter temperature of from ca. 17 to 14°C. Mean rainfall at these three sites range from 281 to 396mm a year.

1980 Trials - Gas Recirculation System

A recirculation fan was placed on each end of the storages adjacent to an access platform. These fans were located 6m above ground level and were connected to a central duct of 150mm P.V.C. piping running along the floor the length of the storage. This system was designed to pull carbon dioxide (CO_2) out of the grain and redeliver it into the head space when the grain is being treated with the gas. These fans were also used to vent the gas out of the storage after treatment with CO_2 or fumigation with Phosphine gas (PH_3). The venting system was added by fitting another pipe on the discharge side of the fan. When ventilating the storage the discharge side of the return head pipe is blanked off and the fans operated in reverse.

The fans which are described in 1980 trials - Pressure decay tests and dust sampling studies - were also used in the venting of fumigants.

1980 Trials - Pressure Decay Tests

Pressure tests were performed before harvest on empty storages and after harvest on filled storages to determine the degree of gastightness of the facilities. Presure tests prior to harvest were performed so that any leaks present could be repaired from the inside prior to loading. After the bins were loaded a final pressure test was performed. The test criteria established was based on pressure created by running the fans until 180 to 200 Pa pressure was obtained in the storage. At this point the fans were shut down and blanked off. If this pressure decayed by over one-half in 15 min. or, for example, from 180 to less than 90Pa the storage was not considered adequately sealed and further sealing was accomplished. These fans were rated at 85m^3/min and were 482mm in diameter.

Pressure decay tests were generally conducted in the evening as the pressure decay is directly related to the volume of grain in the storage, the climatic conditions at the time of the test and the number of leaks within the storage.

1980 Trials - Dust Sampling Studies

The fans located in the end of the storages were used to vent breathable dust, or dust below 10 microns in size during outloading. Since the fans were reversible the operators could, depending on wind direction, direct the air flow into the storage to create a through draught or use them to extract the dust.

Additional studies on dust extraction were conducted in 1981 and 1982 to determine if an additional fan located in the centre of the storage was necessary to adequately remove the respirable dust. Results showed a centre fan was necessary and further trials are being conducted into the size and rates.

1980 Trials - Phosphine Fumigation Studies

In 1980 two PH_3 fumigation trials were conducted at Southern Cross and Burracoppin and one at Cunderdin. One of the trials at Southern Cross is not considered here since the instrument used for analysis of the concentration was found to be faulty. In each test 5 gas sampling lines were placed in the storage, four down in the grain mass and the fifth in the headspace above the grain.

Table 1 presents data on the amount of wheat treated in the 5 tests, the amount of aluminium phosphide tablets used in the equivalent dosage of PH_3 evolved in these tests.

Table 1. Treatment of wheat with PH_3 in sealed horizontal storages in Western Australia, 1980.

STORAGE SITE	WHEAT TREATED (t)	AlP. USED Kg	PELLETS /t	$g/PH_3/m^3$
SOUTHERN CROSS	25,351	25	1.6	0.25
BURRACOPPIN I	26,560	21	1.3	0.20
BURRACOPPIN II	26,560	25	1.56	0.24
CUNDERDIN	22,464	25	1.8	0.28

The length of treatment ranged from 10 to 20 days in these studies.

1980 Trial - Carbon Dioxide Treatment Study

One test was conducted on the use of CO_2 in a sealed storage in Cunderdin. In this test 25,000 t of wheat was treated with 48 t of CO_2. The purge time was 27 hrs and the CO_2 was maintained, with the recirculation system operating, for 24 days. The Australian recommendation for control of stored product insects is the maintenance of a CO_2 concentration of more than

35% for a period of 10 to 14 days. Sampling points in this test were in the same locations as those in the PH_3 tests.

1980 Trials - Heat Reflective Coatings

A two week study was conducted to evaluate the effectiveness of a white roof at Southern Cross with a conventional galvanised roof at Bodallin in reducing the temperature inside the horizontal storage. These tests were performed in December when a very high ambient temperature is encountered in this region (see Table 3).

1982 Trials - Sealing and Pressure Testing

The success of the 1980-81 trials on the three horizontal storages prompted C.B.H. to proceed with the sealing of 20 additional horizontal storages in 1982. These storages range from 19,100 to 34,000 t capacity and are located in different grain growing areas of Western Australia. Of the 20, six were sealed with chloroprene latex two were sealed with modified acrylic-vinyl chloride, one with P.V.C. and the remaining eleven with an elastomeric acrylic coplymer. Pressure decay tests were conducted on all 20 of these storages prior to loading and additional sealing was performed on those that did not meet the half-life decay value established in the 1980 studies.

Information developed in many pressure decay studies conducted in 1980-81 showed that the best time to conduct these tests was between 2 and 3 a.m. So the 1982 tests were carried out during these early morning hours to take advantage of the stable conditions.

RESULTS

1980 Trials - Site Selection and Sealing Materials

The three storages selected, although of similar construction, were situated at three different locations. Three different sealing materials were used which provided useful information for the 1982 sealing of 20 additional horizontal storages.

1980 Trials - Gas Recirculation System

The gas recirculation system functioned adequately in maintaining the CO_2 concentration in the grain mass in the study at Cunderdin. The fans also worked well in venting PH_3 from the storages at the completion of the four tests.

1980 Trials - Pressure Decay Tests

The inital pressure decay tests provided insight into pressure changes in sealed bins during diurnal temperature changes. Figure 1 shows pressure decay rates at the three sites in tests conducted in March and April while the bins were cooling. This figure illustrates the fact that the storages were not sealed to the criteria established for a well sealed bin in that the pressure decayed by more than one-half in less than 15 min. This figure also shows that the storage at Burracoppin was more tightly sealed than were the other two storages.

Figure 2 shows pressure decay times for these storages while they were heating in the morning. Although the decay rate decreased approximatley 30%, only the Burracoppin storage approached the criteria set for a tightly sealed storage. The time was ca. 12 min. for a decay rate of from 200 to 100Pa.

1980-82 Trials - Dust Sampling Studies

During the outloading of the 1981 harvest, dust samples were collected in air space of the three sealed storages. The fans on the ends of the storages were operated according to the prevailing winds and grain dust concentrations were generally found in samples taken from the centre of the storages so further tests were conducted to determine if an additional fan was needed in this area.

These tests were run during filling of the sealed storages and a comparison was drawn between air-borne dust samples taken from these storages and samples taken from two similar unsealed storages. Grain dust concentrations from the centre of the sealed storages ranged from 100 to 226 mg/m^3 while samples from the ends of these storages ranged from 27 to 150 mg/m^3. Samples from the centre of the two unsealed storages ranged up to 593 mg/m^3 while samples from the ends of these storages ranged from nil to 82 mg/m^3. These results confirmed that a centre fan was needed in the sealed storages and they were installed at Southern Cross, Burracoppin and Cunderdin.

In studies during filling in 1982 at Southern Cross and Burracoppin the quantity of dust in suspension in the centre of the Southern Cross site was 500 mg/m^3 with the centre fan off and 300 mg/m^3 with the centre fan on. A similar reduction in grain dust was observed at Burracoppin where the concentration was 156 mg/m^3 with the centre fan off and 60 mg/m^3 with this fan in operation.

Based on this information all sealed sites were equiped with 3 fans and the size of each increased to provide 180 m^3/min of total air flow.

1980 Trials - Phosphine Fumigation Studies

The PH_3 fumigations described in Table 1 were conducted for periods of from 10 to 20 days. Table 2 presents data on the PH_3 concentrations attained and maintained during these tests.

Table 2 PH_3 concentrations in p.p.m. attained and maintained during fumigation of sealed horizontal storages containing ca. 25,000 tones of wheat

	RANGE OF PH_3 AFTER INDICATED DAYS (D) - P.P.M.			
LOCATION	1D	4D	7D	12D
SOUTHERN CROSS	50-70	55-70	53-70	50-55
BURRACOPPIN I (a)	0-100	32-108	80-150	68-84
BURRACOPPIN II	40-60	60-88	77-90	70-90 (b)
CUNDERDIN	50-100	80-98	63-78	60-80

a) Concentrations of 20-30 p.p.m. after 20 days.

b) 10 day readings

All four fumigations trials at the three locations showed that the necessary concentration by time values for successful insect control using PH_3 were maintained. The value of sealing can be seen in the second test at Burracoppin where 20 to 30 p.p.m. of PH_3 was detected in the grain 20 days after the application of the fumigant. Distribution of the fumigant was generally similar at all sampling sites in the grain mass. The concentrations in the headspace were generally higher.

<u>1980 Trial - Carbon Dioxide Treatment Study</u>

In this trial in the sealed storage at Cunderdin the CO_2 concentrations at all sampling points (except the headspace) was 88 to 100% two days after initial application. The concentration dropped to ca. 60% at all sampling points in the grain after four days and to ca. 45% at these points after 10 days. The CO_2 concentration was ca. 34% after 20 days at the in-grain sampling points and dropped to ca. 24 to 28% after 24 days. The concentration of CO_2 in the headspace fluctuated from 44 to 15% during the 24 day treatment. This was apparently due to diurnal temperature changes creating pressure in the headspace which caused the CO_2 to move about in this area.

This test again provided a graphic illustration of the value of sealing of these horizontal structures since the initial CO_2 applied was still in the grain mass at a lethal concentration (more than 35%) 21 days after the start of the test.

1980 Trials - Heat Reflective Coatings

The temperature differences found inside the storages between the white roof at Southern Cross and the conventional galvanised roof at Bodallin are shown in Table 3.

Table 3 Temperature differences (°C) found inside a sealed storage with a white roof (Southern Cross) and inside a storage with a galvanised roof (Bodallin). Temperature readings taken at 2 p.m. on 3 successive days in December, 1980.

DAY	SOUTHERN CROSS*				BODALLIN*			
	A	B	C	D	A	B	C	D
1	36	26	30	38	36	54	53	38
2	32	30	35	36	30	47	47	30
3	28	28	31	30	24	43	42	27

* A : outside storage in shade; B : inside storage under skylight; C : inside storage not under skylight; D : outside storage in sun.

This Table shows -

Temperatures were 23 to 28°C. lower on Day 1 inside the sealed storage in the headspace at Southern Cross than were those temperatures at Bodallin. Similar differences can be seen on Day 2 and Day 3 and point out dramatically the value of the reflective coating in preventing temperature build up and subsequent pressure build up in sealed storages.

1982 Trials - Sealing and Pressure Testing

Fifteen of the 20 storages were found to be sealed to a high degree of gastightness. The remaining five sites required additional work to bring them up to these standards. Figure 3 shows results of pressure tests on four of these storages. Each storage was sealed with a different material and this Figure shows that the pressure decayed only 27 to 36% in 15 min. or much less than the 50% decay criteria established for a well sealed storage.

CONCLUSIONS

The 1980 sealing trials proved that horizontal grain storages in Western Australia could be sealed to a relatively high degree of gas tightness. Studies provided valuable information which was used to seal 20 additional storages to a high standard. These storages can now be fumigated with very low concentrations of PH_3 to obtain a high level of insect control. Carbon dioxide can also be used efficiently and effectively in these storages to accomplish the same goal.

The tests also provided valuable information on grain dust extraction during loading and outloading and on the value of heat reflective coatings to prevent excessive pressure build up during diurnal temperature cycles.

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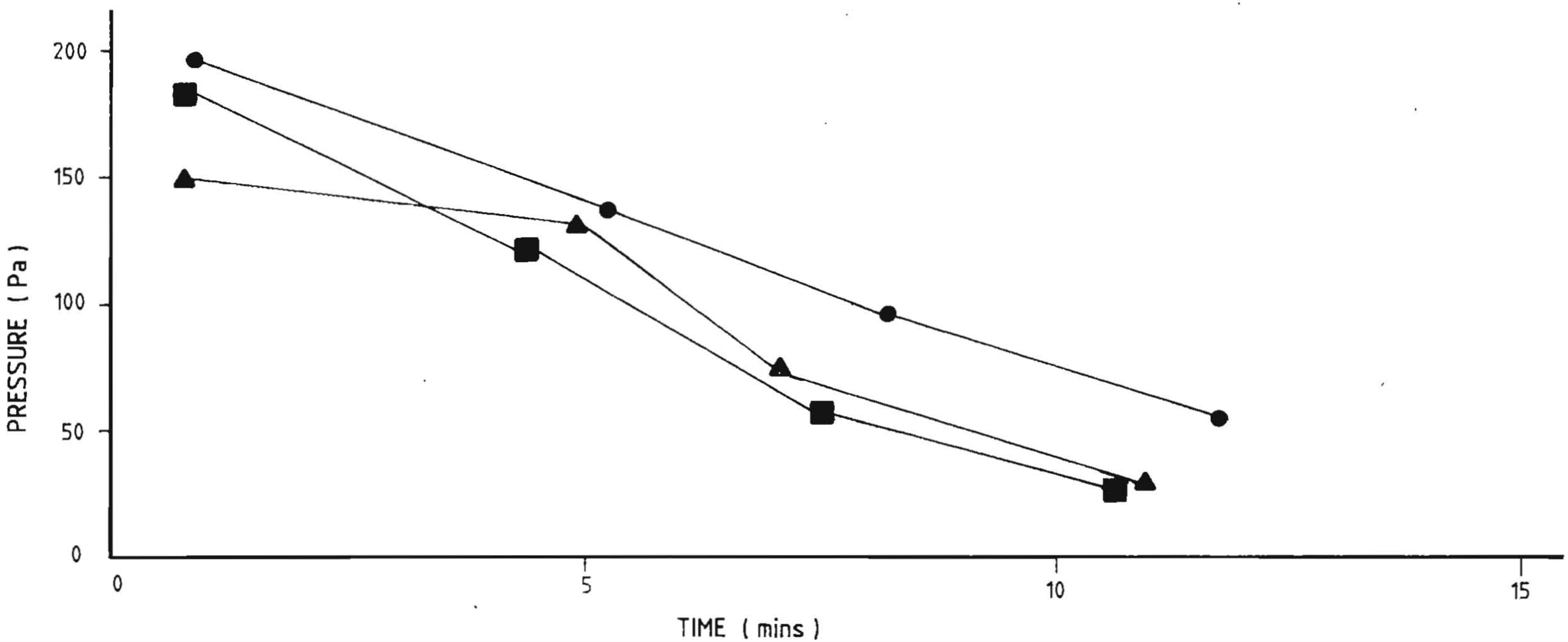

FIG. 1 PRESSURE DECAY TESTS IN 3 25,000 + HORIZONTAL GRAIN STORAGES AFTER HARVEST. TEST CONDUCTED IN MARCH AND APRIL AT EITHER 21.30 OR 22.5 h (STORAGE COOLING)
STORAGE LOCATION : ● BURRACOPPIN
▲ CUNDERDIN
■ SOUTHERN CROSS

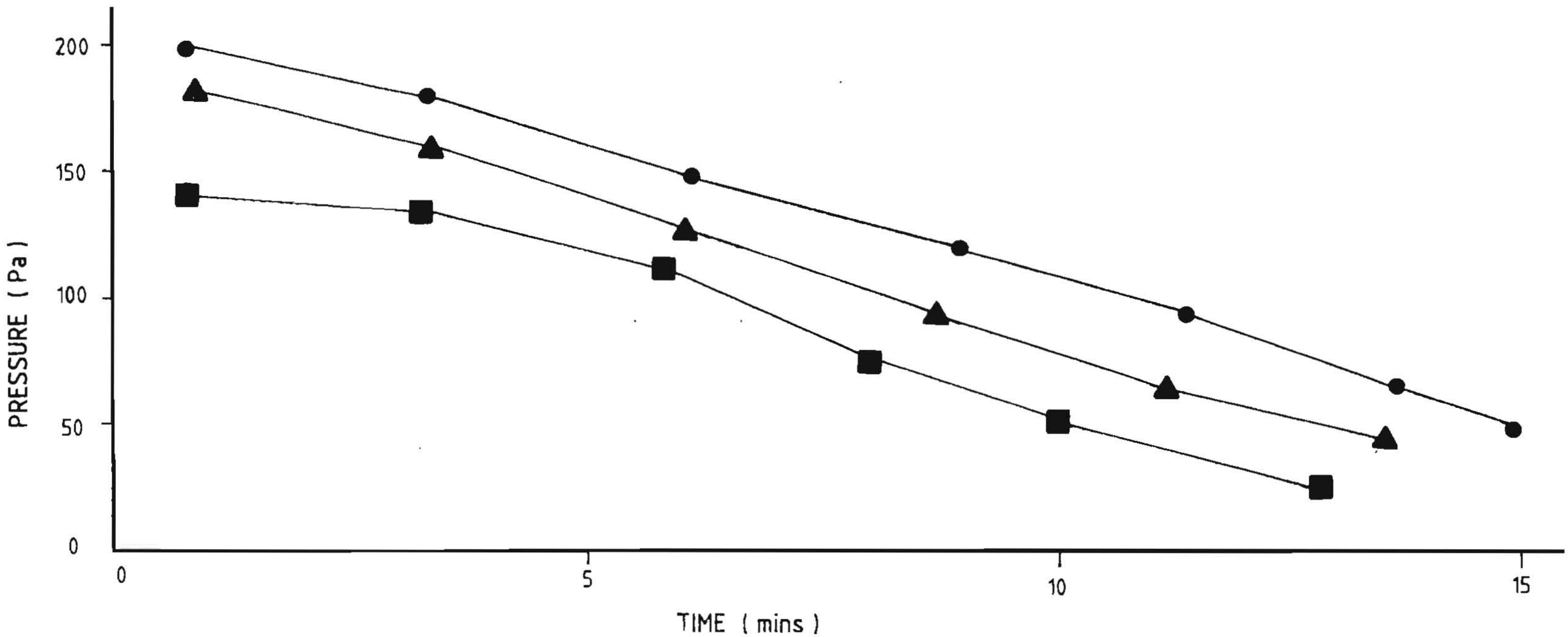

FIG. 2 PRESSURE DECAY TESTS IN 3 25,000 + HORIZONTAL GRAIN STORAGES AFTER HARVEST. TESTS CONDUCTED IN EITHER MARCH OR APRIL AT EITHER 1200 OR 1400 h (STORAGE HEATING).
STORAGE LOCATION : ● BURRACOPPIN
▲ CUNDERDIN
■ SOUTHERN CROSS

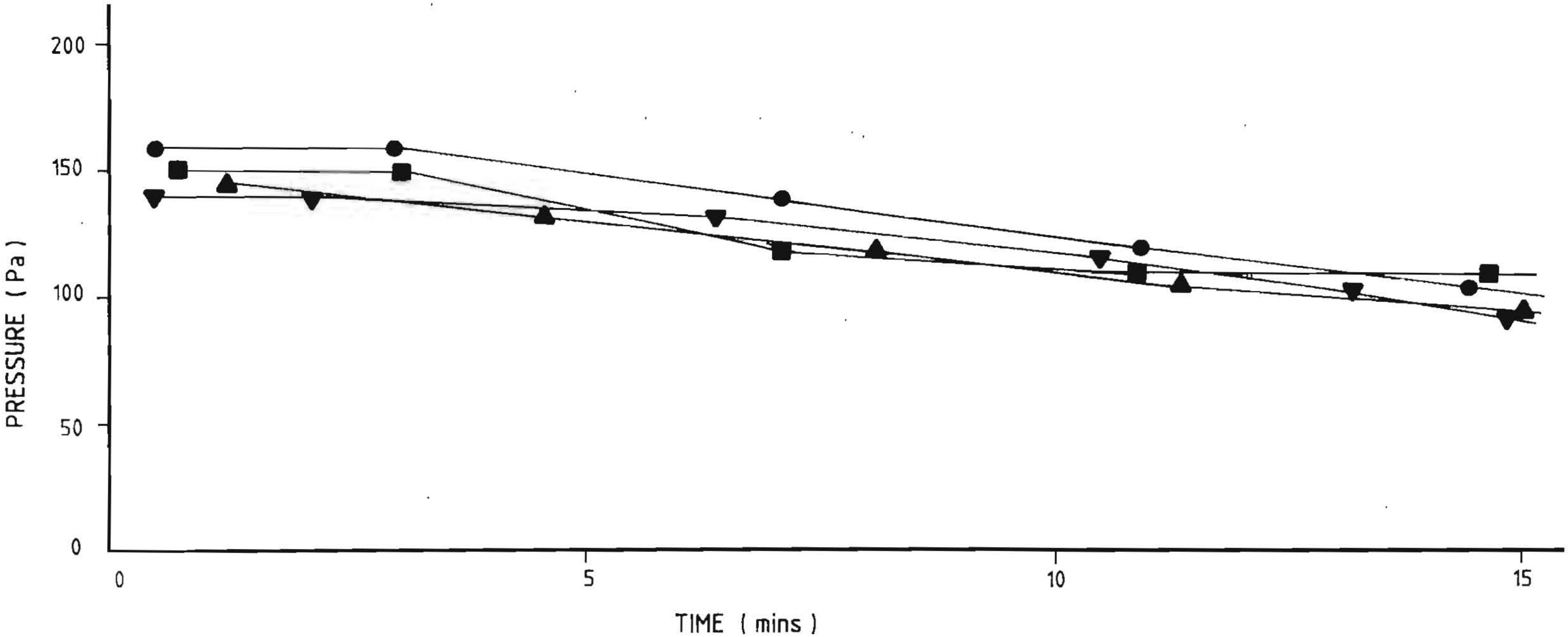

FIG. 3 PRESSURE DECAY TESTS IN 4 19,000 TO 27,000 + HORIZONTAL GRAIN STORAGES AFTER HARVEST. TESTS CONDUCTED IN SEPTEMBER TO NOVEMBER AT 0200 TO 0300 h (STORAGE STABLE).
STORAGE LOCATION AND SEALING MATERIAL :

- ● ONGERUP (P.V.C.)
- ■ KELLERBERRIN (CHLOROPHENE LATEX)
- ▲ MOORA (MODIFIED ACRYLIC - VINYL CHLORIDE)
- ▼ SHACKLETON (ELASTOMERIC ACRYLIC COPOLYMER)

MODIFICATION OF A VERY LARGE GRAIN STORE FOR CONTROLLED ATMOSPHERE USE

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ABSTRACT

Controlled atmosphere techniques of insect control in stored grain are receiving widespread interest as alternatives to chemical protectants. The very large grain storage shed, No. 1 Horizontal Storage (capacity 300,000t wheat) at Kwinana terminal, is currently being sealed and modified so that it can be treated with 40% Co_2 in air as a controlled atmosphere. The storage is close to an inexpensive source of the gas, which will be supplied by pipeline. the various operations required to fit the storage for this use are described. These include use of white acrylic and PVC - based materials to treat the roof and walls, giving a heat-reflective coating and seal, and the fitting of fans to recirculate the gas within the store, vent it after use and, when necessary, prevent build up of airborne dust and CO_2 in the store and work spaces. The cost of CO_2 use, as applied at Kwinana compares favourably with that of the other possible insect control measures available.

INTRODUCTION

In Australia and elsewhere, many of the pest control measures in current use for stored grain are rapidly becoming unacceptable. This may be either through development of insect resistance or because of costs or marketing preferences and requirements. There are several alternatives under consideration, mainly as replacements for the organophosphate insecticides that have hitherto provided such excellent service to the industry. Of these, controlled atmosphere techniques have so far received most attention.

The fact that an atmosphere of sufficiently low oxygen or high carbon dioxide content will control grain pests is not in question, although some of the finer detail of the action of such atmospheres is not known (see Bailey and Banks, 1980). Putting the concept into industrial practice is now the main problem. This paper summarises the work carried out by Co-operative Bulk Handling Limited at its Kwinana export terminal to modify a very large grain storage (dimension and capacities see Table 1) to accept controlled atmospheres generally and CO_2 - based atmospheres in particular. Delegates to this Symposium will be shown the current progress in the conversion and sealing of this shed, believed to be the largest project of this kind yet undertaken in the world.

METHODS

Site

Co-operative Bulk Handling Limited's Kwinana Terminal occupies a 21 hectare site, 40km south of Perth. It is one of the largest integrated grain terminals in the world, with a capacity of 912,300t wheat, held in a large cell block and two large horizontal shed-type storages. The No. 1 Horizontal Storage, a 300,000t capacity shed, was selected for sealing and modification to accept CO_2 as an insecticidal controlled atmosphere.

Use of CO_2

CO_2 was chosen as the basis for the controlled atmosphere systems at Kwinana for three reasons. Firstly, there was a ready cheap supply of the gas close to the site (3km), permitting delivery by pipeline. The gas was produced as a by-product of ammonia production at the Western Mining Nickel Refinery. Secondly, CO_2 will control insects at a much lower concentration than the alternative, nitrogen, and its action is much less sensitive to temperature. Provided the exposure time is long enough, CO_2 atmospheres need only contain greater than 35% CO_2, whereas nitrogen atmospheres must be greater than 98% N_2 (i.e. less than 2% O_2) and preferably greater than 99% N_2 (less than 1% O_2). The exposure periods required for complete control of insects with nitrogen - based atmospheres can be commercially excessive at low grain temperatures (Banks and Annis, 1977). We considered that with CO_2 - even at 40% CO_2, the atmosphere chosen for use at Kwinana - an exposure period of 3 weeks would be adequate no matter what the grain temperature was. Thirdly, we were unsure whether nitrogen could be used to purge such a structure efficiently. Indeed we saw problems in this regard, but we knew that the density of CO_2 gas would assist the efficient distribution of the gas and purging of the bulk from our own and other experience in smaller sheds (Co-operative Bulk Handling Limited, unpublished work; Banks et al, 1980).

Sealing of the Storage

The sealing of the No 1 Storage involved the creation of an almost gastight enclosure of 550,000 m^3 capacity. The contract for the sealing specified that the shed be sealed to a level such that, when full, an applied excess pressure of 200Pa in the shed would decay to 100Pa in not less than 30 minutes under stable temperature conditions. Sealing to this standard would ensure that gas loss from the store was minimised and thus as little CO_2 as possible was required to create and maintain the desired CO_2 concentration within the store.

Table 1 - Dimensions, construction and capacities of No 1 Horizontal Storage, Kwinana.

Dimensions	305m long, 76m wide, 31m high
Construction	Concrete walls and floor, truss roof with aluminium cladding
Wall area	6000m^2 concrete and 3670m^2 aluminium sheeting
Roof construction	Corrugated aluminium sheet, 30^o pitch
Roof area	28000m^2
Enclosed volume	550,000m^3
Rated capacity	300,000t wheat
Filling ratio	0.60
Calculated gas volume when full	310,000m^3

The general approach to the sealing of the storage was based on the technique employed at Harden, New South Wales (Banks et al, 1979) as modified by our experience in our sealing programme for large horizontal storages in Western Australia, of which 24 have so far been completed.

Prior to sealing, the structure was cleaned of contaminants and salt deposits using high pressure water jets. Two materials were used for most of the sealing work: Envelon 441 (Dominion Plastic Industries, Shepperton, Victoria) and Flexacryl (Taubmans Paints, Sydney). Envelon 441 is described as a solvent-based PVC solution that produces a self-supporting membrane. It was applied to lap joins in the aluminium cladding at 800 to 1800 micron thickness without reinforcement. Flexacryl is a thick, modified acrylic, water-based emulsion. It was applied at 150 micron thickness over the roof and externally over the concrete walls at 100 microns to provide a white, heat-reflectant finish and give protection to the Envelon against UV-degradation. The roof temperatures were noticably reduced on painting (Fig 1). The concrete walls were also coated internally at 500 microns thickness with Flexacryl, with a PVC - membrane applied first over any cracks or joins in the wall. The floor of the store was treated with a silicone based sealer (Raffles Floor Sealer) which penetrated into the concrete. The few large cracks in the floor were filled with an acrylic filler. Large holes, such as where the aluminium cladding met the top of the concrete wall, were filled with rigid polyurethane foam before the acrylic top coat was applied.

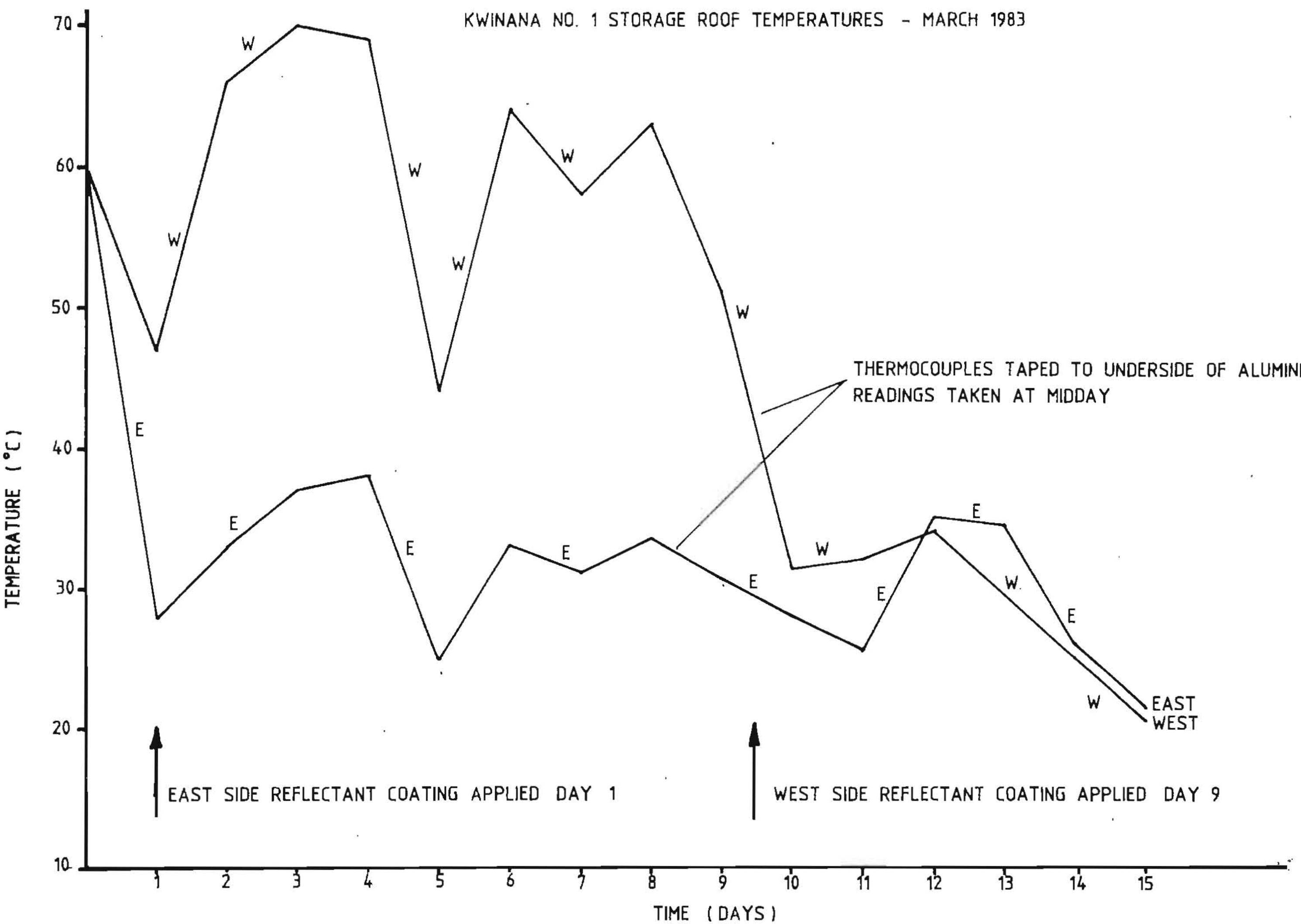
KWINANA NO. 1 STORAGE ROOF TEMPERATURES - MARCH 1983
TEMPERATURE (°C)
70
60
50
40
30
20
10
W
E
THERMOCOUPLES TAPED TO UNDERSIDE OF ALUMINIUM CLADDING
READINGS TAKEN AT MIDDAY
EAST
WEST
EAST SIDE REFLECTANT COATING APPLIED DAY 1
WEST SIDE REFLECTANT COATING APPLIED DAY 9
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
TIME (DAYS)

FIG. 1.

Blanking plates were made to fit over the large doors. These were fixed over the door frames externally with Flexacryl providing a removable seal. Existing man access doors were replaced by specially designed self-sealing doors. A removable form was made to clamp snugly over the conveyor belt to provide a seal at the point where the conveyor entered the reclaim tunnels and the above-grain area.

Modifications for Gas Introduction, Circulation and Removal

The store was equipped with 65 possible gas injection points: these being at ground level at 32 ports in the east and west walls (long axis of the store runs N-S) and at every third one of the reclaim valves in the floor of the store. The ports were fitted in the store with perforated baffles to prevent wheat blocking them. The reclaim valves were modified to admit gas and not to pass wheat by placing a perforated removable screen over the opened valve. The valves are above the two outloading tunnels that run the length of the storage below the floor. The tunnels themselves were converted and sealed to act as the ducts to deliver the gas to the valves. We recognise that the number of introduction points may be excessive, but were provided to ensure that purging and venting could be controlled efficiently. Since neither purging with CO_2 nor venting of such a size of storage had been attempted previously we were not certain how many or few points would, in fact, be required.

To maintain an even concentration of CO_2 in the store after purging, we recognised that it would be necessary to provide recirculation. In particular this is to avoid the formation of regions of low concentration which otherwise tend to form in the top of tall structures under CO_2 (Wilson et al, 1980). The recirculation system fitted (Fig 2) consists of eight ducts on both the east and west walls and a further two similar systems on the north and south gable walls.

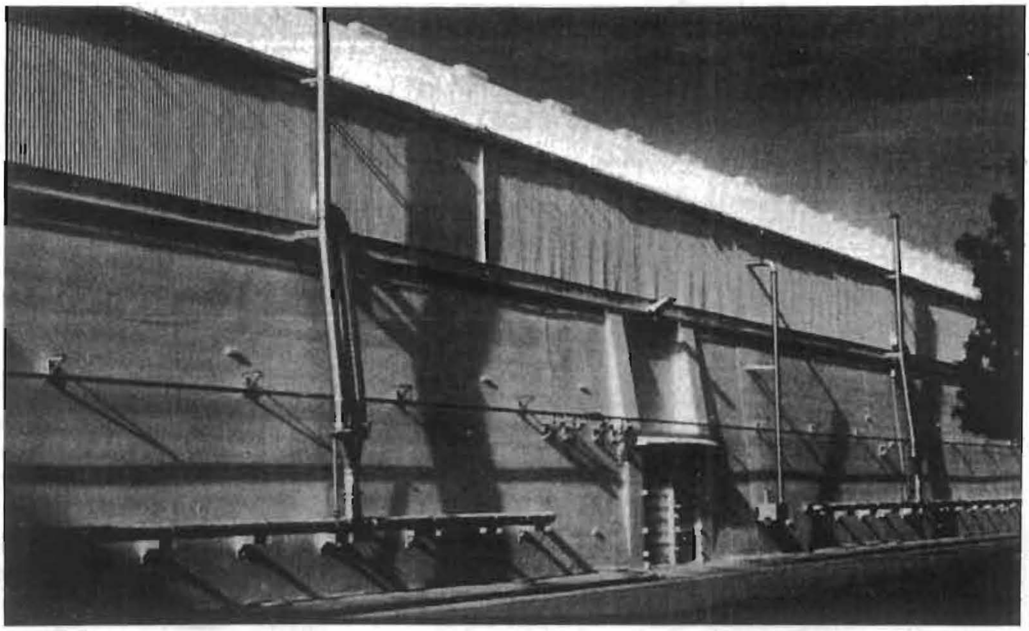

Fig 2 (a) Recirculation/venting ducts - side wall.

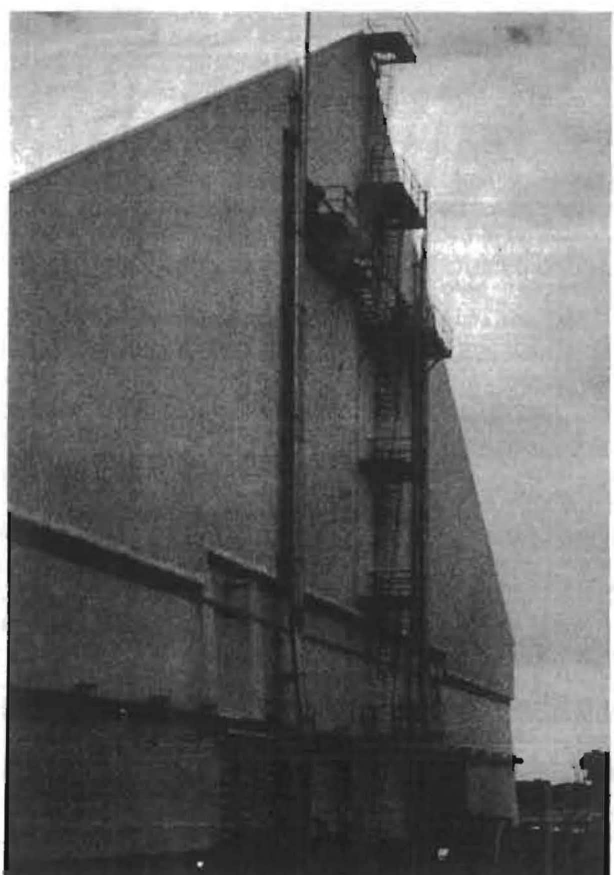

Fig 2 (b) Recirculation/venting ducts - end wall.

The latter systems run from each end of the tunnels. All systems run up to the ridgeline of the roof, running externally up the concrete wall, passing through the bottom of the aluminium cladding and then along the roof trusses or end walls to the roof ridge inside. The total system was designed to recirculate one gas volume of the store per day. The types and ratings of the various fans fitted are given in Table 2. It is expected that it will be found that some of these will not be required, but again in the absence of any experience with CO_2 in such large sheds as a guide we wished to err on the side of caution. It is planned to carry out trials to determine the actual quantity of fans and ductwork required.

A further duct was fitted above each recirculation fan in the recirculation system on the walls (Fig 2). This duct led to atmosphere above the gutter line and was fitted with a valve that could be opened so that gas was drawn out of the shed and vented rather than recirculated. Four large sealable fans (Table 2) were fitted in the gable ends above the wheat level, two at each end to provide ventilation in the headspace. It was so arranged electrically that the fans in one gable blew air into the shed while the others sucked out, thus creating a through draft to remove CO_2 or airborne dust as necessary. The venting fans were sized such that the storage could be cleared of CO_2 in two to three days. The fans can be operated to give flows in either direction through the shed, the direction being chosen so that the wind at the time assists the ventilation rather than blowing against the fans. Two small fans were installed in the bulkhead sealing the tunnels where the reclaim conveyors emerged. These were to clear any residual gas in the tunnels, after the venting of the store, so that it was safe for personnel to enter.

Table 2 - Details of fans fitted to No 1 Storage, Kwinana.

Fan Position	Number	Size	Ratings
On east and west walls	16	300mm axial, 2900rpm, 0.375kW	$0.16 m^3 s^{-1}$ at 425Pa
On north and south ends connected to tunnel	4 of 2 fans in series	300mm axial, 2900rpm, 0.375kW	$0.45 m^3 s^{-1}$ at 500Pa
On gable ends above wheat line	4	1500mm axial, 720rpm, 11kW	$27 m^3 s^{-1}$ at 180Pa
In tunnel at entrance	2	300mm axial, 1480rpm, 0.375kW	$0.45 m^3 s^{-1}$ at 180Pa

Monitoring of Gas Concentration

In view of the experimental nature of CO_2 use in the shed, some 60 gas sampling lines were arranged to give samples of gas from within the storage and grain bulk. Further sampling lines were fitted to give samples from points outside the seal to check for safety, particularly in low lying areas such as by the entrance to the reclaim tunnels where the dense CO_2 gas may collect if it leaked from the store.

Pressure Relief Valves

Twelve oil-filled pressure relief valves (Fig 3), six on the east wall and six on the west, were fitted to the storage. The fitting was arranged to relieve pressures high in the storage. These valves were set to begin to pass air or gas at 70 Pa pressure differential and to give a full flow of air or gas at 300 Pa.

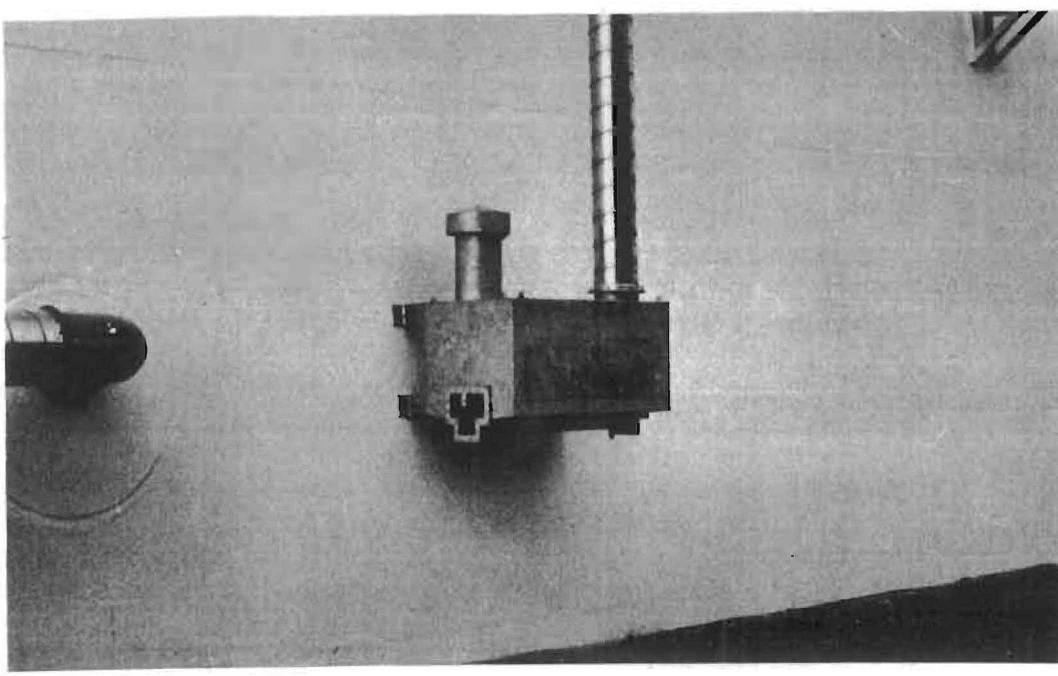

Fig 3 Pressure relief valve.

Safety Aspects

The installation is designed to be at least as safe as the storage was, prior to sealing. Provision has been made for continuous monitoring of CO_2 levels in and around the storage. Self-contained breathing apparatus is available in case of accident for conditions where CO_2 levels may be hazardous to humans.

The four gable end fans (Table 2) are of a size that can provide significant through-flow of air in the storage, thus removing airborne dust and minimising worker discomfort and dust explosion hazard during grain handling.

The skylights in the storage have been sealed into the roof, so that adequate daylight still enters the storage. The white finish on the walls internally is designed to assist visibility within the store.

The pressure vents, set at 70 Pa, are to prevent excessive pressures on the structure. The roof is designed to withstand up to 1000 Pa internal pressure.

Costs of CO_2 Use and Alternatives

One reason for choosing CO_2 as a disinfestation method for Kwinana No 1 Storage was that its cost compared favorably with alternatives. Table 3 summarises running costs of various processes. The cost of grain protectants used at Kwinana is currently much less than in much of Australia since bioresmethrin need not be used. It is unlikely that this situtation will persist and we expect that it will be necessary to use this or a similar material soon with the almost inevitable arrival of OP-resistant Rhyzopertha dominica in the region. It is thus realistic to use the 'most of Australia' cost of protectants as a comparison with that of various fumigants.

Table 3 - Cost of materials used for disinfestation of grain.

	COST (cents per tonne of grain	
	KWINANA	MOST OF AUSTRALIA
GRAIN PROTECTANTS		
Fenitrothion	9	11
Bioresmethrin	Not Used	70
Alternatives to fenitrothion	Not Used	15+
Total	9	81-85+
FUMIGANTS		
Methyl bromide	7	7
Phosphine	2-8	14
CO_2 (Vertical storage)	4	30
CO_2 (Horizontal storage)	5	50

The cost of sealing of a storage can be considered almost completely as a cost of use of fumigants and controlled atmospheres and must be included in a proper comparison of costs of different methods (Connell and Johnston, 1981). For No 1 Storage at Kwinana these costs are:

Sealing	\$1.16 per tonne
Fittings, duct work	\$0.99 per tonne
Maintenance	\$0.28 per tonne
Giving a total cost of	\$2.43 per tonne

Maintenance includes allowance for renewal of the heat reflectant coating on the roof after ten years.

By discounting the value of the capital cost suitably it is possible to make allowance for interest paid on capital invested and for inflation so that a proper comparison can be made with processes such as the use of pesticides in which capital charges can be negligible. Costs for CO_2 use in No 1 Storage adjusted in this way are given in Table 4.

Table 4 - Capital and maintenance costs (1983 prices) for conversion of No 1 Horizontal Storage, Kwinana for use with CO_2 disinfestation.

	Time Scale (Years)	Cost (\$A)
Sealing	20	348,000
Fittings, ductwork, electrical	20	297,000 (b)
Replacement of reflectant finish (a)	10	74,000
Maintenance	1	5,000

Discounted Annual Cost (\$A) (c)	Annual Cost Per Tonne (\$A, One Fill Per Year)	Annual Cost Per Tonne (\$A, Four Fills Per Year)
23,300	0.18	0.04
19,800		
4,800		
5,000		

a) Cost of initial roof treatment included in sealing

b) Cost allows for replacement at 10 and 20 years

c) Costs discounted at a real discount rate of 3% per annum over 20 years

These figures obviously provide justification for our choice of CO_2 as an insect control process at Kwinana. The actual cost is much reduced compared with that for a country site by economies of scale, cheapness of pipeline CO_2 as compared with liquid CO_2 and multiple use of the store each year.

CONCLUSION

Controlled atmosphere techniques are simple in concept. There is now information that they can be applied successfully and routinely in Australia on a commercial scale (Co-operative Bulk Handling Limited, unpublished results; Banks et al, 1980).

The use of CO_2 at the No 1 Storage at Kwinana has several features not yet investigated fully. There is thus an element of experiment in this work. In particular, we plan to use only 40% CO_2 in air as the atmosphere and to maintain this atmosphere for an extended period (several weeks) by adding further CO_2 from the pipeline supply as required to compensate for losses by absorption and leakage. The storage is larger than any which hitherto has been sealed and treated with controlled atmospheres and there is little data to guide us on how gases will disperse and distribute within the store. Despite these unknowns we are confident that the technique will prove successful in this large structure.

In the coming years, assuming the treatment of the No 1 Storage is commercially successful, Co-operative Bulk Handling Limited, plans to extend its program of sealing silos and horizontal stores to a standard suitable for fumigation or controlled atmosphere use. Both the cell block and the No 2 Horizontal Storage (250,000t capacity) at Kwinana are to be included in the program, giving a facility capable of holding over 900,000t of grain under CO_2.

Note Added in Proof

The No 1 Storage at Kwinana gave a satisfactory pressure test (full store, 30 minutes pressure decay, 180-90 Pa) on 5/9/83. it was filled with CO_2 in the period 6th September-13th October 1983 using 527t CO_2 and giving an atmosphere of 40% CO_2 throughout the structure. On 14th October 1983 the atmosphere was removed. Most of the 300,000t of pesticide-free grain, treated in the store was shipped from Kwinana over the period 3/11/83 to 5/2/84. 34,000t still remains in the store. No live insects have been detected in the store or in the grain exported, despite extensive sampling. There was no survival of caged test insects (all developmental stages of Rhyzopertha dominica, strain KRD6 multi O.P. resistant fenitrothion RF114) exposed in the store during CO_2 treatment.

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W.A. Department of Agriulture

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LEAK DETECTION IN SEALED GRAIN STORAGES

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ABSTRACT

In Western Australia, a pressure test specification is written into contracts for construction of sealed grain storages or sealing of existing structures. On the occasions where the specification is not reached it is necessary to be able to detect the unsealed areas, the leaks, so that they can be rectified. Various methods have been tried for this, but no general system has yet been developed that is completely satisfactory. Methods tried include use of fluorescent dusts, smoke testing, tracer gas use, detection by sound, thermographic systems and soap testing. To date, soap testing has been found to be the most effective system, detecting many leaks not obvious by inspection. After rectification of leaks found by soap testing, the structures under test, silos and large horizontal storages are usually satisfactorily sealed. The use of the various methods is described.

INTRODUCTION

A contract for the sealing of a storage will have included in its text a method of assessing the degree of seal achieved and a standard above which the contract completion is acceptable. It is also advisable to include a period of time where maintenance of the seal to that standard remains the responsibility of the contractor. This period in Western Australia is 5 years. The life of the seal beyond that period is a function of the durability of the material used as the sealing membrane, the thickness of the seal coat applied, the perfection of the application and to some extent the stability of the storage structure.

It is inevitable that at some time the seal on the storage will no longer be adequate and the treated storage will not pass the sealing assessment test (eg. a pressure test). The problem may occur during the initial sealing attempt or may arise during subsequent use of the system. In either case it is necessary to have some simple, efficient procedure for location of leaks and imperfections in the overall seal so that they can be treated and the sealing of the storage brought within specification.

In Western Australia, we have tried several methods of leak detection for storages with varying degrees of success. This paper summarises our experience to date. As yet we have not found a universally applicable system.

METHODS OF LEAK DETECTION

There is a wide variety of possible approaches to the problem of detection of leaks in an imperfectly sealed grain storage. After rectification of those faults obvious from visual inspection, it has been our experience that storages often may still not meet our pressure test standard (typically pressure decay of 200 Pa to 100 Pa in 15 minutes) although they may come close to it. In many cases, the success of a sealing contract appears to depend largely on the experience of the contractor and his workforce. Even then the seal may be unsatisfactory. Several methods have been used to detect the remaining leaks in the fabric of the store to be sealed.

Fluorescent Dusts

Fine fluorescent dusts are available (SWADA [London] Ltd or in Australia Abel Lemon and Co Pty Ltd) in a number of different colours. These can be used as tracers to detect leaks. The structure under test is pressurised with a fan and the dust fed into the fan inlet. The dust disperses through the structure and is expelled through the imperfections in the fabric. The outside of the structure is then inspected under a near ultra-violet light ('black light'). The imperfections are indicated by the easily seen fluorescence of the dust adhering around the region of the leak. After a further attempt at sealing the process can be repeated using a different colour of dust to check if the treatment has been effective.

The process requires a substantial pressure differential to force the dust through fine leaks, typically about 2 kPa is used, and thus is unsuitable for use in sheds. We have had some success in detecting leaks in concrete cells using these dusts.

Smoke Testing

Smokes can be generated within a partially sealed system and the leaks detected by the emission of smoke. The smoke can be produced by various means (eg. a 'swing fog' unit or by burning flares or smoke bombs).

We have found this method useful as a means of demonstrating the gas loss that can be expected from small, unsealed structures (eg. 15 tonne farm bins), but it has not been effective on larger, partially sealed systems. Since it operates under very low pressure, the results are influenced by wind and smoke only will issue from leaks in the negative pressure, leeward side of the structure. Leaks to windward are not detected. It suffers from one major disadvantage: the residues from the smoke present a possible contamination hazard and must be washed from the store before it is filled with grain.

Tracer Gas Systems

Any gases easily detectable in the field can be used to find leaks. The system under test must be pressurised slightly if all leaks are to be located. Under natural conditions, gas within a structure will only issue from leaks under negative pressure with respect to the external atmosphere.

This method is somewhat cumbersome and costly, though it can be quite effective. It may find leaks difficult to trace by other means such as those through the ground from the floor of the storage that may issue into the open air some considerable distance from the leak in the fabric itself. The main drawback is that analysing instruments need to take a sample for identification. This requires close and detailed attention to all potential leak areas. Furthermore, the immediate dilution of gases when released to the atmosphere make the detection of small leaks difficult. Leaks from storages under phosphine may be detected initially by smell and then located using gas detecter tubes (eg. Drager tubes).

Audio Detection

A pressure differential forcing air through a restriction may make a sound detectable to the normal ear. Typically a pressure differential of at least 200 Pa is required to make leaks hiss or whistle. Many leaks can be detected in this way and this test is our most commonly used preliminary method of checking for leaks. Very small and large leaks do not make an audible noise and are thus not detected. The test is best carried out in the later part of the night when conditions are usually very still and quiet.

We have tried to increase the sensitivity of this method using directional microphones and amplifiers to pick up the sound but with little success. The sounds of nature, while fascinating, detract from the value of the amplification.

Thermographic Survey

We have attempted to use infra red detection equipment to locate leaks remotely. A scanning device which detects infra red radiation from thermal energy on an object surface and converts this into an electric signal is directed to the storage under test. A monitor presents the information detected by the scanning device onto a T.V. type screen. The differential intensities of infra red radiation received can be readily identified. Various methods of information presentation are available and the greatest problem is the interpretation of that information.

While not immediately successful, it is intended to further investigate the potential of thermography in the near future. Among these potentials are:

a) Use of long focus lenses to study inaccessible parts from ground level equipment.

b) The use of colour thermograms to provide a much greater sensitivity to thermal differences.

c) The infra red absorption band of CO_2 makes this gas detectable with available equipment and provides the opportunity of combining tracer gas leak finding systems with actual controlled atmosphere application.

Soap Bubble Systems

If a detergent/water mixture is sprayed onto the surface of an imperfectly sealed storage that is under slight positive pressure (eg. 200 Pa), bubbles form over the leaks. A mixture of 2% household detergent in water has been found satisfactory for the process. The test is best carried out under windless conditions. It is capable of detecting both large (eg. 10 mm^2) and fine leaks (eg. concrete porosity).

This system has been found particularly useful for the location of leaks remaining in horizontal stores after a first attempt at sealing and after rectification of those imperfections detected by inspection or sound. Finding and marking of the leaks remaining in a 25,000 t store takes about 8 man hours. One person is required to operate the spray equipment, while another carries out close inspection of the surface to see bubbles from small leaks.

Figs 1 and 2 show soap testing of a storage in progress.

DISCUSSION

All the leak detection methods described above have been tried by Co-operative Bulk Handling Limited in their recent sealing programme on horizontal sheds. None have been discarded as useless and particular methods are used for different situations. The soap test method has proved to be the most useful and is now routinely used after a sealing attempt on a storage to detect any imperfections in the coating.

Although these methods have proved successful there is still a need for a remote sensing system capable of detecting leaks in a large structure from ground level and providing a measure of the significance of the leak. It is hoped that some form of radiation sensing such as the infra red thermography may provide this answer.

Figure 1. Detergent solution being applied to the roof of a newly sealed storage.

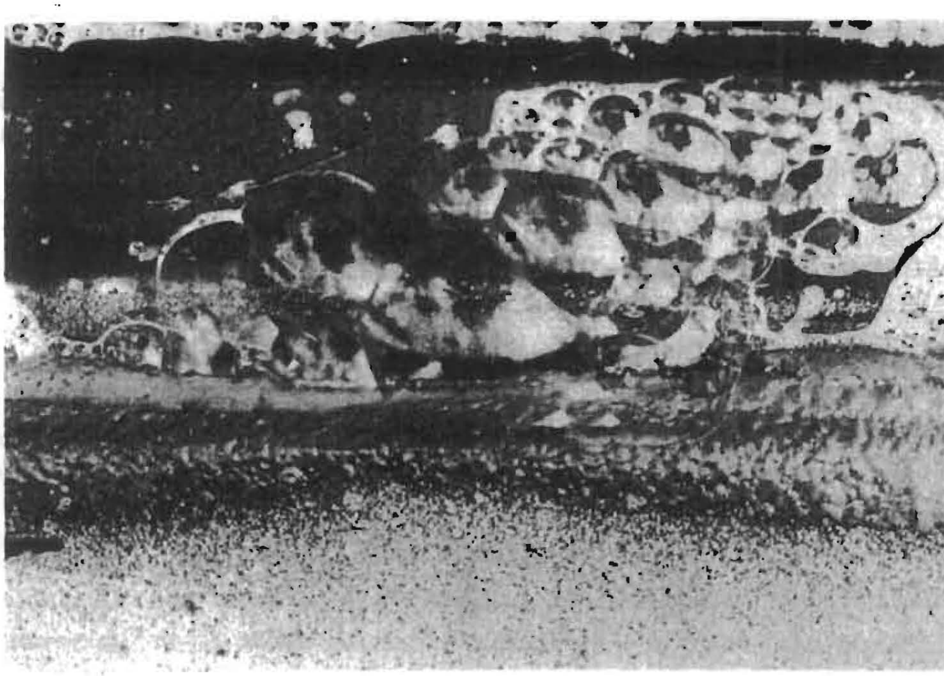

Figure 2. Bubbles developing around a leak in an imperfectly sealed storage that is under slight positive pressure and has been hosed with detergent solution.

IMPORTANCE OF PROCESSES OF NATURAL VENTILATION TO FUMIGATION AND CONTROLLED ATMOSPHERE STORAGE

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Abstract: For either fumigation or controlled atmosphere (CA) treatments, gas losses from the treatment enclosures caused by the processes of natural ventilation must be reduced to a very low rate. A model is presented which may be used to calculate the total rate from the maximum expected individual contributions of the various phenomena giving rise to gas loss. The ventilation rates expected from wind and the chimney effect are dependent on the level of sealing of the enclosure and are given in terms of the decay time, as assessed by a pressure decay test. The ventilation produced by temperature and barometric variation is not very sensitive to the level of sealing. The magnitude of the ventilation expected from the leak-dependent and -independent effects are compared for four types of storage. The relevance of this method of analysis to fumigation and CA treatments is discussed and maximum rates of loss tolerable by various methods are given. It is concluded, *inter alia*, that

(a) the pressure decay test time is only an approximate indication of the possible gas loss rate,
(b) the decay time specified for a particular system should take into account the influence of the expected environmental forces and geometry of the enclosure,
(c) it may be necessary to curtail gas loss by methods other than sealing after a certain minimum standard is reached, and
(d) it is unlikely that the gas loss rate in storage structures can be reduced to a level permitting simple hermetic storage of dry grain without very efficient thermal insulation and sealing.

INTRODUCTION

Both fumigation and controlled atmosphere techniques for insect control in grain rely on alteration of the composition of the gas mixture within an enclosure. The enclosure may be either a permanent structure, such as a grain storage, or a temporary system, such as a fumigation tent or a grain bulk covered with a PVC membrane. The gases added to alter the atmosphere in the enclosure may be either toxic ones, such as in fumigations with methyl bromide or phosphine, or particular atmospheric constituents, such as CO_2 or nitrogen, as used in controlled atmosphere (CA) techniques. After modification of the enclosed atmosphere, the average and local compositions of the gas mixture must be maintained within predetermined limits for the exposure period. If the treatment is to be carried out efficiently and safely with minimal use of added material only a very low rate of natural ventilation is permissible. Table 1 gives the maximum ventilation rates tolerable for various treatments. In simple grain storage enclosures such low rates can only be attained if specific and often costly measures such as sealing are taken to minimise gas losses.

TABLE 1.

Ventilation rates tolerable in various insect control processes using gases.

Process	Maximum ventilation rate (d^{-1})	Reference
Hermetic storage of dry grain	0.026	based on Oxley *et al.*, 1960
N_2-based CA (long term exposure)	0.05	Banks and Annis, 1977
CO_2-based CA ('one-shot')	0.07	Banks *et al.*, 1980
Phosphine fumigation	0.10	Unpublished estimate (Banks and Annis)

For the optimal application of measures designed to reduce gas loss it is important to understand in quantitative terms the natural processes that cause gas loss from a structure. For instance, an appreciation of the underlying phenomena may provide a means to assess whether, in a practical situation, further attempts at sealing are of any practical value. Hitherto, studies of natural ventilation of buildings and other enclosures for grain storage have been concerned either with very well sealed or with intentionally ventilated systems. In the first case, gas loss can be described as a function of the variation in ambient and internal temperature and pressure (Barker, 1974; Newman, 1970; Moller and Pedersen, 1978; Meiering, 1982). In the latter case (see Banks *et al.*, in press), as with habitable structures (Macriss *et al.*, 1979; Anon., 1972; Blomsterberg and Harrje, 1979; Peterson, 1979), the ventilation rate is determined largely by the wind and the chimney effect for a certain level of sealing. In practice the gastightness of most enclosures to be treated with fumigants and controlled atmospheres is intermediate between these two extremes and all these forces can have a significant effect on gas loss.

This paper provides a simplified mathematical description of the action of the various forces involved in the natural ventilation of enclosures. The description can be used to predict the variation in the contribution of the various forces to the total gas loss under different environmental conditions and gastightness of the enclosure. It will be shown that a pressure decay test standard for the assessment of gastightness is not a precise concept and that there is a degree of judgement which must be applied in the setting of standards for particular situations. Furthermore, there are some forces whose contribution to total gas loss can be significant but which can best be restricted by strategies other than sealing.

In general the model is similar to that used by Banks *et al.*, (1975) to describe gas loss from freight containers, but with changes and some minor additions so that it is specifically applicable to grain storage systems and incorporates a term to describe particular flow characteristics of leaks.

Previously, there has been no conceptual framework available which would predict the dominant cause of failure in fumigants or CA treatments. A number of different single forces have been implicated in such failures in the past (e.g. wind (see Mulhearn *et al.*, 1976), chimney effect (Oxley and Howe, 1944; Bond *et al.*, 1977), diffusion (Lewallen and Brown, 1967)). An understanding of the contribution of the various forces causing gas loss, as provided below, may help to determine some of the environmental causes of failures in particular circumstances with more confidence than hitherto, and perhaps show how these problems can be avoided.

2. THEORETICAL BASIS OF MODEL†

The gas loss from an imperfectly sealed enclosure is largely dependent on the pressure across the leaks in the enclosure fabric and the size and flow characteristics of these leaks. There is an additional, small component of loss associated with molecular diffusion and therefore dependent on the gas composition of the enclosed and external systems. A number of simplifying assumptions are made here in order to describe the gas loss rate from an en-

† The notation used is summarized below.

closure with a conservative and easily evaluated model, suitable for design studies. These are:

(a) *The gaseous contents of the enclosure are well mixed, so that any element of volume lost will be at the average concentration of the contained gases at that moment.* In practice some of the gas lost may be regained in a cyclic process. Similarly air entering may be subsequently expelled without mixing. Also occasionally gas may be lost from a region rich in a particular component. The first two processes result in a lower effective ventilation rate than expected (as discussed by Malinowski (1971)) while the latter increases the rate.

(b) *That each of the forces causing gas loss acts independently and that their effects may be summed to give an estimate of the total gas loss.* It can be shown that the total interchange is never greater than the sum of the expected effect of each force acting in isolation (Sinden, 1978).

(c) *That the empirical equation*

$$Q = b\Delta p^n \tag{1}$$

describing the flow of gas, Q, through a leak with pressure difference, Δp, (Anon. 1972; de Gids, 1977; Blomsterberg and Harrje, 1979) holds throughout the range of pressures created by the individual forces without change of either the coefficient, b, *or the exponent,* n. When it is necessary to show the direction of the leakage, Equation (1) is used in the form

$$Q = b\frac{\Delta p}{|\Delta p|^{1-n}} \tag{2}$$

so that, Q, is positive when gas is lost from the enclosure and $\Delta p > 0$ (i.e. internal pressure > external pressure). Equation (1) is known not to hold over a wide pressure range and to be dimensionally unsound (Kreith and Eisenstadt, 1957), but to be a reasonable approximation (de Gids, 1977) over the pressures, 1 to 100 Pa, likely to cause significant ventilation in grain.

(d) *That the total leak area over a storage can be represented by two composite leaks with similar flow characteristics (i.e. same value of* n*) and of equal area and that the two leaks are distributed such as to maximise the effect of each individual force.* This may require that the leaks have different positions simultaneously for different forces.

(e) *That the variation in particular forces (e.g. temperature variation, wind pulsation) can be represented as sinusoidal functions of time.*

The overall effect of these assumptions is to provide a model which gives the maximum effect expected from each individual force and the maximum total ventilation rate for a particular set of conditions. In practice because of interactions between the individual forces and also the distribution of leaks the actual loss rate will usually be substantially less than predicted by the model. Allowance for this will be made in subsequent discussion.

To provide a complete description of gas loss over time, it is necessary first to consider three interrelated problems: (a) the description of gas losses produced by pressure or concentration differences across leaks, (Section 2.1) (b) the description of pressure or concentration gradients as produced by various environmental forces (Section 2.2) and (c) the description of the sealing level of an enclosure in a form which can be used to assess the effect on gas loss of pressure or concentration gradients across the enclosure walls (Section 2.3)

2.1 GAS TRANSFER EQUATIONS

2.1.1 Flow Through Leaks

Bulk flow of gas occurs through a leak where there is a pressure differential Δp, across that leak as described by Equations (1) or (2). The value of n varies between 0.5 and 1.0, depending on the flow characteristics of the leaks. In cases where $n = 0.5$, the value of b can be related to the area of the leak present through Torricelli's law (Kreith and Eisenstadt, 1957). Thus

$$b = \gamma A \sqrt{\frac{2}{\rho}} \tag{3}$$

When $n \neq 0.5$, a true size of leak cannot be given without detailed knowledge of the geometry of the leaks.

Some forces cause losses from all leaks simultaneously (e.g. thermal expansion), while others (e.g. wind) give a flow of gases through the enclosure, air entering at one point and the enclosure gases being lost at another. In the first case, Equation (1) applies. In the second, for the purposes of this model, the total leak of effective size, b, is divided into two leaks with equal value of n. It can be shown that the flow through two such leaks in series is given by

$$Q = b\Delta p^n \left(\frac{1}{\alpha^{-1/n} + (1 - \alpha)^{-1/n}} \right)^n \tag{4}$$

where α is the proportion of the total leak area represented by the smaller leak, and for $\alpha = 0.5$ (i.e. two equal leaks in series)

$$Q = \frac{b\Delta p^n}{2^{n+1}} \tag{5}$$

The magnitude of most of the forces involved in gas leakage varies with time. This gives rise to fluctuating pressures across the leaks with frequencies varying from many cycles per second, as in some components of wind turbulence, to yearly cycles, as with seasonal heating and cooling. Substituting in Equation (2), the flow at any instant then is given by

$$Q = b\frac{\mathrm{f}(t) - \mathrm{f}'(t)}{|\mathrm{f}(t) - \mathrm{f}'(t)|^{1-n}} \tag{6}$$

where $\mathrm{f}(t)$ and $\mathrm{f}'(t)$ are time-dependent functions describing the internal and external pressures.

2.1.2 Evaluation of Ventilation Rates

The ventilation rate†, k, is defined as (e.g. Lagus, 1977):

$$k = \frac{\Delta V}{Vt} = \frac{Q}{V} \tag{7}$$

and, at constant density,

$$k = \frac{\Delta m}{mt} \tag{8}$$

The ventilation rate is used here as a measure of the effect of the various forces, where V and m are the volume and mass of gas within the enclosure, Q is the volumetric rate of loss of gas and ΔV is the volume of gas lost at constant pressure and Δm the mass of gas lost over time, t. Note that the ventilation rate is calculated on the basis of flow in one sense only, in or out of the enclosure.

The ventilation rate is a measure of rate of loss of a gaseous component from a system since

$$c_2 - c_{ext} = (c_1 - c_{ext})\mathrm{e}^{-kt} \tag{9}$$

where c_1 and c_2 are the initial concentrations of the component and the concentration after time, t, within the system and c_{ext} is the

† Also known as the air change rate, the infiltration rate, gas interchange rate or gas loss rate constant and sometimes, incorrectly, simply as the gas loss rate.

external concentration of the component.

For evaluation of k, it is often convenient to express the losses in terms of rate of mass loss from the system. Since

$$Q = -\frac{1}{\rho}\frac{dm}{dt} \tag{10}$$

the rate of loss of mass is given by

$$\frac{dm}{dt} = \rho b \frac{f(t) - f'(t)}{|f(t) - f'(t)|^{1-n}} \tag{11}$$

combining Equations (6) and (10). Generally, for the periodic functions encountered here, this expression is best solved iteratively with the change in mass estimated over one or more complete cycles. The change in mass, δm, during the i^{th} interval of δt is given by

$$\delta m = \rho b \left(\frac{f(t_i) - f'(t_i)}{|f(t_i) - f'(t_i)|^{1-n}} - \frac{f(t_{i-1}) - f'(t_{i-1})}{|f(t_{i-1}) - f'(t_{i-1})|^{1-n}} \right) \tag{12}$$

and

$$\Delta m = \frac{\sum|\delta m|}{2} \tag{13}$$

The average interchange rate can then be found using equation (8).

The actual interchange produced by a fluctuating pressure difference is dependent on the response time of the contained system, here measured as the pressure decay time, t_d (see Section 2.3), relative to the period of fluctuation, τ. When t_d is small compared with τ, the interchange can be calculated directly from the amplitude of the fluctuations (i.e. with no damping of effect). When t_d is similar to or greater than τ the actual interchange is less than this value and dependent on the ratio of t_d to τ and the value of n as shown in Figure 1. The values in Figure 1 were calculated by evaluating the equation

$$\frac{dm}{dt} = -\rho b \frac{\frac{mR\overline{T}}{VM}(1 + a\cos\omega t) - p_{ext}}{|\frac{mR\overline{T}}{VM}(1 + a\cos\omega t) - \overline{p}_{ext}|^{1-n}} \tag{14}$$

iteratively until a stable value of the interchange per cycle was obtained, with b, a function of t_d, given by Equations (33) or (34). This rate was then expressed in terms of time units of t_d/τ Equation (14) is derived from Equation (11) with f(t), the internal pressure, given by

$$f(t) = \frac{mRT}{VM} \tag{15}$$

and $f'(t)$ the external pressure,

$$f'(t) = \overline{P}_{ext}(1 + a\cos\omega t) \tag{16}$$

It can be seen that when $\tau > 10t_d$ there is little damping in effect but when $\tau < 10t_d$ the interchange produced by a fluctuating pressure will be reduced compared with the expected undamped value.

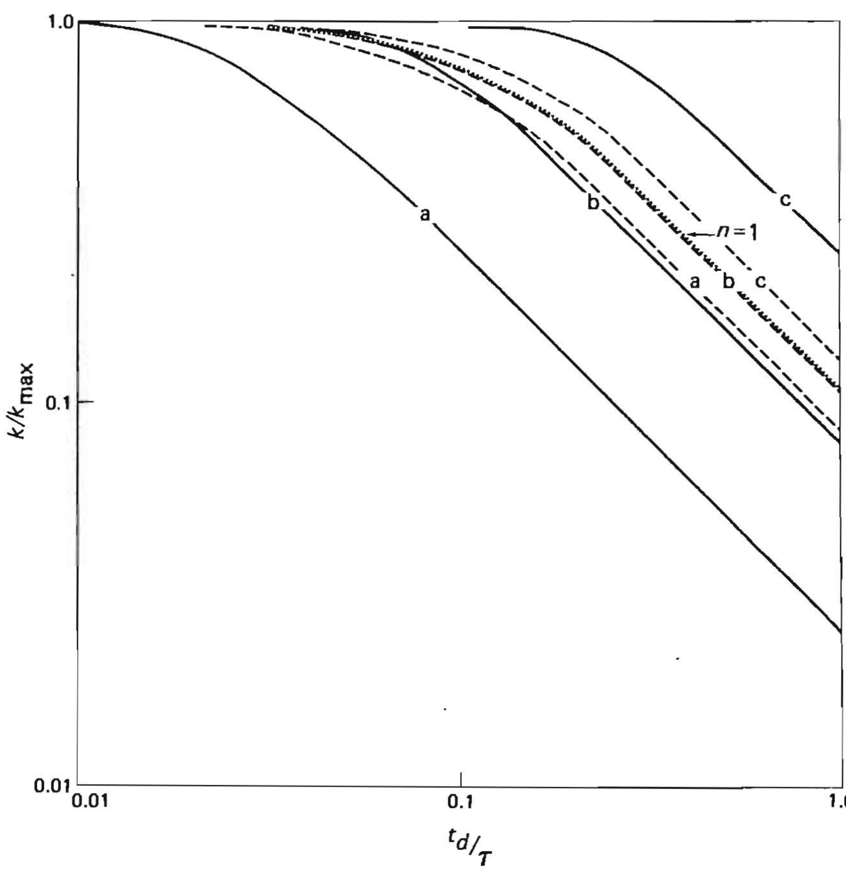

Fig. 1. Damping effects for cyclic phenomena with periods of similar magnitude to the decay time (500-250 Pa), t_d, for amplitude values of $a = 10^{-1}$, 10^{-2} and 10^{-3} (lines marked a, b and c respectively) and values of $n = 0.5$ (——), 0.8 (-----) and 1.0 (·········) (One line only for $n = 1.0$ as this is independent of value of a). Calculated from Equation (15) as described in text.

2.2 EFFECTS OF INDIVIDUAL FORCES

Gas losses through leaks from an enclosure containing stored grain can be caused by the following phenomena:

a. Temperature variation;
   a.1 Variation in the headspace,
   a.2 Variation in the bulk.

b. Barometric pressure variation;
   b.1 Tidal variation,
   b.2 Long term (synoptic) variation.

c. Wind;
   c.1 Wind at a steady speed,
   c.2 Fluctuating components of wind (pulsation and turbulance).

d. The chimney or stack effect;
   d.1 Driven by temperature variation externally,
   d.2 Driven by composition differences.

e. Molecular diffusion.
   e.1 Diffusion through leaks,
   e.2 Permeation.

2.2.1 Temperature variation

There are two thermally distinct regimes in an enclosure around a grain bulk: the headspace and the grain bulk. These must be treated independently to calculate the gas loss from a storage, since while the temperature in the headspace may change rapidly in response to changing ambient conditions, the temperature of most of the bulk changes only very slowly because of its low thermal diffusivity.

2.2.1.1 Temperature variation in the headspace.

The temperature in the headspace fluctuates with the daily cycle of temperature and solar radiation. Superimposed on this daily change there may be shorter period fluctuations caused by changes in solar radiation, wind cooling and ambient temperature. Because the period of the daily cycle is long compared with the pressure decay time of sealed storages (see Section 2.1), pressure equilibrium is maintained across the storage fabric during the heating and cooling cycle. It can be shown, from simple gas laws, that the quantity of gas lost, ΔV, during a cycle of amplitude ΔT is then given by

$$\Delta V = \frac{V_{HS}\,\Delta T}{T_{min}} \qquad (17)$$

and thus the ventilation rate due to temperature variation, k_T, is given by

$$k_T \doteq \frac{V_{HS}}{V}\frac{\Delta T}{T_{min}t}, \; \Delta T > 0 \tag{18}$$

When the time period of the variation in headspace temperature is similar to or less than the pressure decay time of the enclosure, there will be a periodic pressure difference across the leak and the actual interchange will be less than expected from Equation (18). The quantity of gas lost can be calculated using Equation (7) with Δm estimated numerically as previously (Equations (12) and (13)) from the equation:

$$\frac{dm}{dt} = -\rho b \frac{\frac{mRT}{VM} - \overline{p}_{ext}(1 + a\cos\omega t)}{\left|\frac{mRT}{VM} - \overline{p}_{ext}(1 + a\cos\omega t)\right|^{1-n}} \tag{19}$$

an equation derived from Equation (11) with

$$T = \overline{T}(1 + a\cos\omega t) \tag{20}$$

2.2.1.2 Temperature variation in the bulk

External temperature fluctuations cause grain in contact with exposed parts of the enclosure to change in temperature, leading to changes in temperature of the interstitial gases with consequent pressure changes and possible leakage. Daily temperature fluctuations, unlike long term seasonal fluctuations, do not penetrate far into the grain bulk (ca. 10 cm, (Babbitt, 1945)).

It can be shown (see Appendix) that a grain bulk subject to a periodic external temperature variation obeying Equation (20) at its surface, loses gas at a rate, k_{Tb}, given by

$$k_{Tb} = \frac{2aNA}{\overline{T}Vt}\sqrt{\frac{\kappa}{\omega}} \tag{21}$$

2.2.2 Barometric pressure variation

Atmospheric pressure undergoes tidal and long-term cyclical fluctuations with some abrupt changes caused by atmospheric phenomena such as thunderstorms and cyclones. A change in external pressure will result in a pressure difference across leaks, ΔP, in an enclosure resulting in losses given by

$$\Delta V = \frac{V\Delta P}{P_{max}} \tag{22}$$

Thus the ventilation rate, k_p, caused by these changes is given by

$$k_P = \frac{\Delta P}{P_{max} t}, \Delta P \leqslant 0 \tag{23}$$

Because barometric fluctuations have long periods compared with storage pressure decay times, there is no appreciable damping of effect from sealing (see Section 2.1).

Note that barometric fluctuations cause loss from the complete gas volume in the enclosure (i.e. both headspace and interstitial spaces) in contrast to some types of temperature variation.

2.2.3 Wind.

The effect of wind can be divided into two components: that produced by the mean steady wind speed and that produced by fluctuations from pulsations and turbulence about this mean speed.

2.2.3.1 Mean wind speed effect

The pressure induced by wind at a point is given by (Mulhearn *et al.*, 1976; de Gids, 1977)

$$\Delta p = \frac{C \rho u^2}{2} \tag{24}$$

where C is a pressure coefficient varying with the location of the measuring point and orientation relative to the wind. The interchange rate is then given by

$$k_w = \frac{b}{2^{n+1} V} \left(\frac{\Delta C \rho u^2}{2} \right)^n \tag{25}$$

where ΔC is the difference in the pressure coefficients at the two leaks (substituting in Equation (5) and then (7)). The actual value of the interchange produced will be sensitive to ΔC. Many parts of a storage will have C values close to zero, but some small regions may have values exceeding $\pm$ 2.0 (Mulhearn, *et al.*, 1976; Banks *et al.*, in press). ΔC can thus vary from approximately zero up to 4.0 in exceptional cases.

2.2.3.2 Wind pulsation and turbulence

The loss rate to be expected from wind pulsation and turbulence cannot be estimated accurately for grain storage enclosures as it is subject to too many random factors and computational uncertainties.

The mechanisms by which pulsation and turbulence can cause gas loss have been discussed by Malinowski (1971). He recognised that because of the rapid change in direction of flow of gas through a leak driven by these forces, the actual interchange produced may be a small fraction of that of the theoretical maximum. Air entering at one time may be largely expelled at another, bringing with it little of the internal gases (Cockroft and Robertson (1976) found only about one third of the air entering under their conditions mixed with the internal gases). The actual fraction will be dependent on the frequency and amplitude of pulsation and the rate of mixing within the system and has not been studied adequately for reliable values to be available. Because there is a broad range of frequencies of fluctuation involved, the approximations used to calculate the damping of the effect of single frequencies, as for Fig. 1, may not be appropriate. Furthermore, in most practical situations, pressures from wind fluctuations will be superimposed on substantial mean wind pressures. In such cases and where there is already leakage caused by these pressures, the fluctuations will merely modulate the flow and cause little change in interchange rate, except on those occasions where the amplitude is sufficient to change the sign of Δp and thus reverse the flow of gas through the leak.

In view of these uncertainties, we do not attempt to treat the wind pulsation and fluctuation effects mathematically but, based on data in Hill and Kusuda (1975), we assume

$$k_{wf} = 0.2 k_w \tag{26}$$

2.2.4 The chimney effect

The density of the gaseous contents of an enclosure may differ from that externally, either because it is of a different composition or at a different temperature. These density differences result in pressure differences across the enclosure fabric, causing gas loss if leaks are present. This phenomenon is known as the chimney or stack effect. The pressure difference across two leaks separated by a vertical distance, h, is given by (de Gids, 1977)

$$\Delta p = (\rho_{int} - \rho_{ext}) g h \tag{27}$$

With the external density, ρ_{ext}, varying with daily temperature fluctuations thus,

$$\rho_{ext} = \frac{MP}{R\overline{T}(1 + a\cos\omega t)} \tag{28}$$

and with the period of fluctuation much greater than the pressure decay time and the internal density remaining approximately constant, the rate of gas loss from this effect, at time, t, is given by

$$Q = \frac{b}{2^{n+1}}\left[gh\left(\frac{MP}{R\,\overline{T}(1 + a\cos\omega t)} - \rho_{int}\right)\right]^n \tag{29}$$

and the average value of the ventilation rate from the chimney effect, k_c, over the cycle

$$k_c = \frac{1}{2V}\int_0^{2\pi}\frac{b}{2^{n+1}}\left[gh\left(\frac{MP}{R\,\overline{T}(1 + a\cos\omega t)} - \rho_{int}\right)\right]^n d(\omega t) \tag{30}$$

2.2.5 Diffusion and Permeation

Losses by true molecular diffusion through leaks are always small compared with those created by other forces. The ventilation rate from diffusion, k_D, through a leak of area, A, and length, l, is given by (from Lewallen and Brown, 1967)

$$k_D = \frac{DA}{Vl} \tag{31}$$

Losses by permeation through the fabric of an enclosure are described similarly:

$$k_{pe} = \frac{KA'}{Vl'} \tag{32}$$

where l', is the thickness of the film and A', the area over which permeation occurs.

It will be noted that permeation refers to true transfer through the mass of the fabric of a permeable but intact film, such as PVC sheet, but is to be distinguished from gas losses through small imperfections, such as porosity in concrete, where bulk movement of gas occurs.

2.3 RELATING PRESSURE TEST DATA TO LOSS RATES

In the model given here, the pressure decay time, t_d, of the enclosure, as measured by a pressure decay test, is used as a measure of gastightness. In this test, air is introduced into the structure to raise the internal gas pressure to a value Δp_1 above atmospheric. The air supply is then shut off and the pressure is allowed to fall by natural leakage to a new value Δp_2. The time taken to fall from Δp_1 to Δp_2 is then a measure of the degree of sealing. Assuming isothermal conditions the time for the decay, t_d is given by (Sharp, 1982)

$$t_d = \frac{(\Delta p_1{}^{1-n} - \Delta p_2{}^{1-n})\,VM}{(1-n)RT\rho b}, \; n \neq 1 \tag{33}$$

and

$$t_d = \frac{(\ln \Delta p_1 - \ln \Delta p_2)\,VM}{RT\rho b}, \; n = 1 \tag{34}$$

These equations relate t_d to b for a particular value of n. Since these parameters also occur in expressions for k_w and k_c it is possible to relate k_w and k_c directly to a set decay time by substituting for b as found from Equations (25) or (30).

Pressure decay testing in its simple form does not determine the value of n. The value of n lies between 0.5 and 1.0 and, generally, for well sealed storage structures $0.8 < n < 1.0$ (Banks and Annis, unpublished data). We will use $n = 0.8$ as a the minimum likely value for subsequent example calculations. (Note: Meiering (1982) gave estimates of gas loss rates from silos under various levels of sealing but did not include wind and chimney effects and considered $n = 0.5$ only).

3. VALUES OF THE CONTRIBUTION OF INDIVIDUAL FORCES TO LOSS

Using the expressions given above, it is possible to calculate the maximum contribution of each individual force causing gas loss from an enclosure of defined standard gastightness, if the values of the various environmental parameters concerned are known. Table 2 gives the calculated gas interchange rates caused by the various forces for four enclosures, detailed in Table 3, sealed to give a 5 min decay time (t_d) from 500 to 250 Pa in a full structure. This time has been adopted by the Coordinating Committee on Silo Sealants in Australia as a design standard for fumigable structures. The parameter values used here to define the main forces are reasonable high values for grain storages in the open in summer in inland Australia, chosen from our own experience.

It can be seen from Table 2 that the effect of some forces is very dependent on the value of n, even though the enclosures give a fixed decay time. Furthermore, some forces cause negligible gas losses, < 0.005 day^{-1} (e.g. diffusion, synoptic variation of barometric pressure), but others have a major effect, notably wind and temperature variation. Furthermore, the ventilation produced by diffusion and by wind and the chimney effect is dependent on the

TABLE 2.

Interchange rates (day^{-1}) calculated for individual phenomena for a sealing level giving a 5 min pressure decay time (500 - 250 Pa).

Phenomenon	Parameter values used	Case 1 Farm bin			Case 2 Bag stack			Case 3 Silo bin			Case 4 Shed		
Temperature variation in headspace (daily)	Range: 10-40°C (Case 1, 2, 4) 20-35°C (Case 3)	.025			.012			.007			.045		
Short term temperature fluctuation in head space	± 2°C (Case 1, 2) ± 1°C (Case 4) ± 0.5°C (Case 3) every 30 mins for 12 hours	.034			.007			.009			.042		
Daily variation in grain bulk	Skin temperature variation 12-40°C (25.5 - 29.5, Case 3) $\kappa = 10^{-2} m^2 d^{-1}$, $N = 0.38$	.009			.002			<.001			<.001		
Barometric pressure variation (tidal)	± 1.5 mb about 1013 mb twice daily	.006			.006			.006			.006		
Barometric pressure variation (synoptic)	± 12 mb about 1013 mb every 6 days	.004			.004			.004			.004		
Permeation	Permeation coefficient .0025 $g\ mm\ d^{-1}\ m^{-2}\ (g\ m^{-3})^{-1}$, thickness 0.8 mm (Case 2 only)	-			.004			-			-		
	VALUE OF n	0.5	0.8	1.0	0.5	0.8	1.0	0.5	0.8	1.0	0.5	0.8	1.0
Steady wind	6.4 $m\ s^{-1}$ (Case 1), 7.9 $m\ s^{-1}$ (Case 2), 12.0 $m\ s^{-1}$ (Case 3), 10.9 $m\ s^{-1}$ (Case 4). $C = 2.0$	.090	.041	.024	.112	.057	.036	.169	.111	.083	.155	.095	.069
Wind pulsation and turbulence	Assumed 0.2 x steady wind value	.018	.008	.005	.022	.011	.007	.034	.022	.017	.031	.019	.014
Chimney effect driven by temperature variation	Internal temp. 27.5°C, daily external variation 15-40°C.	.010	.001	<.001	.014	.002	<.001	.037	.010	.004	.029	.007	.003
Chimney effect driven by composition differences	As above, but with 60% CO_2 internally	.035	.009	.004	.045	.016	.007	.135	.077	.053	.105	.051	.032
Diffusion	Diffusion coefficient $2 \times 10^{-5} m^2 s^{-1}$, path length 3mm, area of leak $1.6 mm^2$ (Case 1), $76 mm^2$ (Case 2), $650 mm^2$ (Case 3), $2000 mm^2$ (Case 4).	<.001	a	a	<.001	a	a	<.001	a	a	<.001	a	a

a Area of leak not calculated for $n \neq 0.5$. Rate constant likely to be $<<0.001\ d^{-1}$.

TABLE 3.

Details of storage enclosures used as examples for calculation of interchange rates.

Case No.	Storage (construction material)	Nominal capacity (wheat, tonnes)	Gas volume (loaded, m^3)	Head space volume (m^3)	Exposed Surface area (m^2)	Dimensions
1	Farm bin (unpainted, galvanised iron)	5	3.0	0.6	15.9	Cylindrical wall 2.1 m diam., 1.8 m to eaves. 30° roof pitch.
2	Bag stack (PVC - covered)	100	140	5	190	Rectangular 11.5 x 4.7 m, 4.2 m high.
3	Silo bin (unpainted, concrete)	2200	1220	160	1090	Cylindrical wall 11 m diam., 30 m to eaves. 30° roof pitch.
4	Flat storage (unpainted, galvanised iron)	55000	41300	15200	14200	Rectangular plan 137 x 52 m, 5.2 m to eaves. 30° roof pitch.

size and type of leak in an enclosure (i.e. dependent on values of b and n) while the effect of others is independent of leak size, except at very high degrees of sealing, where there is some damping of effect.

The sum of the interchange rates resulting from individual forces in the two groups are referred to here as $(k_{dep})_{max}$ and $(k_{indep})_{max}$ respectively. Thus

$$(k_{dep})_{max} = k_w + k_{wf} + k_c + k_d \tag{35}$$

and

$$(k_{indep})_{max} = k_T + k_{Tb} + k_P + k_{pe} \tag{36}$$

These rates are calculated using high values of the controlling parameters and without allowance for interactions and chance effects. They thus represent maximum values and are unlikely to be attained in practice, except in exceptional combinations of circumstances. Generally:

$$k_{dep} + k_{indep} \ll (k_{dep})_{max} + (k_{indep})_{max} \tag{37}$$

The actual value of k_{indep} may be slightly less than the estimate, $(k_{indep})_{max}$, as there may be some interaction between temperature and barometric effects so as to reduce the total effect. However, even when k_p is acting in directly the opposite sense to k_t its influence on k_{indep} will be small since k_p itself is small. The actual value of k_{dep} is much less accurately known but may possibly be less than half that of $(k_{dep})_{max}$. To achieve maximal effect from wind and the chimney effect the effective leaks must be of equal size and located both in regions of high and low C values, effectively across the structure, and also at the top and base of the bin. These two conditions cannot hold simultaneously. Furthermore, if the two effective leaks in series, required for the wind and the chimney effect to produce gas interchange, are not of equivalent size the expected interchange will be reduced. This effect is summarised in Table 4 (see also Anon. 1972, p.344). Also there is an interaction between the wind and chimney effect such that their combined action is less than the sum of the expected individual contributions.

Despite these uncertainties, the calculated values of $(k_{dep})_{max}$ and $(k_{indep})_{max}$ provide useful semiquantitative information on the effects of sealing and whether other strategies may be necessary to reduce gas losses to within a tolerable range for a particular process.

Figures 2a-2d show the variation in $(k_{dep})_{max}$ with pressure decay time, t_d, for the four types of storage treated in Table 3. The values of $(k_{indep})_{max}$ are also shown. The values of the two interchange rates are summarised for t_d = 5 mins in Table 5.

The following deductions can be made from information in Fig. 2 and Table 2.

(1) Sealing to a level that gives at least a few minutes pressure decay test time reduces $(k_{dep})_{max}$ very substantially. (Note: in the model used here $(k_{dep})_{max}$ is inversely proportional to t_d.)

(2) That the value of n has an important influence on the value of $(k_{dep})_{max}$ with the greater interchange expected with lower values of n for the same decay time.

(3) That the relative importance of leak-dependent and -independent interchange rates varies with type of structure. With the silo bin, $(k_{dep})_{max}$ is large relative to that from the farm bin, suggesting a higher sealing standard should be applied in the former case to give the same rate of interchange.

(4) The value of $(k_{indep})_{max}$ in three cases considered is similar in magnitude to the rates tolerable for phosphine fumigation and CA processes (Table 1). Even allowing for the uncertainty in the actual value of this parameter, some reduction in its value appears necessary as there is otherwise little latitude in the specification for the additional effects of wind and the chimney effect. Since reduction of k_{indep} cannot be achieved by sealing, it must be done by some other strategy (e.g. reduction of temperature variation by white-painting or shading, and use of breather bag systems).

(5) It appears unlikely, in the structures assessed, here that the leak-dependent losses can be reduced by sealing to a level which would permit hermetic storage (Table 1) even allowing for the interactions and chance factors discussed above.

(6) The value of $(k_{dep})_{max}$ appears excessive in the case of the silo bin at the currently used standard pressure decay time (5 mins, 500 - 250 Pa in a full system) for fumigable bins. It may be that some increase in the decay time standard is warranted to ensure that gas loss is not excessive even under very adverse environmental conditions. However it may also be

TABLE 4.

Influence of leak distribution on calculated loss rate for Case 3 (t_d = 5 mins, 500 - 250 Pa, full structure) for wind and chimney effect.

Ratio of leak area for air entry to gas loss	Value of α in Equation (4)	Calculated ventilation rate (day^{-1})[a]	
		Wind	Chimney effect with 60% CO_2 internally
1 : 1	0.50	0.084	0.053
1 : 2	0.33	0.074	0.047
1 : 3	0.25	0.063	0.040
1 : 5	0.167	0.046	0.029
1 : 10	0.091	0.028	0.018

[a] Calculated using Equations (4), (7), (24) and (27) with n = 1.0 and parameter values as Table 2.

TABLE 5.

Values of the sum of the leak-dependent and of the leak-independent components of ventilation for four types of storage (t_d = 5 mins, 500 - 250 Pa, full structure).

	$(k_{dep})_{max}$ (d^{-1})		$(k_{dep})_{max}$ with 60% CO_2 in enclosure (d^{-1})		$(k_{indep})_{max}$ (d^{-1})
	n = 0.8	n = 1.0	n = 0.8	n = 1.0	
Case 1, farm bin	0.05	0.03	0.06	0.04	0.08
Case 2, bag stack	0.07	0.04	0.08	0.05	0.05
Case 3, silo bin	0.14	0.11	0.21	0.15	0.03
Case 4, shed	0.12	0.09	0.17	0.11	0.10

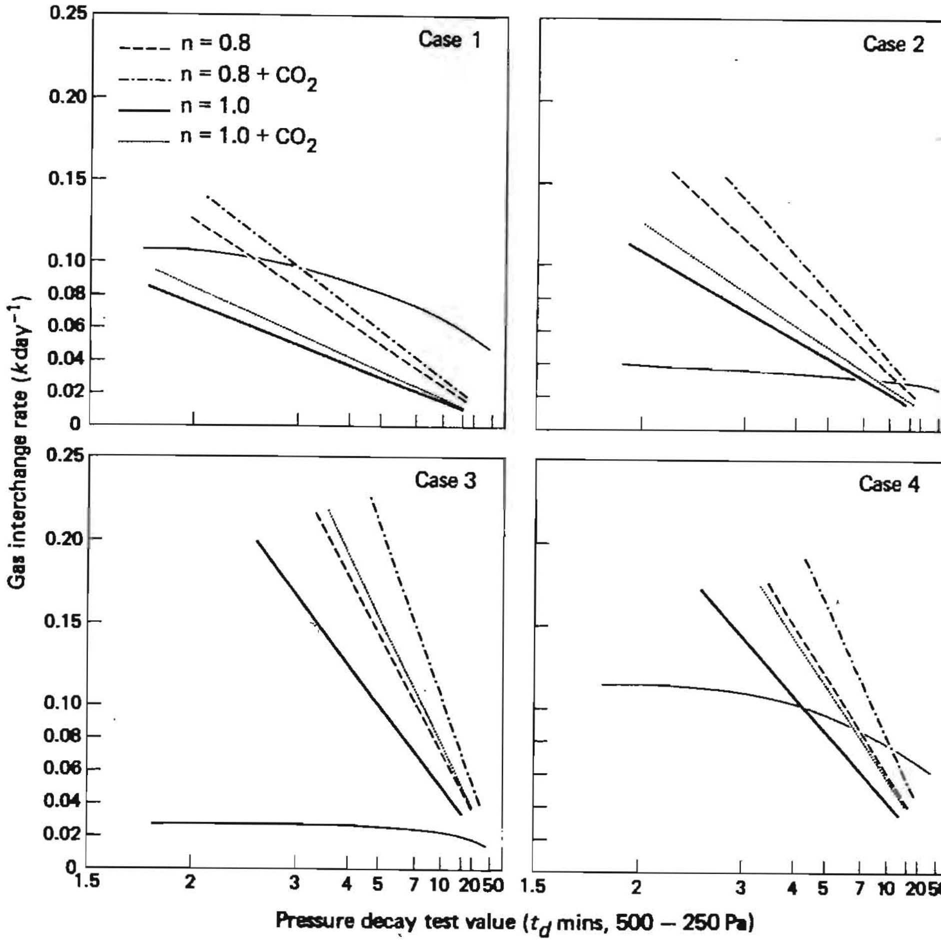

Fig. 2. Comparison of values of $(k_{dep})_{max}$ (straight lines) and $(k_{indep})_{max}$ (curved line) for four enclosures (see Table 3) showing the influence of level of sealing, as assessed by a pressure test, and the value of n and presence of 60% CO_2 in internal atmosphere.

that the 5 min decay standard is, in fact, adequate for large silo bins and that it is therefore too stringent for farm bins of the type considered here. On the basis of the model it is not possible to tell which of these two inferences is correct as the value of k_{dep} cannot be established sufficiently accurately.

(7) The specification of a particular decay time as a standard for gastightness without knowledge of the value of n, allows a substantial range of interchange rates, depending on n.

(8) The presence of 60% CO_2 substantially increases the value of $(k_{dep})_{max}$ but, since an additional 0.02 day^{-1} can be tolerated

in actual loss rate compared with that for nitrogen (Table 1), the gastightness requirements (pressure decay test time) are similar for the two techniques.

(9) While treatment failures may be caused by inadequate sealing, leading to excessive k_{dep} values, this need not always be so. Excessive values of k_{indep} can also occur.

(10) Variation in barometric pressure is unlikely to cause treatment failure except in combination with other phenomena.

(11) Short term temperature fluctuations can be a very significant cause of gas loss in some situations.

CONCLUSION

The model presented here is a useful tool for design work on storages and for investigation of some of the reasons for treatment failures and excessive gas usage in fumigations and controlled atmosphere use. It provides a framework for understanding the influence of a specific pressure decay test time on gas retention under particular circumstances. The model also exposes some of the ambiguities inherent in using simple pressure decay test times as a method of specification of gastightness.

It is hoped that this model and discussion has explained why a single pressure test standard to cover all storage structures is inappropriate. The pressure decay value, being in the order of minutes, should be set to meet the needs of particular storage types and environmental circumstances. With judgements of this kind applied well it should be possible to optimise the technology of fumigant and controlled atmosphere use. The incidence of treatment failures may then be reduced without resorting to unnecessarily high standards of sealing and unnecessary and often costly modifications to storages in misdirected attempts to reduce gas losses.

ACKNOWLEDGEMENTS

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NOTATION

A	Area (m^2)
C	Wind Pressure coefficient
D	Diffusion constant ($m^2\ s^{-1}$)
M	Molecular weight of air
N	Porosity of grain
P	Pressure (Pa, absolute)
Q	Volumetric flow rate ($m^3\ s^{-1}$)
R	Gas constant (J kg-mol^{-1} K^{-1})
T	Temperature (K)
V	Volume (m^3)
a	Amplitude of oscillation
b	Gas flow across a leak at 1 Pa ($m^3\ s^{-1}$)
g	Acceleration due to gravity (m s^{-2})
h	Vertical distance between leaks (m)
k	Rate constant or ventilation rate (s^{-1})
l	Length of diffusion path (m)
m	Mass of gas in enclosure (kg)
n	An empirical exponent
p	Pressure (Pa)
t	Time (s)
u	Wind velocity (m s^{-1})
x	Distance (m)
α	Proportion of total leak represented by the smaller leak
γ	Orifice coefficient
κ	Thermal diffusivity ($m^2\ s^{-1}$)
ν	Density (g m^{-3})
τ	Period of fluctuation (s)
ϕ	Phase angle
ω	Frequency of fluctuation (Hz)

Subscripts

C	Chimney effect
D	Diffusion
HS	Headspace
P	Barometric pressure
T	Temperature variation
Tb	Temperature variation in grain bulk
W	Wind
Wf	Wind fluctuations
d	Decay time
dep	Dependent on leak size
ext	External
$indep$	Independent of leak size
int	Internal
pe	Permeation
s	Surface

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APPENDIX. CALCULATION OF VOLUME OF GAS LOST THROUGH TEMPERATURE VARIATION IN A GRAIN BULK RESULTING FROM DIURNAL HEATING OF THE SURFACE.

Consider a semi-infinite grain mass, initially at uniform temperature, bounded by a wall at $x = 0$, where x is the distance into the mass from the wall. It can be shown (Babbitt, 1945) that, with a variation in wall temperature following

$$T_s - \overline{T} = a\cos(\omega t - \phi) \tag{38}$$

the temperature at any point in the bulk is given by

$$T - \overline{T} = a\,e^{-\sqrt{\frac{\omega}{2\kappa}}x}\cos(\omega t - \sqrt{\frac{\omega}{2\kappa}} - \phi) \tag{39}$$

The change in gas volume, dV, in an element dx, of cross-section, A, brought about by an increase in temperature from $\overline{T}$ to T is given by (derived from Equation (17))

$$\mathrm{d}V = \frac{T - \overline{T}}{\overline{T}}\mathrm{d}x\,NA \tag{40}$$

where N is the porosity of the grain bulk. Thus

$$\mathrm{d}V = \frac{aAN}{\overline{T}}\,e^{-\sqrt{\frac{\omega}{2\kappa}}x}\cos(\omega t - \sqrt{\frac{\omega}{2\kappa}} - \phi)\,\mathrm{d}x \tag{41}$$

The total volume excess, ΔV, over the bulk will be given by

$$\Delta V = \int_0^\infty \frac{aAN}{\overline{T}}\,e^{-\sqrt{\frac{\omega}{2\kappa}}x}\cos(\omega t - \sqrt{\frac{\omega}{2\kappa}} - \phi)\,\mathrm{d}x \tag{42}$$

$$= \frac{aAN}{\overline{T}}\sqrt{\frac{\kappa}{\omega}}\cos(\omega t - \frac{\pi}{4}) \tag{43}$$

During one complete cycle the total volume excess will be approximately twice the maximum value of V, since the loss occurs during expansion from $\overline{T}$ to T_{max} and from T_{min} to $\overline{T}$. Substituting for ΔV in Equation (7), the ventilation rate, k, from this process is thus given by

$$k = \frac{2aNA}{\overline{T}Vt}\sqrt{\frac{\kappa}{\omega}} \tag{21}$$

SESSION 6.

ALTERATION OF STORAGE ATMOSPHERES

Papers by:

H.J. Banks and P.C. Annis	-	Australia
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T.S. Krishnamurthy:		
K.S. Narasimhan:		
J.R. Rangaswamy: M. Jayaram		
and S.K. Majumder	-	India
J.H. Viljoen: J.J. Coetzer		
and C.J. Vermaak	-	South Africa
J.S. Cook	-	United States of America
F.B. Boland	-	Australia
B.E. Ripp	-	Australia
A.R. Green: S. Sheldon and		
H.J. Banks	-	Australia
E.J. Bond and T. Dumas	-	Canada

ON CRITERIA FOR SUCCESS OF PHOSPHINE FUMIGATIONS BASED ON OBSERVATION OF GAS DISTRIBUTION PATTERNS

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Abstract: Recent changes in the basis of practical use of phosphine have created a need to re-evaluate the distribution of gas produced during fumigation. This evaluation must be made against an awareness that survival of insects may increase the risk of selection of strains resistant to phosphine. Using examples from recent Australian full-scale field trials, illustrations are given of various defects in fumigant retention distribution and application that may lead to inefficient use of material and survival of insects. The ratio of minimum to maximum concentration is used as an indicator of distribution in unsealed systems with uniform admixture of phosphine releasing agents and sealed systems with surface application.

Based on the gas distribution patterns in the examples presented, a set of criteria are proposed by which the success of a treatment can be judged, and a commercially successful result and a completely successful one, i.e. one in which complete insect kill may be expected, can be differentiated. In increasing order of stringency the criteria are (a) the grain be found free of insects by inspection after treatment, (b) the average maximum concentration of phosphine be >50% of that expected theoretically, (c) the concentration at the end of the exposure period be greater than the minimum effective against insects and (d) that the ratio of minimum to maximum concentration exceed 0.25 after not more than 25% of the exposure period.

1. INTRODUCTION

Phosphine has been used routinely for grain fumigation in many parts of the world for more than 25 years. Until recently, it was general practice to add phosphine-generating material, typically Phostoxin tablets, in a way designed to provide as even a distribution of the formulation as possible throughout the grain bulk (Munro, 1969). The formulation was either placed in the grain stream during the loading of large silo bins or was distributed by probing into the grain bulk in both large flat storages and farm bins. Attempts were sometimes made to restrict gas loss by sheeting the surface of treated bulks and sealing access doors and other penetrations in the the storage fabric. Nevertheless, phosphine was regarded as a fumigant suitable for poorly sealed enclosures and high application rates, up to 10 g t^{-1}, were recommended in such cases in an attempt to compensate for losses through leakage from the system. Furthermore, some dosage schedules for stored grain recommended exposure periods of only 2 days (e.g. Anon., 1972).

This approach is now recognised to be unsound (Winks *et al.*, 1980). As a result in Australia, there have been important changes in the basis of the way in which phosphine fumigations are carried out. These changes have substantial practical consequences:

(i) Recommendations for phosphine use in large structures now state that the structure must be well sealed and, at a dosage of 2 g t^{-1}, set a minimum exposure period of 5 days (7 days if *Sitophilus* spp. are present) at > 25°C for a completely effective fumigation. In addition, it is now recognised that it is not possible to achieve a good fumigation in the face of a high gas loss rate from the system merely by inceasing the rate of phosphine applied (Winks *et al.*, 1980).

(ii) There is increasing use of surface application techniques. In these the phosphine-generating material is applied onto the grain surface or the headspace above the grain (e.g. on the conveyor catwalk).

(iii) There is also a tendency towards the use of reduced dosage rates (down to 0.5 g t^{-1}) made possible by low rates of gas loss from sealed systems. These reduced rates are usually combined with much increased exposure periods to the fumigant to take advantage of the increased sensitivity of the insects infesting the grain to phosphine under these conditions (Reynolds *et al.*, 1967; Heseltine, 1973).

The changes from use of poorly sealed to well sealed systems, from uniform admixture to surface application, bring with them substantial changes in the patterns of phosphine gas distribution occurring during treatments. Good distribution patterns are important to the success of phosphine fumigations. Accordingly, there is a need to re-evaluate the effectiveness of particular fumigation systems in the light of currently accepted requirements. Underexposure of a region to phosphine may result in survival of some insect pests, giving an unacceptable result in a particular fumigation. More importantly, if inadequate fumigations are carried out, strains of pests resistant to phosphine may be selected. This may lead, at first, to the need for increased exposure periods and dosage and finally to phosphine being rendered ineffective.

It is thus remarkable that despite the long history of use of uniform admixture and the importance of proper distribution of gas, there are still very few detailed data available on the distribution patterns occurring in large scale fumigations (but see Schuyler, 1963; Mori *et al.*, 1966; Conway and Mohiuddin, in press). There is also little information on the more recently developed surface application procedure (but see Snider and Allen, ca. 1977; Cook, 1980; Banks and Sticka, 1981; Winks, 1981; Zettler *et al.*, 1982).

This paper provides examples of the gas distribution patterns found in some large scale fumigations. It contrasts the results from uniform admixture in unsealed systems and surface application techniques in sealed storages, and illustrates particular defects in the two processes. These defects are discussed in terms of a broader study on particular modes of failure of phosphine fumigations brought about by defective gas distribution. A method of analysis of gas distribution data is presented that provides a means of determining particular defects in technique. Finally, a set of criteria is proposed which can be used to define the degree of success of a treatment, distinguishing between what is regarded as a commercially adequate result and a treatment in which no insects survive. Data is presented that can be used to justify why some current practices should be discouraged and others adopted, sometimes at substantial cost in modification of structures and changes in procedures.

2. BACKGROUND TO EXAMPLES

2.1 Choice of examples

With a dosage nominally capable of controlling insects, factors that may lead to fumigation failure are:

(i) Excessive overall loss of fumigant;

(ii) Inadequate fumigant dosage in localised regions;

(iii) Excessive delay between application and fumigant reaching some regions resulting in an inadequate exposure period.

All these factors may occur simultaneously.

Examples of each of these types of failure are evident in the data from uniform admixture of phosphine-generating preparations in unsealed, large, tall, narrow bins, and in the data from on surface application in sealed storages. These two combinations are taken as paradigms of two extreme forms of phosphine application: that where the initial distribution of gas generator is good but the sealing is poor and that where the initial distribution is poor but the sealing good.

Although there is a wide variety of other combinations of application process with enclosure size, degree of sealing and shape, no data for these various combinations is given here as the same types of failure will occur as in the examples given. The physical processes involved in distributing the gas are the same in both small and large storages and similar defects in distribution may be expected although they may vary in their magnitude with the size of system. If non-uniform application of the gas generator gives an adequate fumigation in a particular situation, it is assumed that uniform admixture also will do so in the same situation. With surface application and other non-uniform techniques, the main limiting factor is the rate of dispersion of the gas from the region where it is generated. The distance required for the gas to travel is much greater in these cases than with uniform admixture of formulation to the grain.

2.2 Data sources

Data for the examples given below are taken from various field trials carried out in Australia since 1973 by CSIRO Division of Entomology, usually in collaboration with a state grain handling organization. General details of the trials are given in Table 1, the trials being referred to by locality. All trials were carried out on wheat of < 12% moisture content at grain temperatures >20°C.

In each case, the fumigation was of a standard such that it gave what would have been regarded as a commercially successful result. Phosphine concentrations were measured with various indicator tubes (Drager, Auer, Kitagawa, Gastec). It should be noted that this method of phosphine analysis can be subject to substantial error (sometimes > ±30%, (Leesch, 1982)) if not corrected for the variation in sensitivity of the tube batches under the particular conditions of usage. Readings from later trials (Harden, Bordertown, Newcastle, Meandarra) were corrected for sensitivity and temperature, (Banks and Sticka, 1981), but those from other trials were uncorrected. A large number of sampling points was used in each trial (Table 1). They were distributed to give both a good estimate of the general distribution of the gas and to monitor critical areas, such as close to the floor or wall-to-roof joints. Average concentrations of a storage were calculated from the observations at particular points weighted by the gas volume of the region that

TABLE 1.

Details of field trials used to provide illustrative data.

Trial designation (Locality of trial)	Bordertown	Cunningar	Harden	Meandarra	Newcastle	Walleroo A	Walleroo B
Storage type	Steel bin	Concrete cell	Shed	Concrete cell	Steel bin	Concrete cell	Concrete cell
Storage dimensions	24 m high, 21.6 m diameter cylindrical, roof pitch	30.5 m high, 10.9 m diameter cylindrical, 28° roof pitch	12.9 m high, 121 m long, 30.8 m wide, 30° roof, 2.5 m wall	30.4 m high, 12.0 m diameter cylindrical, 28° roof pitch	9.4 m high 2.2 m diameter cylindrical, 26° roof pitch	32.1 m high, 10.7 m diameter cylindrical	32.1 m high 10.7 m diameter cylindrical
Degree of sealing	Sealed, pressure test (full, 500- 250 Pa) 480 secs	Roof vents open	Sealed, pressure test (full, 125 - 62.5 Pa) 300 secs	Sealed, pressure test (full, 1000 - 500 Pa) 660 secs	Sealed, pressure test (full, 500 - 250 Pa) 84 secs	Cell top open, aeration duct unsealed	Cell top open
Load (wheat, tonnes)	6800	2215	16470	2460	294	2140	2100
Phosphine application system	Surface application	Into grain on loading	Surface application in 'blankets'	Surface application	Surface application	Into grain on loading	Into grain on loading
Phosphine releasing preparation	Phostoxin pellets	Phostoxin tablets	Detia sachets	Phostoxin tablets	Phostoxin tablets	Phostoxin pellets	Phostoxin pellets
Total phosphine added (kg)	2.99	6.1	26.4	1.92	0.27	2.7	2.7
Number of sampling points used	46	28	51	28	44	60	48

they were sampling. The weighting factors were approximate only, but it was found that because of the large number of sampling points used, the average value is not very sensitive to the magnitude of the weighting factor used for individual points.

The ratios of the minimum to maximum concentration found in a system are a measure of evenness of phosphine distribution. This ratio shows when a significant concentration has been achieved at all points in a system and is thus an indication of the time at which the exposure period for the entire system can be taken to have started.

Phosphine concentration data are given as a percentage of that expected if all the phosphine potentially available was present in the gas space in the enclosure (i.e. without leakage or any sorption into the grain or on other materials therein). Since the concentration at a point at a given time under particular conditions will be approximately proportional to dosage applied, data presented in this way can be converted into concentration terms for any applied dosage. It is then possible to assess if a particular rate of application satisfies some set dosage parameter (e.g. a *Ct*-product value or a minimum effective concentration level). The actual values of these parameters are not discussed in detail here.

3. EXAMPLES

3.1 Uniform admixture in poorly sealed systems - deficiencies in fumigant retention and distribution.

(i) Excessive loss of fumigant. In the past, phosphine has been used in structures that were so poorly sealed that the maximum concentration of phosphine achieved was only a small fraction of that theoretically available from the applied formulation.

Figure 1 shows the average concentration in an open cell in which the aeration duct at the base was unsealed. The estimated theoretical concentration curve, calculated on the basis of total gas volume, is shown. In this case the fumigant was not used efficiently† and was lost very rapidly by leakage.

A similar situation (Fig. 2) was observed in a concrete cell which was not well sealed and in which the roof ventilators were kept open in order to vent dust-laden air displaced during the

† We consider 'efficient' use in this context to be that more than 50% of the theoretical phosphine concentration be observed at some time during the treatment.

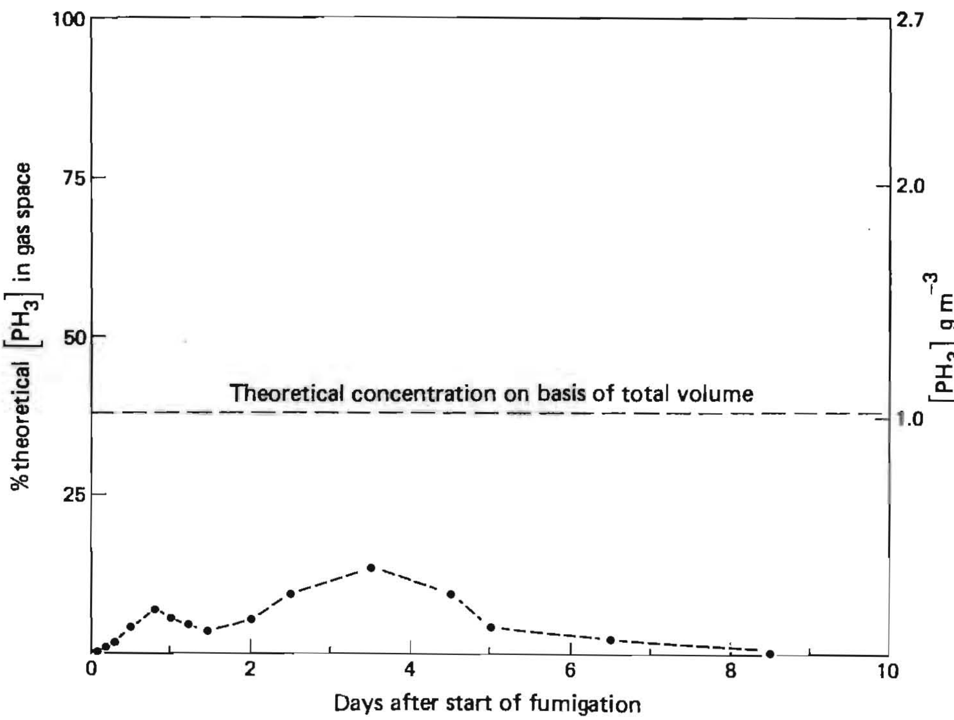

Fig. 1. Variation of average phosphine concentration with time in an open top concrete cell in which the aeration duct at the base had not been sealed off, showing inefficient use of fumigant (Walleroo A).

filling of the bin. Not only was there a loss rate resulting from the poor sealing of the system, but further substantial losses were caused by the protracted loading of the bin, which was carried out in four working periods spread over four days. The grain added during each successive period rapidly displaced the phosphine accumulated in the free space in the bin from the decomposition of phosphine-releasing material added on the previous days.

When fumigations are carried out in very leaky structures, as in these examples, the fumigant concentration may not be maintained at an adequate level until the nominal end of the exposure period and some survival of insects may be expected throughout much of the treated system.

(ii) Inadequate exposure in localised regions. Some fumigations are carried out under conditions where the gross leakage is slow enough to give an adequate average dose, but where some regions within the fumigation enclosure may receive insufficient dosage. In such cases, although the average concentration-time curve for the

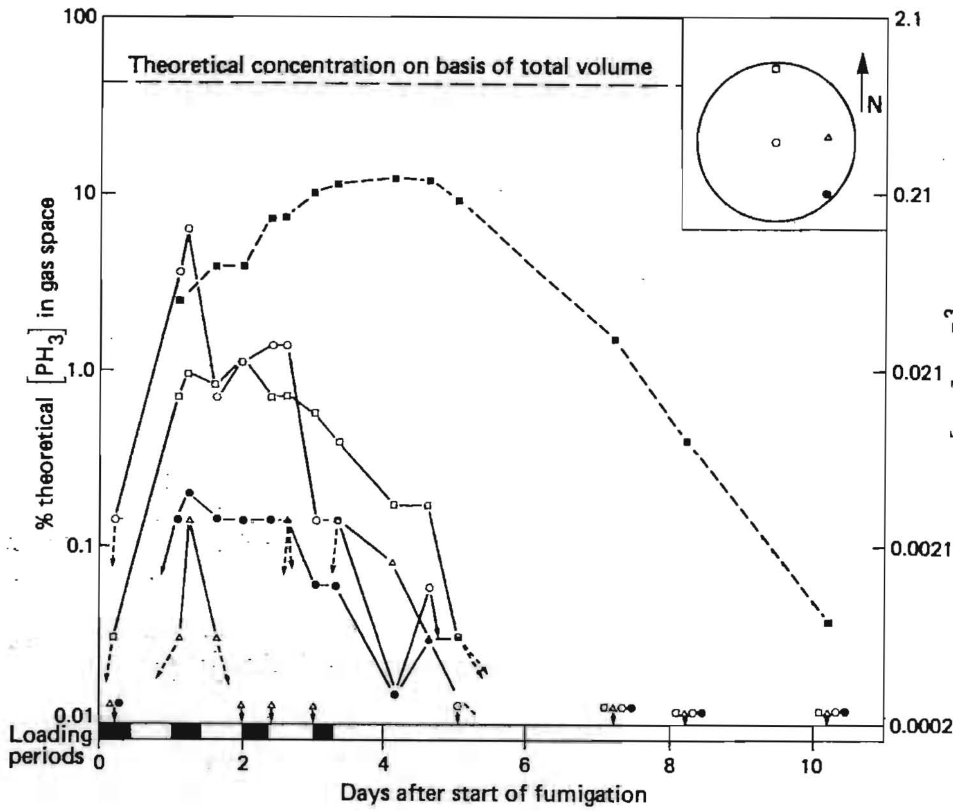

Fig. 2. Variation of phosphine concentration with time in a concrete cell loaded over four days and with roof ventilators open (Cunningar), showing a low efficiency of use of the fumigant overall (average line, ■-----■) and low exposure to phosphine at points at the base of the cell (inset shows plan of sampling position).

fumigation suggests that the treatment was successful, curves for specific regions may show this is not true. Figure 3 shows the concentrations of phosphine achieved in the treatment of an open topped concrete cell. Overall, the loss rate, though substantial (13% day^{-1}), was not sufficient to displace the fumigant within the recommended 7 day exposure period. However, points both close to the grain surface (Fig. 3) and also at the base of the bin (not shown) received very low dosages. On several other occasions (e.g. Fig. 2) we have observed a similar rapid loss of phosphine from regions close to the bin base during treatment of large, tall, unsealed, concrete cells (approx. 2000 tonnes capacity).

Regions that receive inadequate dosage regimes in such cases may be quite restricted, in contrast to the situation given in the previous example. Nevertheless they may be an important haven within which some insects survive and produce a general infestation

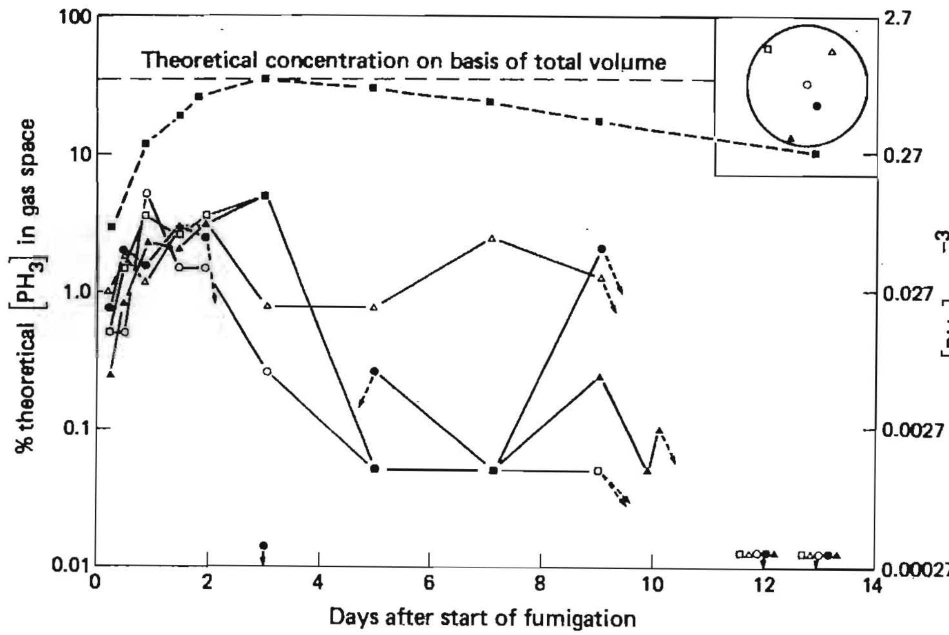

Fig. 3. Variation of phosphine concentration with time in an open-top concrete cell with a reasonable average concentration (■------■) but low exposure at points 15 cm below the grain surface (inset shows plan of sampling positions) (Walleroo B).

after further storage, and, thereby possibly contribute to selection for tolerance to phosphine.

3.2 <u>Surface application technique - deficiencies in fumigant distribution</u>

(i) <u>Excessive delay in reaching some regions with low efficiency of utilisation</u>. The surface application technique has a number of practical advantages that render it preferable to the systems involving addition of phosphine-generating preparations to grain during filling of cells or by probe. These include the ability to remove spent preparations, the ability to treat grain *in situ*, and the rapidity with which the fumigant application can be carried out. Indeed, in its simple form, it might rapidly replace other techniques were it not for some important limitations.

When fumigations are carried out with the fumigant preparation evenly mixed through the system, the gas needs to disperse only over relatively short distances to give significant concentrations throughout the system. In the absence of defects of the type cited above, the speed with which this occurs is rapid and the effective start of the exposure period is close to the time the preparation is added. In contrast, when the preparation is concentrated on the grain surface, the gas may need to disperse over substantial distances (up to 35 m downwards in some silo bins) to reach all parts of the bin. With natural mixing only, this may be slow. The effective start of the exposure period, i.e., the time at which all points in the system reach a significant fumigant concentration, may thus be many days after application. Furthermore, in tall, narrow structures, such as many concrete cells, the rate of dispersion of the gas may be so slow that a significant proportion of the gas applied is lost through leakage or sorption before concentrations begin to build up in the more remote parts of the system.

Data illustrating these problems is presented in Fig. 4a, which shows the average concentration-time curve and the change in minimum to maximum ratio with time during the fumigation of a large concrete cell at Meanderra. Here the gas reached all parts of the system only after about 14 days from application and after about 70% of the material applied had been lost by leakage and sorption.

Others (R. Sticka and B.E. Ripp, pers. comm.) have noted similar behaviour of the gas during fumigation of tall silo bins. They also have found that if the bin is not well sealed, the gas can be completely lost before it has time to disperse adequately within the system.

3.3 Examples of successful treatments using surface application technique in sealed systems

In the example shown in Fig. 4a, although the overall gas retention was adequate and there was no observed formation of havens for survival of insects through localised air ingress, the treatment was not completely satisfactory. The rate of the dispersion of the phosphine from the point of release was so slow that it could not provide a significant concentration throughout the structure before most of the gas added had been lost. However, dispersion is affected significantly by the geometry and size of the system treated and in many situations the surface application technique gives an excellent fumigation in a sealed system.

Figures 4b, c, d, show further examples of the average concentration-time curves produced from surface application of phosphine-generating material in sealed systems. These data are for a large shed (Harden) and small (Newcastle) and large cylindrical steel bins (Bordertown). The observed maximum concentration for each structure was > 55% of that expected and the loss rate was sufficiently low (< 12% day^{-1}) for there to be a substantial fraction of the original

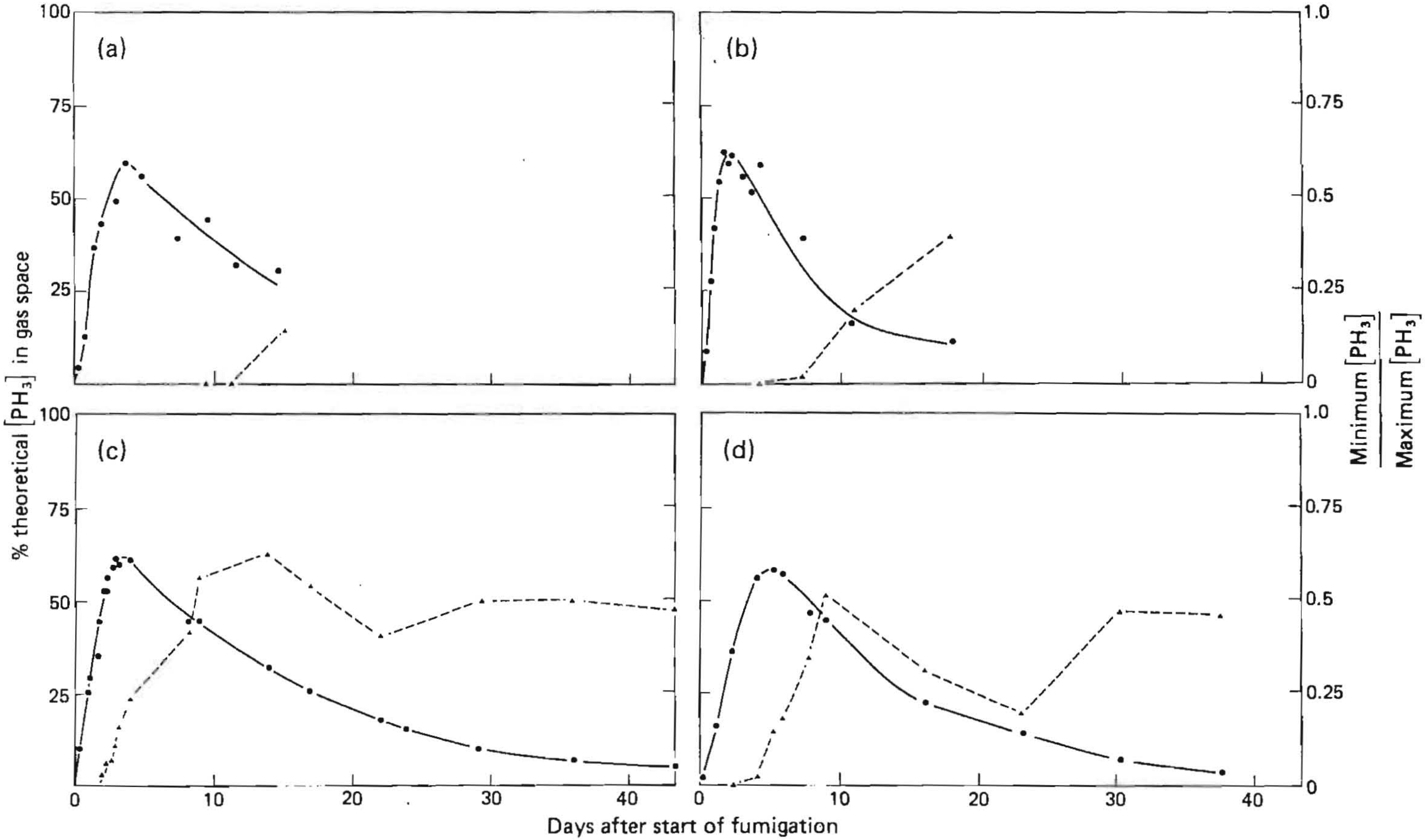

Fig. 4. Variation in average concentration of phosphine (●-----●) and minimum to maximum concentration ratio (▲-----▲) with time in sealed structures after surface application of phosphine generating material for (a) a large concrete bin (Meandarra), (b) a large steel bin (Bordertown), (c) a small steel bin (Newcastle) and (d) a large shed (Harden), illustrating variation in rate of dispersion of phosphine in different types of storage.

dosage present after more than 20 days from addition of the phosphine-generating preparation. These data can thus be taken as examples of where the material applied was used efficiently and where sealing of the fumigated system results in good retention of the fumigant.

The ratio of minimum to maximum concentration eventually exceeded 0.3 (Fig. 4) thus confirming that there was effective natural mixing in the system and that there were no havens observed where survival could be expected. In one case (Fig. 4a), data was not collected for long after phosphine had dispersed throughout the storage but it can be expected that the forces that were sufficient to even out an initially very uneven concentration pattern would have continued to operate adequately to maintain the approximately uniform pattern observed in examples in Figs 4b, c, d.

4. PHOSPHINE DISTRIBUTION CRITERIA FOR A SUCCESSFUL FUMIGATION

The data used in the examples was drawn from various large scale treatments, each of which was effective enough for it to be regarded as commercially successful. Yet, as we have seen, many of the treatments did not use the applied fumigant efficiently and, in several cases, there were regions of the treated system that did not receive an adequate dosage regime for complete kill. In others, the phosphine concentration built up in some regions only long after application of the fumigant material. These deficiencies can be incorporated in a set of criteria classifying the success level of a treatment and ranging from what is regarded as a 'commercial success' to a practical definition of a perfect result.

In the case of a 'commercial success', insect numbers must be reduced to a level where they are not detected by the subsequent handler or purchaser of the grain or where an acceptable period of storage is obtained before retreatment is required. In the perfect result there is no survival of pest insects, adult or immature forms, in the system after treatment and thus no chance of selection of strains resistant to phosphine.

We propose the following criteria for success of a phosphine fumigation, in increasing order of stringency. Each successive criterion includes, by implication, the previous ones. The trend is towards maximum likelihood of a perfect fumigation with applied dosage of fumigant.

(a) <u>The grain bulk be found free of insects by conventional sampling at the end of the treatment period.</u> This is one current definition of a commercially successful treatment, but is dependent on the intensity and method of sampling and may not detect either survival of immature stages or low numbers of adult insects.

(b) The average maximum concentration of phosphine in the system shall be not less than 50% of the expected quantity based on the dosage applied and the total gas volume in the fumigated system. This criterion implies that the gas is used efficiently and, indirectly, requires that the system is sealed sufficiently for the gas loss rate not to substantially affect the peak concentration.

(c) That an average concentration greater than the minimum effective against insects be present at the end of the exposure period. This criterion is an indirect constraint on the rate of loss of gas. It ensures that the period thought to be the exposure period is, in fact, so. Otherwise, under conditions of excessive leakage, the fumigant may have been lost before the end of the required exposure period. The minimum effective level has not yet been adequately defined but is here taken to be 0.01 g m^{-3} following Reynolds *et al.*, (1967) and Bell (1979).

(d) That the ratio of minimum to maximum concentrations of phosphine be not less than 0.25 after not more than 25% of the total exposure period and remain greater than that value for the remainder of the exposure period. This criterion is a measure of the evenness of distribution and how rapidly dispersion occurs. We have chosen the value of 0.25 for the ratio of minimum to maximum as a realistic value consistent with the need to define an approximately even distribution. It implies that there are no regions either of excessively high concentration, showing inefficient use of the material added, nor ones with very low gas concentrations where insects may survive. The restriction on the fraction of the exposure period is to ensure that the distribution process is not so slow that when an even distribution is achieved the phosphine concentration has not already decayed to a small fraction of that applied.

On the basis of these criteria only two of the examples given here, Harden and Newcastle, may be judged to be completely successful fumigations. Two others, Meandarra and Bordertown, fulfil all criteria proposed except (d). The slow rate of mixing in tall narrow cells is a known problem (e.g. see Conway and Mohiuddin, in press) which restricts the use of surface application in these structures. Assisted natural convection using an external circulation assisted by the sun and very gentle forced convection have both been used successfully to overcome this problem (see Boland, in press; Cook, 1980).

5. CONCLUSIONS

The criteria given above can be used to judge the level of success of a fumigation. To provide as perfect a result as possible all four criteria for success should be met. As some of the examples show, it is possible to meet this standard even when using an application technique in which the fumigant material is initially applied in one restricted region. Clearly, some fumigations are currently carried out in situations that can give an incompletely effective though commercialy acceptable result. In such cases, a decision must be made either to continue such practices knowingly for short-term economic benefit at the risk of provoking the development of resistance to phosphine or to bear the cost of altering the techniques to produce a better fumigation and hence minimise the risks.

6. ACKNOWLEDGEMENTS

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PRACTICAL APPROACHES TO PURGING GRAIN STORAGES WITH CARBON DIOXIDE IN AUSTRALIA

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ABSTRACT

Carbon dioxide used to produce modified atmospheres containing greater than or equal to 70% CO_2 for disinfesting stored grain in Australia is manufactured and delivered to country storage terminals as a pressurised bulk liquid by road tankers for distances of up to several hundred km. On-site purging costs and delivery costs can constitute a substantial part of the total CO_2 purging cost. For instance, when purging fourteen 2000 tonne capacity vertical steel bins the purging cost can rise by about 8.5 cents/t of wheat if the purge rate is reduced from 3 to 1 t CO_2/h due to higher on-site costs. Similarly it will rise by about 15 cents/t of wheat when purging a 28,000 t capacity horizontal bin due to larger amounts of CO_2 required for this type storage.

The additional tanker delivery cost, excluding on-site tanker charges, can add about 8.5 cents/t of wheat to the cost of purging the vertical bin facility when it is 100 km away while the difference for the horizontal bin is about 13 cents/t of wheat. On-site costs can be reduced appreciably by the selection of adequately sized equipment for the site to allow high CO_2 purge rates. Details are given of a low pressure distribution circuit and bin piping arrangement for a 28,000 t multi-bin terminal which will allow several bins to be purged simultaneously at a total CO_2 rate in excess of 3t/h. The liquid CO_2 can be conveniently vapourised at site and the gas heated to wheat temperatures with a transportable 3t/h nominal capacity forced draught vapouriser with electric superheater. Total power consumption for an output of 3.5t/h is 47 kW compared to about 340 kW for all-electric vapourisation. Air can be drawn in and mixed with the CO_2 stream to form mixtures with an air content ranging from zero to greater than 30%.

INTRODUCTION

Full scale trials involving the use of modified atmospheres containing CO_2 to disinfest stored grain in Australia commenced with tests in a 7000-t vertical steel bin at Bodertown, South Australia, in 1977. This research was followed with other trials involving vertical bins of 1500 to 3000-t capacity in Victoria and Queensland and in horizontal shed-type bins of 16,400 t and 25,000 t capacity in New South Wales and Western Australia, respectively. Carbon dioxide has been used commercially on a small scale for grain disinfestation during the past few years in Queensland and Victoria (Banks and Annis, 1980).

A substantial part of the cost of purging grain storages which are remote from the CO_2 production plants is the cost of the liquid CO_2 delivery and the on-site purging cost. These costs can be reduced by using a suitable high capacity CO_2 vapouriser and adequately sized bin CO_2 injection and venting arrangements and, for sites containing clusters of smaller bins, by connecting these to a properly sized CO_2 distribution network to allow a number of bins to be purged simultaneously. The possibility that small quantities of other fumigants may be required to be added to a CO_2 or CO_2-air mixture before it enters a bin should be considered when designing CO_2 purging systems (Williams, 1983).

Bulk liquid CO_2 is transported to the wheat storage sites in conventionally insulated road tankers generally having a net capacity of 20 tonnes. The total road weight of the tank and the prime mover is around 38 t which is the maximum allowed by most State authorities. These tankers regularly move 20 t lots of CO_2 across and around the Australian continent for distances of up to 3000 km. The total cost of a complete rig is in the vicinity of $220,000 and thus the cost of delivery including the on-site tanker costs can represent up to 50% of the total purging cost.

The exposure periods and CO_2 concentration levels in air for modified atmospheres currently being used in Australia to disinfest stored grain and which are believed to give complete insect mortality are as follows: 10 days or longer (in practice 14 days) with initial CO_2 levels greater than or equal to 70% and final levels greater than 35% for grain with a moisture content of less than 12% and at a temperature of greater than or equal to 20°C (Banks, 1979). Since the treatment is generally carried out during or immediately following the harvest period in summer or early autumn most grain temperatures in sealed non-aerated storages are in excess of this temperature.

Before purging with CO_2 it is essential that wheat bins and any interconnecting ductwork and fans be sealed to an adequate standard of gastightness to ensure that the CO_2 concentration does not fall below 35% within the 14 day treatment period (Banks and Annis, 1977). For structures of 300–10,000-t capacity a minimum decay time, depending on size, of 5 minutes for an applied pressure drop of either 2500 to 1500 Pa, 1500 to 750 Pa or 500 to 250 Pa in a full storage is considered a satisfactory gastight standard (Banks and Annis, 1980). The CO_2 is applied in a one-shot operation without any subsequent make-up and the atmosphere within the bin allowed to degrade (due to leaks) during the 14 day treatment period. In order to maintain reasonably uniform concentrations throughout the bin and also to ensure that the CO_2 concentration near the apex is maintained at the desired level a small stream of CO_2 rich gas is recycled from the base of the bin to the apex (Wilson et al., 1980).

It has been found during full scale trials and commercial purging operations in Australia that the above criteria are met when using 1 t

CO_2/1000 t of wheat in vertical steel bins with 5% headspace and when using 2 t CO_2/1000 t of wheat in horizontal shed-type bins with 40 to 45% headspace (Banks et al, 1980). The density of CO_2 gas is about 50% greater than that of air at ambient temperatures and when it is injected into the base of a vertical bin it floods rapidly across the bin floor and then rises through the wheat mass, displacing the air present with a minumum of mixing. A single injection point is thought to be satisfactory for cylindrical vertical bins of up to 10,000 to capacity. In large horizontal bins the CO_2 can be readily distributed throughout the bin via the existing below floor aeration ducts or through temporary perforated aeration ducting installed on the floor.

The application of CO_2 to a wheat storage bin involves some type of structural addition and/or modifications to the bin either in a minor or of a more substantial nature to allow the gas to be injected and distributed within the bin and to allow the displaced air and exhaust CO_2 to escape from the bin without over pressurising and damaging the bin fabric. These changes or additions are generally made with medium to long term use in mind. The system finally chosen should provide the optimum CO_2 treatment of the grain at acceptable cost/t and, depending on cost constraints, be suitable for handling other gases or gas mixtures such as CO_2 -air, CO_2 -air- methyl bromide, CO_2-air-phosphine, etc., which may possible by required in the future. Consultation between grain handling authorities, C.S.I.R.O., relevant engineering groups and industrial gas companies prior to the design and construction phases of new installations, or the modification phases of existing installations, should prove beneficial in this regard.

CARBON DIOXIDE PRODUCTION AND DISTRIBUTION

Most of the CO_2 production plants in Australia, with the exception of brewery CO_2 plants, which supply CO_2 to the various industry markets, are located in cities and towns along the Eastern, Southern and South-Western seaboard. Therefore there is good access by road and rail to the grain storage sites of the wheat growing areas of Australia. Production plants are located at Townsville and Brisbane, Queensland; Newcastle and Sydney, New South Wales; Melbourne, Victoria; Launcestion, Tasmania; Mt. Gambier and Adelaide, South Australia and Perth, Western Australia. The combined installed production capacity of these plants is c.a. 500-600 t/day.

Sources of raw CO_2 include flue gas from oil burning and from cement works, byproduct CO_2 from ethanol fermentation plants, synthetic ammonia plants and other chemical processes and CO_2 from a natural well at Mt. Gambier. Carbon dioxide is produced and marketed in three forms: bulk liquid, high pressure cylinder liquid and dry ice (solid CO_2). Because most of the CO_2 produced by these plants is used by the food and beverage industries it is manufactured to a very high degree of purity. Bulk liquid would generally contain about 99.9% CO_2, be odour free and have a moisture content on delivery of well below 50 ppm. Bulk liquid used for the purging of wheat storages is produced, stored and delivered at a pressure of 2067 kPa and a temperature of -16.7°C. Factory storage facilities for bulk CO_2 vary from around 100 t to over 1000 t per site.

MULTI-BIN PURGING

The pressure of the CO_2 stream reaching the bin wall when purging a wheat storage facility consisting of, for example, fourteen 2000 t vertical steel bins may be chosen from near tanker pressure of 2067 kPa to near atmospheric pressure. The following describes a low pressure (50 kPa) multi-bin CO_2 circuit suitable for purging three bins simultaneously with an 80% CO_2-20% air mixture at purge rates of up to 1.25 t CO_2/h/bin. One advantage of this low pressure CO_2 circuit is that, when required, it allows the initial pressurised CO_2 stream to be used with a suitable injector to draw in and mix air with the CO^2 thus avoiding the need for a multi-stage blower or screw or piston compressor and the associated high power requirements. The purging circuit should be of simple design, leakproof, reliable, safe, require minimum on-site labour and have all valves, hoses and other equipment within easy access of the operator at ground level.

The individual bin modifications required to allow a purge rate of 1.25 t CO_2/h as an 80% CO_2-20% air mixture may be similar to those shown in Fig. 1. This shows a 75 mm dia. CO_2 injection tube covered by an internal 600 mm x 450 mm grain baffle which is used to ensure that the back-pressure at the grain face is less than 10 kPa under most operating conditions encountered. These baffles are essential to minimise back pressure at this point. During tests, pressures as high as 400 to 500 kPa were recorded with a number of bins not fitted with grain baffles. A 200 mm dia. vent gas tube is connected to the bin apex adjacent to the infeed chute and is carried down the outside of the bin to a 75 mm water seal located near ground level. The seal provides the bin with positive protection against over or under pressurisation. The maximum bin pessure during the purge phase is less than 50 mm water gauge. During the purge the injection tube is connected to the CO_2 distribution circuit (Figs. 2 and 3) with a 75 mm dia. flexible hose. The same hose is later used to connect the injection tube to a 3 m^3/min blower for recycling a stream of gas from the base of the bin to its apex during the subsequent treatment period.

Figs. 2 and 3 show a low pressure (50 kPa) CO_2 circuit suitable for purging fourteen 2000 t vertical steel bins at rates of up to 3.5 t CO_2/h, with three bins being purged simultaneously. A flow-restricting orifice in the line to each bin which provides a substantial part of the pressure drop in the CO_2 distribution circuit plus the very low (near atmospheric) back-pressures at the wheat face under each grain baffle ensure reasonably uniform purge rates for each set of three bins which are being purged. A sudden increase in line pressure and thus flow to one bin which may result if the CO_2 to the other two bins is shut off prematurely, before opening new bins, causes a rapid rise in the pressure drop across the orifice plate and in turn a rapid pressure rise at the vapouriser end. This opens a low pressure line dump (relief) valve to help protect the bin structure from over-pressurisation. The piping at each bin is arranged so that once purged the bin is isolated from the CO_2 distribution circuit as well as from all the other bins. During the purge phase the CO_2 content of the vent gas stream may be conveniently measured with a thermal conductivity type CO_2 meter (Gow-Mac Inc. Bound Brook, N.J., USA).

CARBON DIOXIDE VAPOURISER

It is desirable to vapourise the bulk liquid CO_2 at site and heat the resulting gas so that it enters the storage bin at near grain temperature to avoid any localised cooling of the grain. The CO_2 may be vapourised under pressure in a suitably designed receptacle using a heating medium such as electric power, steam, diesel fuel, propane, forced draught or natural draught. Bulk liquid CO_2 requires 350 kj/kg to convert it to gas at atmospheric pressure and at a temperature of 25°C.

The vapouriser unit should be robust, transportable, reliable, safe to operate, of relatively simple construction and of a type that can be easily maintained and readily repaired by local technicians in remote country towns. These requirements can be met by using a forced draught type vapouriser with electric superheating. A flow diagram of a unit with a nominal vapourising capacity of 3 t CO_2/h at an ambient temperature of 27°C is given in Fig. 4. This figure shows that at a rate of 3.5 t CO_2/h the forced draught vapourising element provides in excess of 90% of the total heat input using a fan power consumption of 15 kW compared to less than 10% of the heat input for a power consumption of 32 kW by the electric superheater elements. The air content of the exit CO_2 stream may be varied from zero to over 30% with an injector element. Both 100% CO_2 and 80% CO_2-air mixtures have been used successfully to purge wheat bins on a commerical basis in Australia. Fig. 5 shows the variation in the average vapouriser output for ambient temperatures varying from 0 to 40°C. Vapourising rates in the range of 2.5 to 3.5 t CO_2/h generally apply at the temperatures prevailing in the wheat areas of Australia during summer and early autumn. Optimum performance is obtained when the forced draught element is defrosted after each hour of operation for a period of 10 to 15 min by simply shutting off the CO_2 flow at the exit. Defrosting requires ambient temperatures in excess of $0^{o}C$. Figs. 10 to 13 show details of the vapouriser and its use in a number of purging operations including 16,400 t and 25,000 t horizontal shed-type bins.

The purging of very large storages of over 100,000 t capacity requires special consideration with respect to purging rate and CO_2 supplies. Unless the purge rate is sufficiently high, the CO_2 consumption can become excessive due to leakage of CO_2 from the bin to the atmosphere. A situation could occur where the CO_2 leakage rate just balances the CO_2 injection rate into the bin and a lethal concentration would be unobtainable.

SAFETY

Carbon dioxide gas is colourless, odourless and often difficult to detect by people particularly at low concentrations. The T.L.V., threshold limit value, for CO_2 in air is 5000 ppm which can be compared to the T.L.V. for methyl bromide of 5 ppm and for phosphine of 0.3 ppm. Whilst CO_2 is not necessarily dangerous at concentrations of a few per cent in air it will produce symptoms of deeper and faster breathing, nausea and dizziness when a person is exposed to concentrations of up to 9% for several minutes. Recovery from these symptoms is usually fairly rapid when the person is placed in a source of fresh air. Unconsciousness can occur in 5 to 10 min. when breathing air with a CO_2 content greater than 9%. With CO_2 concentration above 20% in air, death is likely in 20 to 30 min. unless the person is moved to fresh air sooner, artificial respiration is applied and medical attention is sought.

Since CO_2 gas is c.a. 50% heavier than air at ambient temperatures it will generally attain its highest concentration at the lowest parts of the workplace such as elevator pits, stairwells, drains, tunnels, etc. Adequate ventilation should be provided either by natural or mechanical means to ensure all work areas contain less CO_2 than the T.L.V. value before operators are allowed to enter. The CO_2 in many vertical bins may be exhausted to atmosphere with the recycle fan after opening the vent tube to allow air to enter at the bin apex. With some types of horizontal storages there will be a need to fit suitably sized exhaust fans to remove the CO_2 from the storage area prior to entry. Carbon dioxide concentrations in air may be conveniently measured with a Drager gas detection kit or other equipment (Dragerwerk A.G., Lubeck, Germany).

COSTS

The total cost for purging a grain bin with CO_2 excluding bin modification and sealing costs, may be broken up into three parts; CO_2 cost ex works, CO_2 delivery costs and the on-site purging costs. There can be differences in the CO_2 cost ex works from one centre to another due to various factors, one large one being the highly competitive nature of the gas industry. Delivery costs based on the use of 20 t capacity tankers can also vary between locations although they are usually very competitive. Both CO_2 ex works cost and delivery cost provide areas for reducing overall purging costs in a significant way but this depends on factors such as production and distribution costs, competition, marketing strategies, contractual arrangements and other factors applicable to a particular treatment.

The choice of equipment at site does not, apart from the on-site tanker costs, have a great bearing on the above costs. However, the on-site purging costs which are directly related to the purging time and which can amount to a fair proportion of the total cost can be minimised with the correct selection of equipment at the site. Particularly important is the CO_2 distribution, injection and vent gas piping necessary to allow bins to be purged at high CO_2 rates with maximum efficiency. On-site purging costs include charges for tanker waiting time, driver and operator expenses and wages, vapouriser hire and towing charges and power costs.

The following costs have been based on the premise that the bins at a terminal have been sealed and made suitably gastight by grain handling authority personnel and that the complete CO_2 purging operation is carried out by gas company operators. Some reduction in these costs could result if the purging operation was carried out by on-site grain handling people or by private contractors.

Fig. 6 shows the effect on the total CO_2 cost/t of wheat treated when the CO_2 purge rate is varied from 1 to 3 t/h for a terminal containing 14 x 2000 t capacity vertical steel bins filled to 95% volume when the terminal is located 100 km from the CO_2 plant. The comparison is also made for the terminal which is located 600 km from the CO_2 plant. The carbon dioxide cost ex works is taken at \$100; \$150 and \$200/t to allow for the variation in the ex works cost of CO_2 from one production plant to another. For example when the ex works cost of CO_2 is \$100/t and the wheat terminal is 100 km from the CO_2 plant the total CO_2 cost/t of wheat is 26.5, 20.0 and 18.1 cents for CO_2 purge rates of 1, 2 and 3 t/h, respectively. Similarly when the terminal is 600 km from the CO_2 plant these costs rise to 35.1, 28.4 and 26.7 cents, respectively.

Fig 7 shows a similar cost comparison for a 28,000 t horizontal bin filled to 60% volume. Here 56 t of CO_2 are necessary for an adequate purge of the bin compared to 28 t for the terminal described in Fig. 6. When the ex works cost of CO_2 is \$100/t and the wheat terminal is 100 km from the CO_2 plant the total CO_2 cost/t wheat treated is 48.6, 38.8 and 33.8 cents for CO_2 purge rates of 1, 2 and 3 t/h, respectively. If the terminal is 600 km from the CO_2 plant these costs rise to 61.5, 51.7 and 46.6 cents, respectively.

The effect on the total CO_2 cost/t of wheat treated due to varying the distance of the wheat terminal from the CO_2 plant when purging at a constant CO_2 rate of 3 t/h is shown in Figs. 8 and 9. Fig. 8 shows these costs for a terminal containing 14 x 2000 t capacity vertical steel bins filled to 95% volume for CO_2 ex works costs of \$100, \$125, \$150, \$175 and \$200/t. As an example when the CO_2 ex works cost is \$200/t the total CO_2 cost/t of wheat is 28.1, 31.6 and 36.7 cents for distances of 100, 300 and 600 km. respectively. Fig. 9 shows the same cost comparison for a 28,000 t horizontal bin filled to 60% volume. For a \$200/t ex works CO_2 cost the total CO_2 cost/t of wheat is 54.9, 60.1 and 67.8 cents for distances of 100, 300 and 600 km. respectively.

CONCLUSION

Carbon dioxide has been used successfully in Australia over a period of several years in trials and on a commercial basis to disinfest wheat which has been stored in bins that have been sealed to a suitable gastightness. It can be handled efficiently and economically by transporting it to site as a pressurised liquid in 20 t road tankers and vapourising it at low cost at rates in excess of 3 t/h with forced draught vapourisers. It is relatively safe to use and far less toxic to humans than phosphine and methyl bromide. The method does provide wheat which is free of insects and chemical residues at treatment costs which are very competitive with some currently used contact type insecticides. There is a strong and growing interest in the use of CO_2 for disinfesting stored grain by producers and grain handling and marketing authorities within Australia for the supply residue free grain to local and overseas markets as the demand for this type of grain increases.

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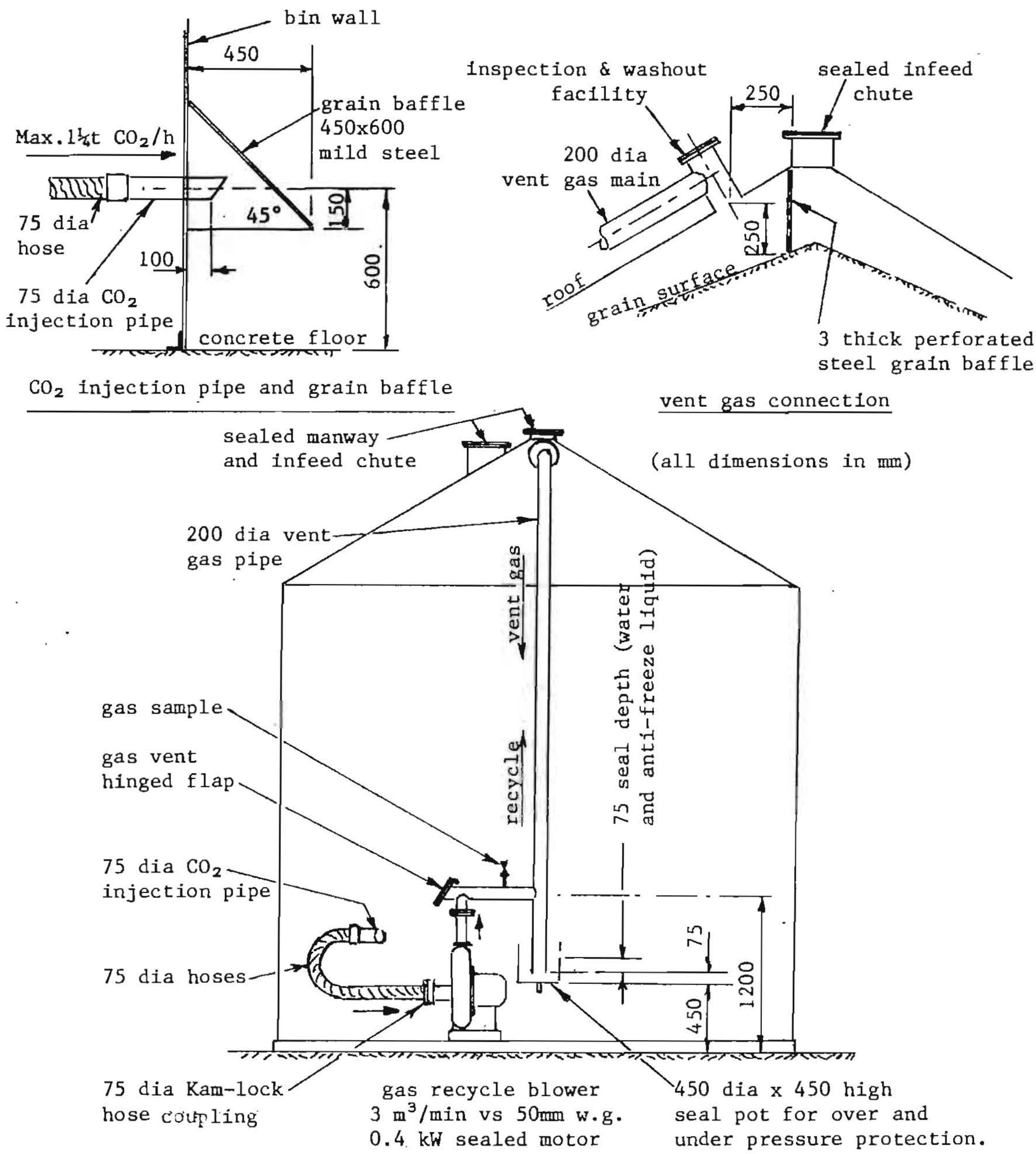

FIG. 1 Details of CO_2 piping and recycle gas fan for purging 2000 t capacity vertical steel wheat bin at rates of up to 1.25 t CO_2/h.

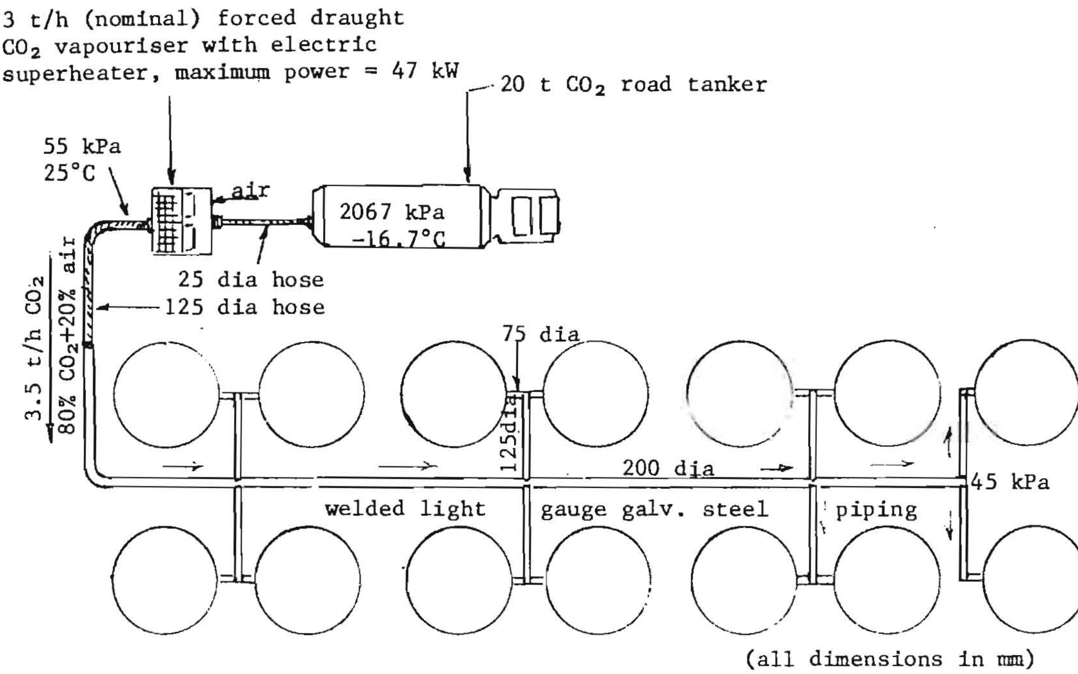

FIG. 2 Arrangement of low pressure gas circuit for purging fourteen 2000 t vertical steel wheat bins with an 80% CO_2-20% air mixture at a maximum rate of up to 3.5 t CO_2/h.

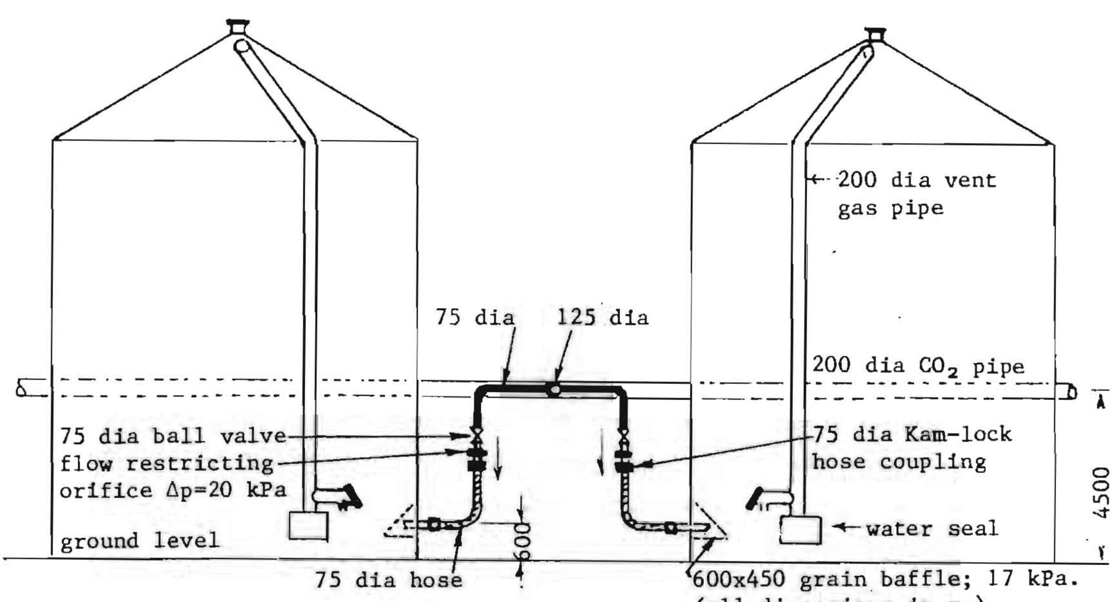

FIG. 3 Details of piping connections for purging 2000 t vertical steel wheat bins with an 80% CO_2 - 20% air mixture at up to 1.25 t CO_2/h each

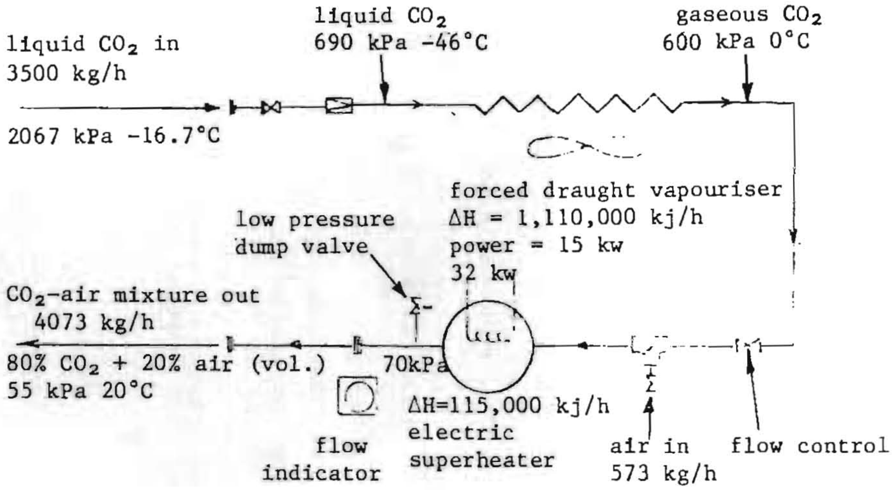

FIG. 4. Flow diagram of a nominal 3 t/h forced draught CO_2 vapouriser with electric superheater.

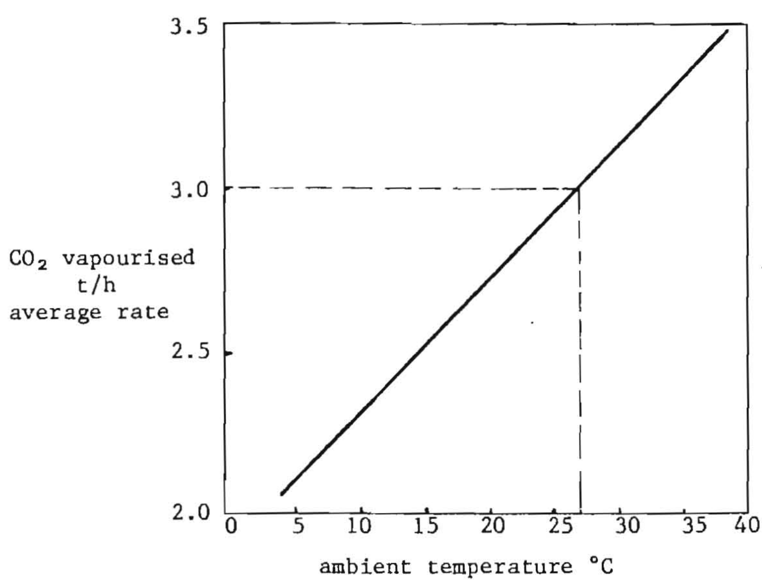

FIG. 5. CO_2 vapourising capacity of nominal 3 t/h vapouriser at various ambient temperatures. Total cycle = 75 min (60 min operating + 15 min defrost).

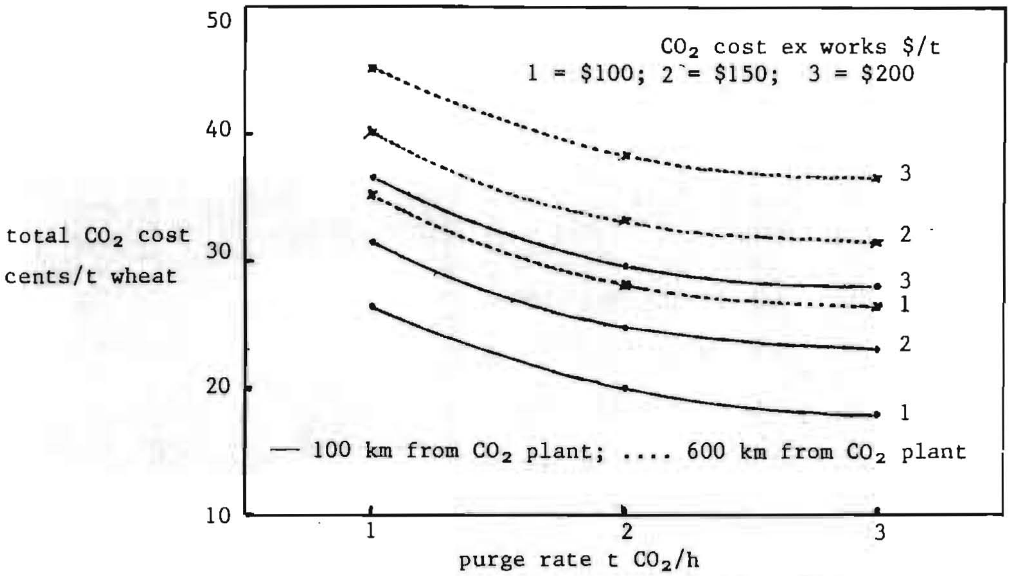

FIG. 6. Effect of purge rate on the total CO_2 cost when purging a 28000 tonne vertical, steel, multi-bin wheat terminal. Bins 95% full 5% headspace. CO_2 consumption 1 t/100 t wheat.

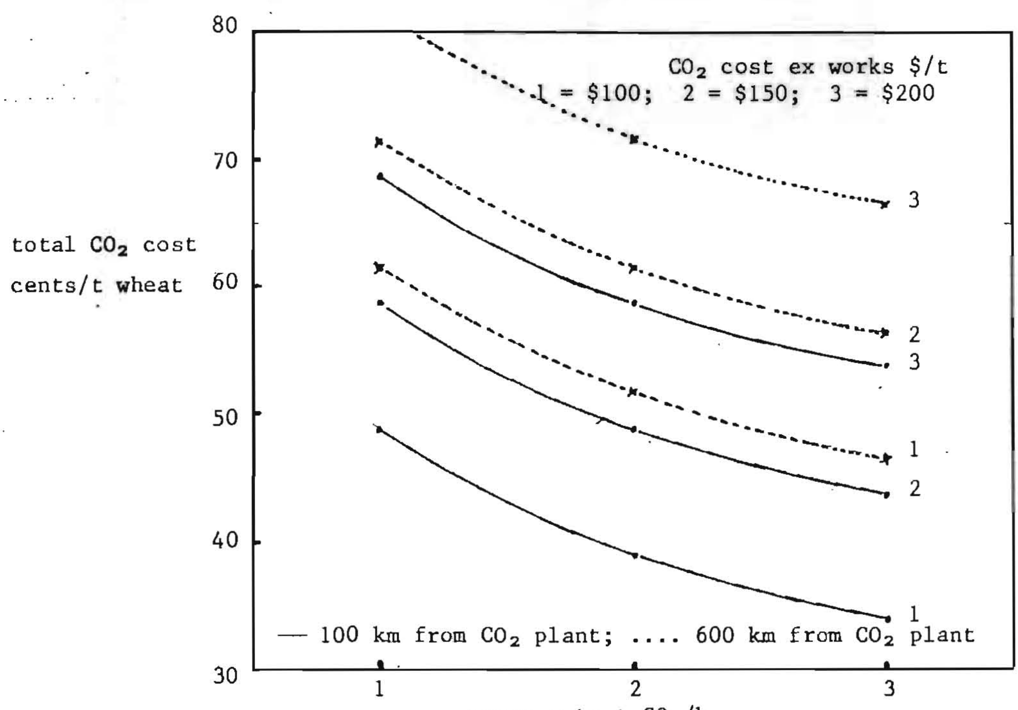

FIG. 7. Effect of purge rate on the total CO_2 cost when purging a 28000 tonne horizontal shed type wheat bin 60% full 40% headspace. CO_2 consumption 2 t/1000 t wheat

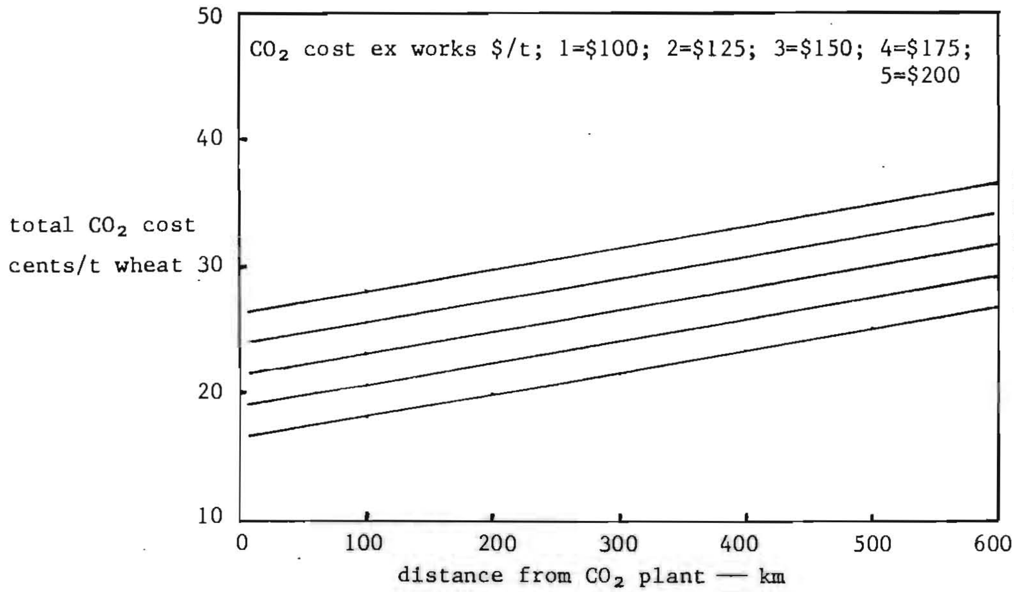

FIG. 8. Effect of distance from CO_2 plant on the total CO_2 cost when purging a 28,000 tonne vertical, steel, multi-bin wheat terminal at an average purge rate of 3 t CO_2/hr. Bins 95% full 5% headspace. CO_2 consumption 1 t/1000 t wheat.

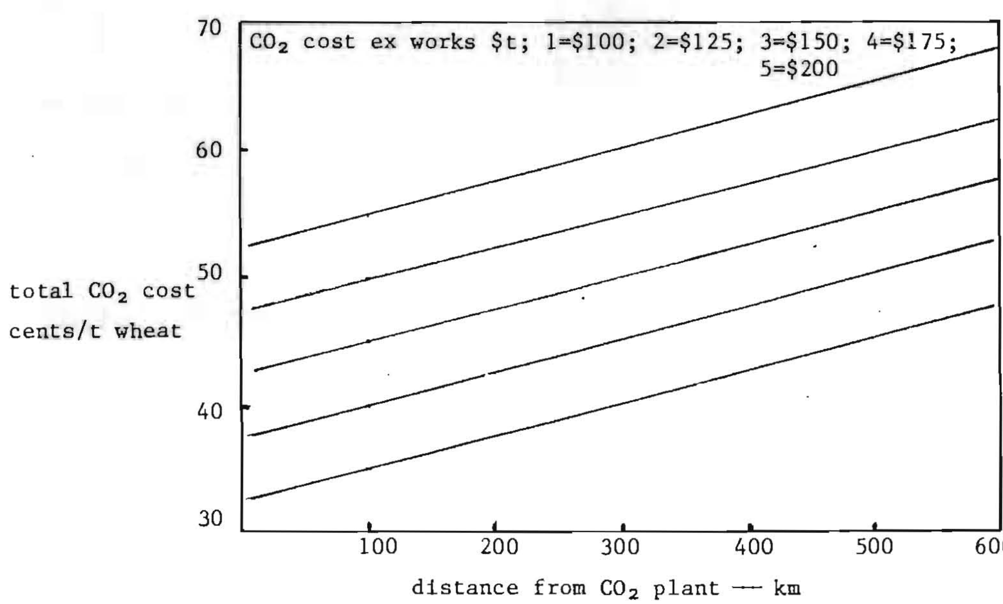

FIG. 9. Effect of distance from CO_2 plant on the total CO_2 cost when purging a 28,000 tonne horizontal shed type wheat bin at an average purge rate of 3 t CO_2/h. Bin 60% full 40% headspace. CO_2 consumption 2 t CO_2/1000 t wheat.

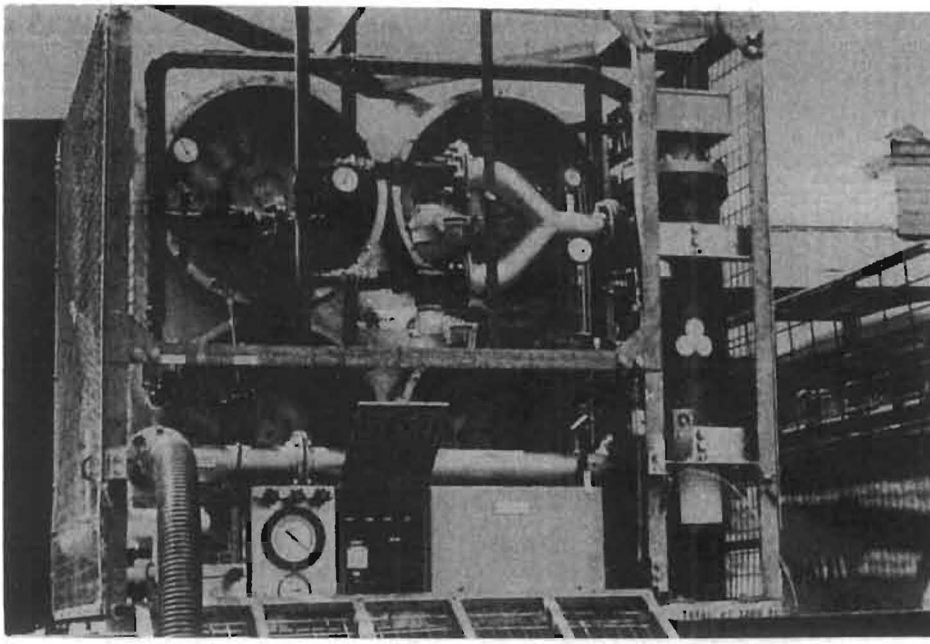

FIG. 10 View of nominal 3 t/h forced draught CO_2 vapouriser with electric superheater.

FIG. 11 Purging 2000 tonne capacity vertical steel bins with 3 t/h CO_2 vapouriser (Yarrawonga, Victoria, 1980)

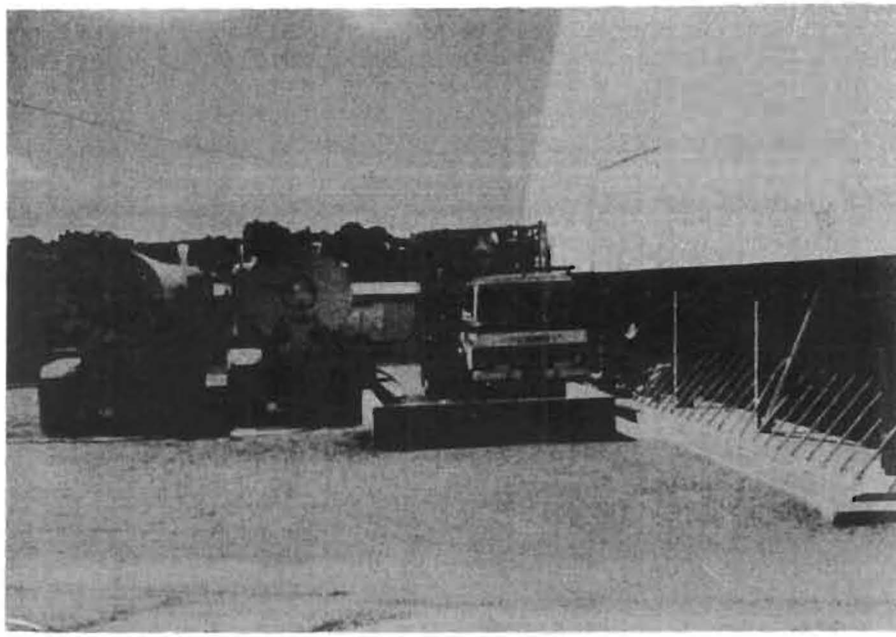

FIG. 12. Purging 16,400 tonne capacity horizontal shed-type galvanised iron bin with 3 t/h CO_2 vapouriser (Harden, N.S.W., 1979)

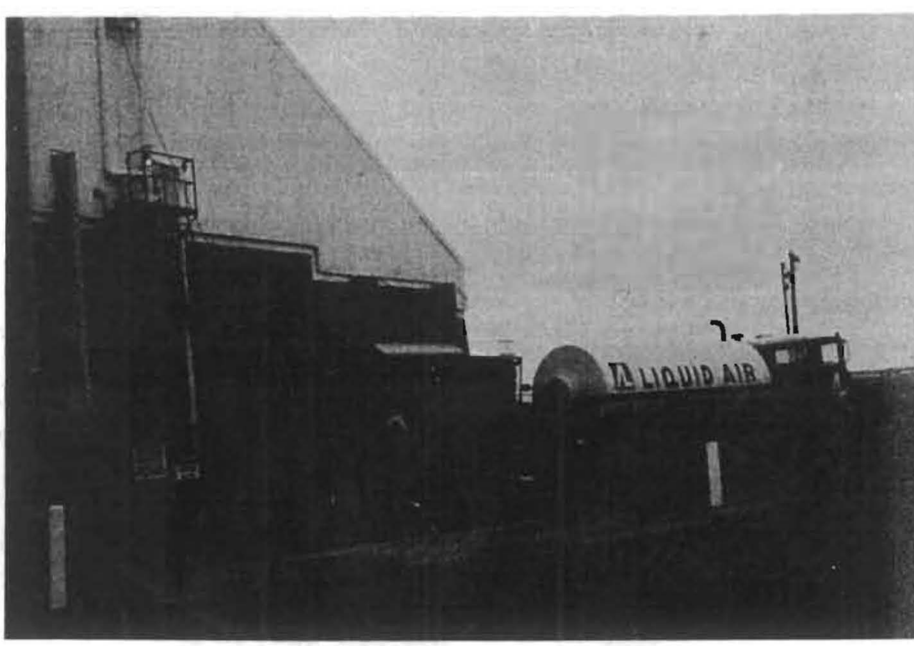

FIG. 13 Purging 25,000 tonne capacity horizontal shed-type concrete-galvanised iron bin with 3 t/h CO_2 vapouriser, (Cunderdin, W.A., 1981)

THE "DETIA BAG BLANKET", ITS USE AND APPLICATION

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ABSTRACT

The "Detia bag blanket" fumigation technique has been developed for the fumigation of huge quantities of stationary bulk grain without the necessity of circulation for fumigation and with a minimum of labour and cost.

Each blanket consists of a gas permeable plastic non-woven material containing 100 Detial fumigation bags (Detia Gas-Ex-B) and it liberates about 1.13 kgs of phosphine.

After the bag blanket is unrolled on the surface of the grain, moisture is absorbed and the liberated phosphine is able to penetrate the grain mass up to a depth of 30 metres. This fumigation technique is successfully used in shipholds, steel tanks, concrete silo bins, bulk storage warehouses and bunker storages under plastic sheets. The penetration time can vary with the different kind of storages as is shown by concentration/time tables. Lethal concentrations can be measured at the bottom of the grain storages in 3 to 10 days. The dosages vary between 0.5 grams (bunker storage, Australia) and 28 grams of phosphine per ton of grain (bulk storage, Germany). Depending on prevailing conditions the exposure time should range between 8 and 20 days.

INTRODUCTION

The "Detia bag blanket" fumigation technique has been developed for the fumigation of huge quantites of bulk grain with a minimum of labour and cost and without the necessity of circulation for fumigation. A bag blanket consists of a gas permeable plastic non-woven material and is about 5 m long with 100 Detia fumigation bags in two rows.

The bags that make up the blanket are well known under the trade name "Detia Gas-Ex-B"; they have been used for the treatment of bulk grain for many years. Normally they are probed singly into the grain to a depth of 3 - 4 m, with two workers being required to make the application. However, if the quantity of grain is more than 10,000 tonnes it becomes more and more problematic to finish the application before hazardous concentrations of phosphine gas are liberated from the bags. Therefore the Detia bag blanket has been developed for rapid application to overcome this hazard and to reduce labour costs.

Each bag blanket is packed as a roll in a gas tight metal can. For application the blanket is removed from the can and unrolled on the surface of the grain. As moisture from air and from the grain is absorbed by the formulation about 1.13 kg phosphine per blanket is liberated. Because phosphine is a small molecule with a density similar to air and because of its high vapour pressure at room temperature (35 bar) it penetrates rapidly and deeply through the interstitial air of the grain kernal into the grain mass.

Since phosphine is almost insoluble in water and does not react with the chemical components of the grain it will be nearly completely desorbed after a relatively short period of aeration. No appreciable residue of the fumigant is left in the fumigated grain if the degassed bag blankets are removed.

Within the last two or three years the Detia bag blanket fumigation technique has been successfully used in shipholds, steel tanks, big and wide concrete silo bins, bulk grain storage warehouses and bunker storages under plastic sheets.

SHIPHOLDS

The fumigation of ships in transit is restricted to bulk carriers and tankers. For the application of the blankets in shipholds it is useful to charge them with grain in order to keep them in their original position even in heavy seas. Concentration measurements have shown that the phosphine will penetrate through 19 to 20 metres and arrive at the bottom of the hold in about 7 days (Leesch et al 1978). This gives an average penetration time of about 3 metres per day.

STEEL TANKS

Similar results were observed in a steel tank with a capacity of 14 000 m^3 (400 000 bushels) (Allen et al 1979) The dimensions of the tank were 30 m (100 ft) diameter, 15 m (50 ft) sidewalls and 20 m (65 ft) peak. Since phosphine is a space fumigant the total volume of the tank was used to determine the quantity of fumigant required.

The chosen dosage was 3.5 Detia fumigation bags per 28.3 m^3 (1 000 ft^3) which corresponds to a phosphine dosage of approx. 1.4 grams PH_3 per cubic metre. For the whole tank 18 Detia bag blankets were applied. These blankets were simply unrolled down the grain slope. The average outside temperature during the exposure period was only 11°C.

Table 1 shows the phosphine concentrations between 1 day and 17 days of exposure in the different places of the tank. The deepest sampling point was 13.7 m (45 ft). A lethal concentration of 160 ppm was observed at this point on the 5th day. On day 9 the average concentration observed was about 600 ppm. The average penetration time was almost 3 metres per day.

TABLE 1 PHOSPHINE CONCENTRATIONS IN WELDED STEEL TANK (400,000 BU.) FUMIGATED WITH DETIA® GAS EX-B (3.5 BAGS PER 1000 CU. FT.). (1,4 GRAMS OF PHOSPHINE PER M^3)

PHOSPHINE CONCENTRATION (PARTS PER MILLION)

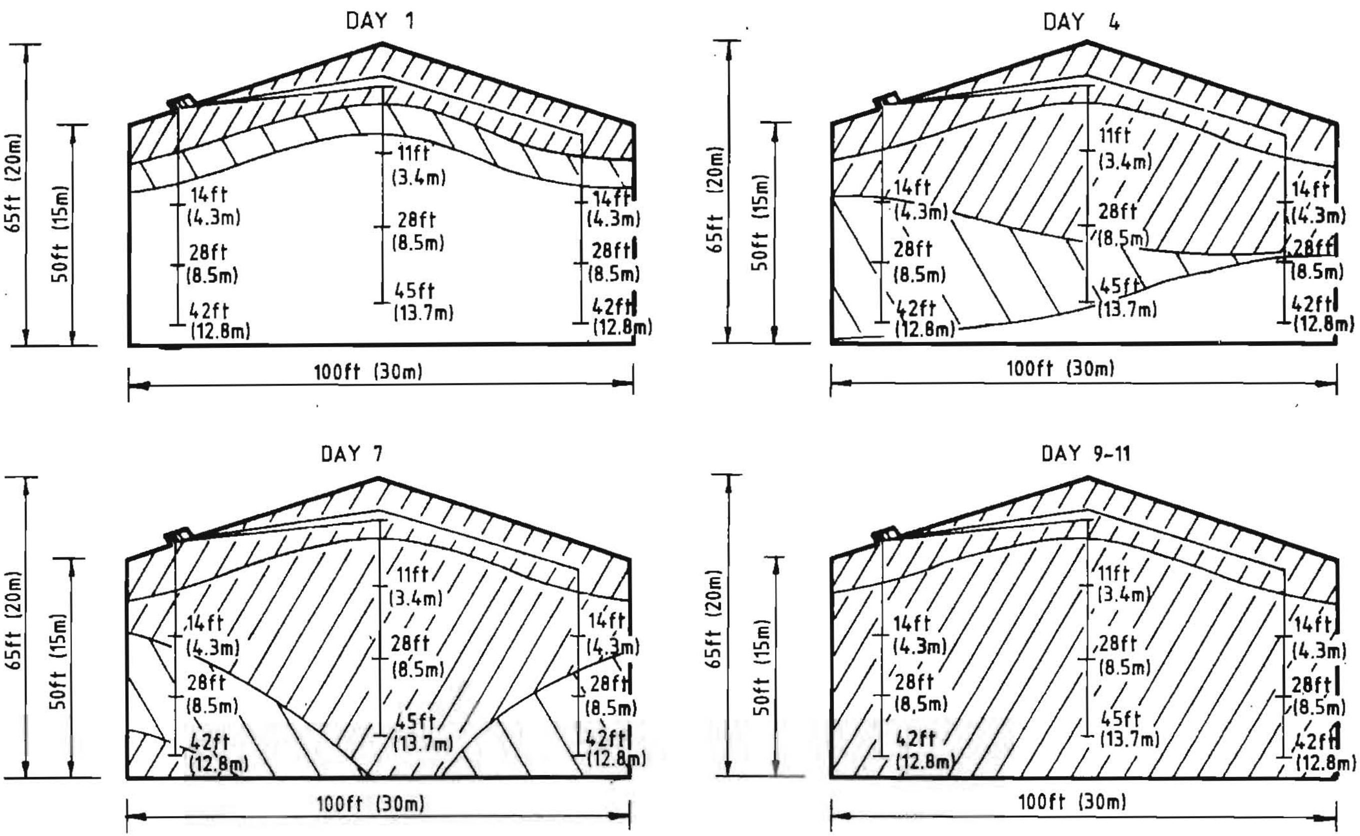
	CENTER				SOUTH SIDE			NORTH SIDE				Sampling Point ← Depth
xposure Period (Days)	Over-space	3.4m (11 ft)	8.5m (28 ft)	13.7m (45 ft)	4.3m (14 ft)	8.5m (28 ft)	12.8m (42 ft)	4.3m (14 ft)	8.5m (28 ft)	12.8m (42 ft)	Aeration Duct	
1	1875	t[1]	0	t	20	t	t	1350	t	t		
2	2940	460	75	t	65	0	10	1455		5		
3	3150	770	600	5	105	5	t	2640	10	10		
4	3375	1800	900	60	200	25	50	1515	20	40		
5	2700	705	1300	160	600	60	75	1200	40	50	390	
6	1830	1250	990	350	520	100	140	1300	50	90	650	
7	1650	1300	1650	550	700	160	200	1300	100	120	900	
8	1275	1230	1250	710	400	240	280	1000	180	190	1020	
9	930	825	925	730	350	320	400	910	230	270	910	
10	930	710	760	1150	575	410	490	825	285	250	875	
11	870	600	750	800	640	490	500	710	350	340	800	
13	700	550	725	580	390	40	280	605	410	415	540	
15	490	375	425	500	205	140	150	270	300	240	490	
17	390	265	205	450	345	255	280	345	320	305	295	

ace concentrations of Phosphine - less than 5 ppm.

FIGURE 1 MOVEMENT OF PHOSPHINE IN WELDED STEEL TANKS FUMIGATED WITH DETIA GAS EX-B ON SUCCESSIVE DAYS (1.4 GRAMS OF PHOSPHINE PER M^3 OR 3.5 BAGS PER 1 000ft^3)

GREATER THAN 200ppm

50–100ppm



The movement of phosphine in such welded tanks can be demonstrated by the schematic drawings of the tank shown in Figure 1. The light hatchings indicate the concentrations between 50 and 100 ppm and the heavy hatchings above 200 ppm. The different sampling points are marked with their depth in feet and metres. As can be seen, after 9 days the concentration of 200 ppm is exceeded on all sampling points. After 17 days the concentrations are still between 205 and 450 ppm.

CONCRETE SILO BINS

In many countries there are big concrete silo bins with a capacity between 2 000 and 4 000 tons. Since all of them are filled after the harvest there is no possiblity to move the grain into an empty bin if fumigation is necessary. Therefore the only possibility is a surface fumigation.

It has been shown by several test fumigations in USA and Zimbabwe that the Detia bag blanket technique can be used for this purpose successfully. However it seems that a certain proportion between diameter and height of the silo bin should not be exceeded. Good results have been obtained with a proportion of 1 : 2. When this proportion exceeds 1 : 4 or 1 : 5 the concentrations that can be attained at the bottom of the bin become increasingly smaller. If it becomes 1 : 10, as in small German silo bins, concentrations of more than 2 ppm will not be attained at the bottom of the bins even after two weeks.

In Zimbabwe a concrete silo bin filled with 3 600 tons of maize was fumigated with 700 Detia fumigation bags (i.e. 7 Detia bag blankets were unrolled on the surface of the grain to give a dosage of about 1 bag per 5 tons or 1.5 grams of phosphine per cubic metre). The bin measured 15 m (diameter) by 30 m (high) to give an almost ideal proportion of 1 : 2. The headspace above the grain was about 1 m and gave an empty volume of about 180 cubic metres.

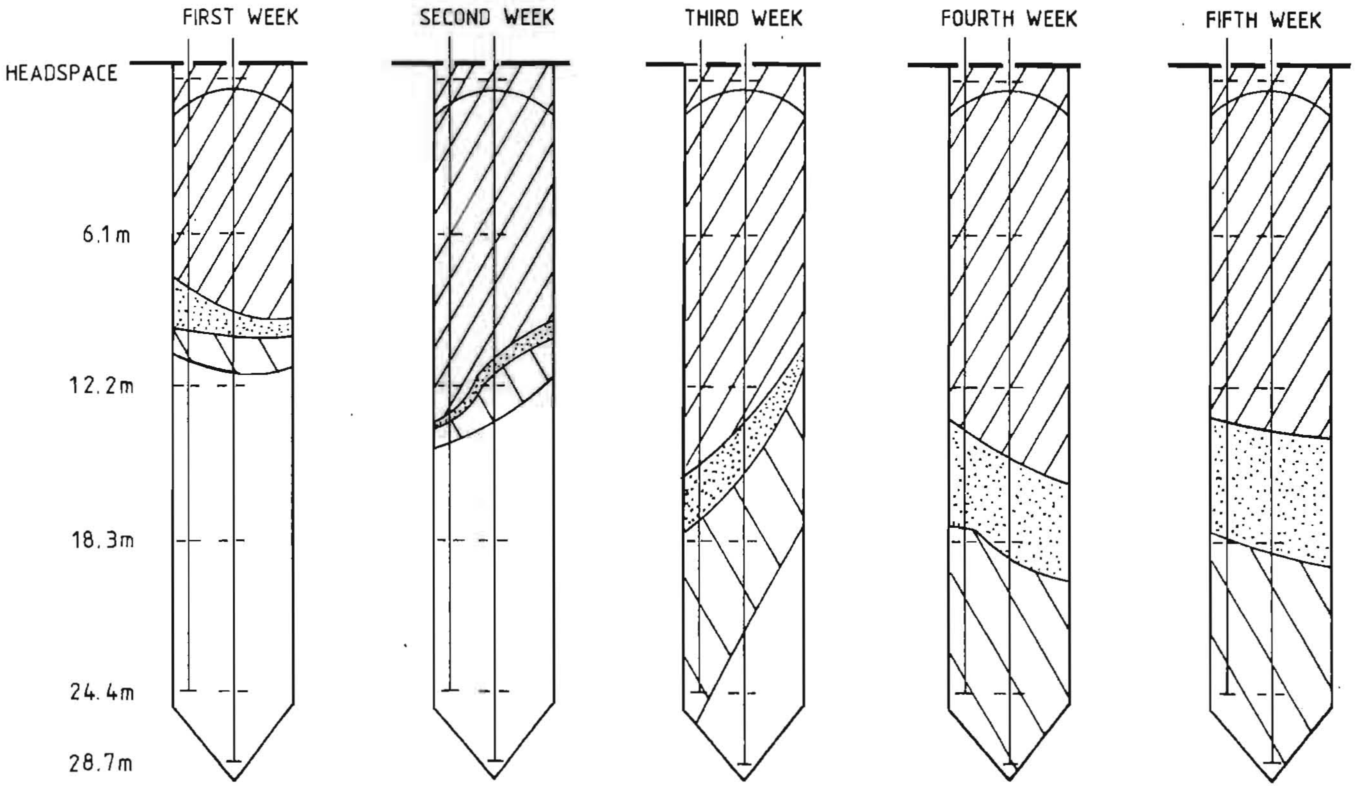
The highest concentration observed in the headspace during the test was 3 600 ppm after 1 day of exposure. After 10 days the concentration at the bottom of the bin exceeded 50 ppm. The highest concentration at the bottom was 450 ppm after 18 days of exposure and the concentration in the headspace at this time was still more than 800 ppm. (Caley 1981)

Distinctly different was the result in an American silo bin where the diameter was 7 m and the height 29 m, with the proportion between diameter and height of only 1 : 4. Using a dosage of 3.2 Detia bags per 1 000 cubic feet (1.3 g PH_3/m^3) it took 27 days to establish concentrations over 20 ppm at the bottom of the bin. Therefore the average penetration time was only 1

FIGURE 2

MOVEMENT OF PHOSPHINE IN A 29 METRES (94 FEET) DEEP CONCRETE SILO FUMIGATED WITH DETIA GAS EX-B
(1.3 GRAMS OF PHOSPHINE PER m^3 OR 3.2 BAGS PER 1 000ft^3)

10 - 50ppm
50 - 100 ppm
GREATER THAN 100ppm



metre per day in this bin. During the following 10 days the average concentration at the bottom was 30 ppm. According to Lindgren and Vicent (1966) and Reynold et al. (1967) such concentrations are sufficient to kill immature stages of various pests if these concentrations can be kept over a longer period of time. The highest concentration in the headspace, was about 3 000 ppm and after 5 weeks it was still above 200 ppm.

The schematic drawings (Fig. 2) show the movement of the gas during five weeks of exposure.

BULK GRAIN STORAGE WAREHOUSES

In bulk grain storage warehouses in Germany and other European countries where large quantities of grain between 5 000 and 20 000 tons are stored the heights of the grain layers vary in most cases between 2 and 5 metres. It has been found that the fumigant can be applied most effectively by covering the blankets with a 5 to 10 cm layer of grain and placing polyethylene sheets on the surface. The liberation and the distribution of the gas under the plastic sheets are then more uniform, the storages have wooden sidewalls covered with plastic sheets in order to prevent gas from escaping. The dosage used in Germany is much higher than in other countries due to the severe climatic conditions and the low temperatures of the grain. An application of 2 $\frac{1}{2}$ Detia bags per ton (28 g of phosphine per ton) with an exposure time of 2 weeks for grain between 9 and 15^{o}C will kill all immature stages of insects present in the grain. The penetration time of the phosphine is about 4 - 5 m per day.

Several tests with wheat, barley, oats and maize have shown that the penetration time is in the same range for the first three grain types but it is significantly shorter for maize.

In tests with maize, concentrations at the bottom of the grain mass exceeded 2 000 ppm but for wheat, oats and barley it was between 300 and 700 ppm after 2 days. Directly above the plastic sheets concentrations of 100 to 300 ppm were measured. This indicates that large quantities of phosphine can penetrate the thin polyethylene sheets to escape into the empty space above the grain. In laboratory tests a new plastic sheet, a polyethylene sheet impregnated with a layer of polyvinylidene chlorides, was found to be reasonably impervious to phosphine. According to these test results the penetration is about 100 times smaller than that of a simple polyethylene sheet.

A fumigation test in a warehouse with bag blankets under these special sheets resulted in much higher concentrations in the grain with the same dosage and exposure time. The concentration at a depth of 4 metres was about

5 000 ppm after 2 days and still 1 500 ppm after 8 days while in the headspace concentrations up to 30 ppm were measured. All of the test insects including the immature stages were killed after 14 days.

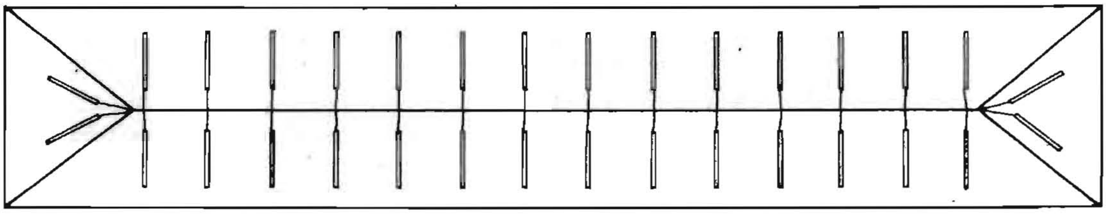
BUNKER STORAGE

In countries where the climatic conditions allow a temporary grain storage in large heaps of grain stored in the open air or underground, the Detia bag blankets can also be used. In the case of Australian bunker storages the grain is only 1 m or 1.5 m underground and is "enveloped" in thick PVC-tarpaulins. The dimensions of these bunkers can be up to a height of 11 metres, 50 metres in width and 100 to 150 metres in length with capacities between 10 000 and 60 000 tons of grain.

For the application of the Detia bag blanket a horizontal slit of about 25 cm is cut into the tarpaulin about 2 metres down from the crest of a bunker. With an iron rod the blanket can be pushed through this slit down the slope of the grain and then the slit can be sealed with a small piece of tarpaulin and glue. Since the PVC-tarpaulins are relatively gas-tight and the exposure time is measured in months, it is possible to use a very economic dosage. Moreover the relative high contents of carbon dioxide in these bunkers (up to 5%) has a synergistic effect on the phosphine. In one test only 29 blankets (about 0.5 g PH_3/ton) were necessary to fumigate a bunker system containing 60 000 tons of wheat (Fig. 3). The whole fumigation procedure was finished in a very short time with only two or three labourers.

FIGURE 3

DISTRIBUTION OF DETIA BAG BLANKETS IN A PVC COVERED BUNKER SYSTEM (60.000 TONS)



The distribution and effectiveness of gas in such bunker systems has been investigated and published by Banks and Sticka (1981). Their results show that within 3 or 4 days insecticidal concentrations of phosphine penetrate to the bottom of the grain. The lowest concentration they found was in one corner of the bunker. Nevertheless a Ct product of 20 gh per m^3 could be achieved over 28 days and none of the caged insects survived at this place. In these tests a phosphine dosage of 0.75 grams per ton was applied.

In Argentina, so called "silos subterraneos" where the grain is stored below ground level, have been successfully fumigated with the Detia bag blanket fumigation system.

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ETHYL FORMATE AS A SAFE GENERAL FUMIGANT

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ABSTRACT

Ethyl formate has been successfully used for individual package fumigation for many years and in tests reported here it has shown promise as a safe general fumigant for stored foods in large quantities. Studies on the use of ethyl formate for the control of stored grain pests have shown that dosages of 300–400 gm/m^3 in an exposure period of 48-72 hours control all stages of insects in stored grain and their products. Large scale disinfestation has been carried out on various agricultural commodities including cereals, pulses, spices, dry fruits, nuts and dried tubers. Fumigation methods have been suggested. The residues of ethyl formate or formic acid are much below the permissible limit of the 250 ppm prescribed. Simple methods of detection of the above have been developed.

INTRODUCTION

Ethyl formate (EF) is a low boiling point liquid insecticidal fumigant with a pleasant aromatic odour. It is used as an intermediate in the synthesis of pharmaceuticals, as an additive of synthetic flavours, production of resins and other compounds. The specifications and physical properties are shown in Table 1 (ANNONYMOUS 1971).

Ethyl formate has been successfully used for individual package fumigation of dry fruits since 1927. The usual dosage for a 11.4 kg box of raisins ranges from about 4 ml in hot weather to 7 ml in cold weather (318–556 mg/l) (SIMMONS AND FISHER, 1945).

Ethyl and methyl formates (MF) were tested by NEIFERT *et al.*, (1925), COTTON AND ROARK, (1928) and ROARK AND COTTON (1929). SHEPARD *et al.*, (1937) found them to be more toxic than carbon disulphide to *Tribolium confusum* Duv., and *Sitophilus granarius* L. VINCENT *et al.*, (1972) have evaluated EF against insects infesting dates and other dried fruits and compared it with phosphine.

Ethyl formate is being produced as a by product in the manufacture of explosives by M/s IDL chemicals Ltd., Hyderabad, India. Therefore a project was initiated at the Central Food Technological Research Institute, Mysore to find a good use for EF. The product had no free formic acid.

MATERIALS AND METHODS

Insect toxicity tests

The toxicity of EF was determined in the laboratory against all life stages of *Sitophilus oryzae* L. and *Tribolium castaneum* Herbst. The insects were reared respectively on *Sorghum vulgare* and whole wheat flour with 5% yeast at 25°C and 70± 10% r.h. For immature stages of *S. oryzae*, about 500 unsexed adults were allowed to oviposit on 500 g of sorghum for 48 hours. The insects were sieved out and the infested grain was held at the rearing conditions till the developing stages attained the required age. Then the grain was weighed into 25 g aliquots for fumigation. Pre-adult stages of *T. castaneum* were obtained from regular cultures. Similarly 2-3 week old unsexed adults of both species of insects obtained from established cultures were tested. In these cases there were 30 insects in each replicate. In each age group 5-11 doses were tested with 4-9 replicates per dose. All fumigations were carried out for 24 hours at the laboratory conditions of 27.4 ± 2.2°C and 60-80 r.h. in 850 ml desiccators provided with septum caps on the lid for fumigant dosing.

EF was drawn as a vapour in a 20 ml gas-tight syringe from a gas-wash bottle containing excess liquid to obtain a saturated concentration (BROWN, 1951). The vapour was injected into the desiccators through the septum caps after establishing a slight vacuum to draw the vapour in.

Following treatment, mortality of the immature stages of *S. oryzae* was assessed based on the number of adult emergences and that of *T. castaneum* on development to the next metamorphic stage. Adult mortality was assessed 10 days after the treatment. The data were corrected for natural control mortality and analysed statistically by the method of LITCHFIELD and WILCOXON (1949) (Table 2).

Effective doses of ethyl formate in foods

Three hundred gm each of 27 commodities were fumigated in triplicate in 850 ml desiccators with EF to ascertain the effective dose required in a 24 hour treatment. The test insects were *T. castaneum* adults in open end glass tubes with cloth diaphragm closures. The EF was administered as a liquid to give dosages ranging from 300-500 mg/L. The sorption ratios were calculated by dividing the effective commodity dose (LD_{100}) by the LD_{95} dose (32.2 mg/L; upper limit) (Table 3).

Penetration of ethyl formate in grain columns

The penetration of EF was studied in 1-5 meter tall 20.25 cm diameter (i.d) air tight mild steel (pipe) columns fitted with spouts and stopcocks at the top, middle and bottom locations. Each spout contained 40 kg of wheat and insect exposure tubes containing 30 *T. castaneum* adults. The lethal CT product was determined when there was cessation of all movement of the insects.

EF at dosages of 100, 200 and 300 mg/L was administered as a liquid in a syringe at the top spout. Mortality time was noted at each of the three locations (Table 4).

Trials on paddy rice

EF and MF were poured over seed paddy in 0.5 tonne galvanised iron (GI) bins, carrying an infestation of *Sitotroga cerealella* moth. Mixtures of EF and ethylene dibromide (EDB) absorbed in pieces of chalk were also probed to different depths of the bin using a hollow metal probe. Table 5 depicts the details of these tests.

Penetration of EF through sealed flexible packs

Cereal based "Energy food" (100g) in sealed flexible pouches were fumigated separately in 2.5 L desiccators with Ef at a dosage of 300 mg/L with an exposure of 48 hours. Each pouch had 30 *T. castaneum* adults in glass tubes with cloth diaphragm closures. The penetration of EF into the packages was computed by estimating the residual gas concentration by gas chromatography, as described in this paper, and deducing the quantity that penetrated the packages from concentration of EF in a control desiccator with no packages. The data are shown in table 6.

Determination of EF in air and as residue in foods

1 Qualitative tests:

Blue litmus paper (E. Merck) (pH 4.5 red:8.3 blue), methyl red treated paper strips (pH 4.2 red:6.2 yellow), bromo creso green treated paper strips (pH 3.8 yellow: 5.4 blue) were placed inside 250 ml gas bubblers and vapour of EF to give a concentration of 100 ppm (the T.L.V. in air Torkelson et al 1966) was injected into the gas bubbler after creating a slight vacuum. The change in colour of the strips was recorded after 30 minutes of exposure.

2 Quantitive tests:

Quantification of the blue litmus colour change was attempted by using a Reflectance meter (Table 7).

2.1 Colourmetric method: Alzarin; 1,2 - dihydroxy anthraquinone was used as the reagent and the pinkish violet colour developed was read at 520 nm in a Bausch & Lomb Spectronic 20 colorimeter.

Reagents: (a) 1ml of 1% sodium hydroxide solution:

(b) 0.5 ml of 0.1% alizarin

(c) EF standard solution:

The final volume was made up to 10 ml in distilled water.

Procedure: A standard solution of EF consisting of 20 mg EF in 1 ml of methyl alcohol was prepared and 1-10 ml was taken in separate tubes. 0.5 ml alizarin solution and 1 ml of 1% sodium hydroxide was added and shaken well before taking the reading. Beer's law was applicable in the range of 20-80 μg of EF.

2.2 Interferometric analysis: A "Riken 18" interferometer was calibrated for EF. The soda lime scrubber was replaced with a paper towelling scrubber to avoid reaction of EF. Known concentrations of the vapour were prepared in a 100 ml syringe attached to the intake end of the interferometer and the concentrations were analysed. The interferometer was used in all field experiments along with a bioassay method to monitor the concentrations.

2.3 Gas chromatography: AIMIL gas chromatograph model 5580 with an FID mode was employed under the following conditions for analysis of EF-

Detector temperature	105 - 112°C
Oven temperature	34 - 42°C
Injector temperature	77 - 84°C
Balance current	x 100
Attenuation	x 16
Carrier gas flow(Nitrogen)	60 ml/min
Hydrogen	40 ml/min
Column	2 m, 1/8" dia SS loaded with 80-100 mesh chromosorb W coated with 5% dinonyl phthalate.
Retention time	40-45 seconds
Sensitivity	1 μg EF.

Table 8 gives the standard curve for EF.

2.4 Detector tube method: Methyl red solution (0.1g in 18.6 ml of 0.02 N sodium hydroxide made up to 250 ml; PH 4.2 red; 6.2 yellow) was impregnated on 30-50 mesh celite, dried at room temperature and packed into 17.8 cm x 0.2 cm (i.d.) glass tubes with a rubber septum cap for injecting samples from a syringe (MUTHU AND MAJUMDER, 1973). The tube was tested for both EF (60 ml air sample) and formic acid (100 ml air sample) with encouraging results.

Table 9 dipicts the calibration between quantity of vapour of EF and formic acid and the length of the red band formed. There were three replicates per concentration of each vapour. A regression line was drawn for EF whereas for formic acid, as there was perfect correlation between quantity of sample and band length, no further statistical treatment was applied.

Determination of residues in foods

Residue as EF

Fourteen commodities were fumigated in 100 gm lots in 210 ml gas washing bottles with three replicates per commodity. EF was dosed at the rate of 300 mg/L with an exposure period of 3 days and an aeration of one week under static conditions. The head space gas was analysed by gas chromatography as described earlier after keeping the gas-wash bottles in a water bath for 30 minutes at 80°C to desorb the EF sorbed. The calculated values are shown in Table 10.

Residue as formic acid: As the tolerance of 250 ppm as total or combined formic acid is the residue stipulated by the food and Drug Administration of USA (Federal Register, 1979) it is important to estimate this in foods.

Blue litmus paper was again used to determine the formic acid in vapour form. Graded concentrations of formic acid vapour were established in 210 ml gas bubblers with blue litmus paper strips in each of them (4-5 replicates per concentration). The colour formed was read in the Reflectance meter (ELICO REFLECTOMETER CL-28). Data are shown in Table 11 along with the regression equation. The residues determined by the above method in certain foods are given in Table 12.

Large scale fumigation trials

Attempts to use EF for large-scale fumigations have been rather sporadic. PELIKH *et al.*, (1940, 1941) found it effective against warehouse insects at a dosage of 250 g/m^3. Surface treatment of wheat infested by *R. dominica* and *Lathericus oryzae* at dosages of 454-568 ml per 0.84 square meter proved in-effective (WILSON and MILLER 1946). NEIFERT *et al.*, (1925) found it quite effective for grain fumigation in box-cars. Wheat germination was not adversely affected (COTTON and ROARK, 1928).

In view of lack of enough data on EF for large scale treatments several trials were conducted on bag stacks of wheat, pulses, spices, dried tapioca chips and deoiled copra cake.

Fumigations were mostly carried out under gas-proof sheets (rubberised fabric tent, low density polyethylene sheet tent, high density polyethylene laminated on ether side with low density polyethylene sheets, neoprene coated nylon sheet (balloon cloth). A wheat stack was enveloped by both a rubberised fabric tent and a 1000 gauge low density black polyethylene tent. The gas-proof sheets were sealed to the floor by mud-plaster or sand.

Arecanuts (betel nuts) were fumigated in a brick masonry room with wooden ceiling made airtight by mud plaster sealing. The doors and windows were sealed using gummed paper strips.

Fumigation distribution: The fumigant distribution system consisted of dichotomously branched rubber pressure tubing laid on top of the stack before covering, and secured with strings to prevent 'whipping' at the time of discharge of the fumigant. At the exit ends of the branches gunny sacking was placed to absorb and vaporise the liquid EF. The main fumigant feeder tubes were brought out of the stack at the floor level and connected to a sprayer pump. After pressurizing the unit the fumigant was discharged into the tubes.

Unglazed baked clay pans were also used for distributing the liquid EF in some fumigantions especially on stacks in rooms. They were distributed on top of the stacks.

Test insects

The test insects consisting of 30, 2-3 week old *T. castaneum* adults were placed in glass tubes plugged with cotton wool which, in turn, were inserted in brass perforated capsules that were screwed on to 1M long galvanised iron probes (GI) with a 'T' handle. A gas sampling tube ran inside the core of the tube, with the sampling nipple in the middle of the 'T'. The probes were randomly thrust into the top, middle and bottom strata of the stack into the bags.

Gas concentrations

EF concentrations were determined during exposures at the top, middle and bottom locations from the probes using the Riken-18" interferometer. The concentrations were plotted against the exposure times and the concentration time (CT) curves drawn. The areas encompassed by the curves represent the integrated CT products expressed as mg. hrs/L. This gave an indication of the effectiveness of gas distribution and toxicity to insects.

Bio-assay of gas concentrations

The bioassay method for assaying gas concentrations (MUTHU *et al*, 1971) was also tried. Fifty ml capacity U-tubes with stoppers and side spouts containing 30 *T. castaneum* adults were scrubbed with the gas samples connected to the interferometer intake line so that the reading or the interferometer denoted the concentration in the U-tube as well. After taking the reading the U-tubes were closed and the time of sampling noted. The knock-down time was recorded to arrive at the total time that had elapsed from the time of sampling. The EF concentrations as determined by the interferometer multiplied by the knock-down times give an estimate of the CT products that could be employed for determining the concentrations by the bio-assay method.

Aeration was done at the end of the exposure periods by cross ventilation. About 500 gm of samples were drawn from the top, middle and bottom locations of the stacks and sieved to gauge the mortality suffered by the resident infestation. The commodity samples were incubated in the laboratory for a period of one month at 25-27°C and sieved every week to determine the effect of the treatment on the pre-adult stages of insects (on emergence). Germination tests were conducted on samples of wheat drawn before and after fumigation. Residues as EF were determined in some of the samples after fumigation.

RESULTS AND DISCUSSION

Insect Toxicity tests

Eggs of both *S. oryzae* and *T. castaneum* were found to be most susceptible and pupae most tolerant. The maximum CT product *viz* 1110 mg hrs/L, the LD_{95} for *S. oryzae* pupae, is much below that of 1,1,1 Trichloroethane (Methylchloroform) which requires a CT product of over 33,300 mg hrs/L (PICL REPORT 1977-79) and carbon tetrachloride which is effective against all stages of *S. oryzae* at 22,000 mg.hrs/L (PICL Report 1974-76)

Effective doses of EF in foods

Rice bran required the highest dose of 500 mg/L followed by copra cake and semolina (400 mg/L). Split pulses, walnuts, pepper and copra needed 150-200 mg/L. followed by ginger, paddy and coriander (100 mg/L). All the rest could be effectively treated at 50-75 mg/L with the exception of green gram which showed the least sorption (30 mg/L being sufficient for the treatment). SIMMONS and FISHER (1945) have recommended a dosage of 4 ml per 25 lt box of raisins which works out to a dose of 318 mg/L. This could compensate for sorption and leakage losses from the containers.

Penetration of EF in grain columns

The data show that a dosage of 300 mg/L is required for adequate penetration of EF through the grain and this took about 8 hrs. It would be possible to fumigate wheat in farm bins with EF, extrapolating these results.

Seed paddy in bins

An examination of table 5 shows that for paddy a dosage of 712 g/m^3 of EF is necessary to control *S. cerealella.* Mixtures of 1.5:1 (W/W) of EF and EDB absorbed in pieces of chalk is also equally effective. Mixture, of EF-MF, 1:1 (V/V) at 300 ml for 2 quintals of paddy gave only partial control of *S. cerealella.* MF did not control the insect even at the effective dose of EF, *viz.* 712 g/m^3 (600 ml). The bin lids were found to be illfitting although efforts were made to seal them well with gum-paper strips. MF having a lower boiling point (31.5°C) might leak out if the system is not air tight. Germination of seed paddy (12%m.c) was not hampered by the treatments.

Penetration of EF through sealed flexible packs

It appears that EF can penetrate sealed flexible packs, especially low density polyethylene, and kill the resident infestation. The fumigant may not pose problems of toxic residues by the time it is delivered to the consumers as the quantity of residual EF would be low due to outward diffusion. The technique used is a new approach to in-package fumigation as presently understood and simplifies the procedure considerably.

Determination of EF in air and as residue in foods

1. Qualitative tests: Blue litmus, methyl red and bromocresol green paper strips were equally effective in signalling EF at 100 ppm, the TLV in air.

2. Quantitative tests:

(1) Reflectance meter readings (Table 7) were linear between 100-500 ppm of EF.

(2) Colorimetric method using alizarin also proved promising.

(3) In the Interferometric analysis one division on the ocular scale of 0-10 corresponded to 8 mg/L EF. Concentrations down to 0.16 mg/L could be read on the vernier scale, carbon dioxide concentration interfered with the reading at concentrations above 1%.

(4) Gas Chromatographic method could analyse concentrations down to 1 μ gm EF and proved useful in residue determination.

(5) The Detector Tube Method could be employed in determining air concentrations of EF as well as its residue in foods as EF or formic acid.

Residues as formic acid

The residue as formic acid in the foods fumigated with EF were far below the stipulated level of 250 ppm by the method used. Although the method of residue determination may not account for all the residual formic acid present it serves to indicate the presence of low levels. The saturation concentration of formic acid is around 134 mg/L at 30°C. The natural formic acid occurring in several foods and beverages far exceeds the residue levels from fumigation with EF (fruits 20–40 ppm, coffee roasted 1350–2200 ppm/. Cheese, 20–30 ppm Evoporated milk 30–40 ppm; FDA, 1976). In the FEDERAL REGISTER (1979) the following comments are made "the agency proposes to affirm the generally recognised as safe (GRAS) status of ethyl formate as a direct human food ingredient and of formic acid and its sodium salt as indirect human food ingredient..." current good manufacturing practice results in a maximum level, as served, of 500 ppm in baked goods, 400 ppm in chewing gum, hard candy and soft candy, 200 ppm in frozen dairy desserts, 300 ppm in gelatins, puddings and fillings and 100 ppm in all other food categories.

No adverse effect attributable to formate were found in five successive generations of rats given up to 200 mg of calcium formate per kg of body weight daily (FDA 1976).

Large scale fumigation trials

Wheat bag stacks: When EF was applied to stacks about 20 minutes were required to discharge the fumigant and there was a faint smell of EF in the vicinity. Integrated CT products of 2573–4054 mg hrs/L were obtained with 100% mortality of *T. castaneum* adults at top middle and bottom locations in the probes (Table 13). The effective CT product estimated by the bio-assay method using *T. castaneum* adults worked out to 123 mg hrs/L at 32–35°C (Table 14). The smell of EF dissipated rapidly and the pest control personnel did not feel any discomfort during the degassing operations.

Incubation tests

The data on insect emergences from the grain at the end of each week and the associated life stages are shown in Table 15. Although a good control of insects was observed as evidenced by the incubation tests the integrated CT product of 2573 mg. hrs/L appeared inadequate as denoted by some surviving insects at the top; CT products of over 3000 mg. hrs/L appeared to be necessary for an overall 100% effect. Good gas-tight covers and a dose of 400 gm/m^3 may be necessary to achieve this. Germination of the seed was unaffected.

The residues determined after 9 months storage were negative to EF.

Fumigation of turmeric

The data are shown in Table 16. There was 100% mortality of both test insects and resident insects in the turmeric samples (*Stegobium paniceum;* all stages). Integrated CT products of 2690 and 2570 mg. hrs/L were recorded at the top and bottom locations.

Fumigation of field beans and coriander

Unglazed baked clay pans were used to distribute EF on the top of the bag stacks in this trial. 100% mortality of *T. castaneum* adults used as test insects and the resident infestation of *Collosobruchus chinensis* and *S. paniceum* in all their stages of development was obtained. Integrated CT product of 2394 mg hrs/L was estimated in a 48 hrs exposure (Table 17).

Fumigation of arecanuts (Areca catechu)

Fifteen clay pans were used to distribute EF in a go down with wooden plank ceiling. The pans were spaced uniformly on the 300 bags. A dosage of 300 g/m^3 with an exposure of 70 hrs was given. Results are shown in Table 18. The mortality of test insects (*T. castaneum*) adults were inconsistent with integrated CT products obtained. However the nuts remained insect free on incubation. There was also no "off" taste in the nuts when chewed.

Fumigation of tapioca chips

High moisture tapioca chips (750 bags; 50 tonnes) infested by *Araecerus fasciculatus*, the coffee bean weevil, was fumigated under a balloon cloth gas proof sheet for 42 hrs at 30°C. As the interferometer could not be relied upon to give accurate values for EF due to a high carbon dioxide build-up from fungal infection, only bio-assay with *T. castaneum* adults was attempted. The results are shown in Table 19. Heavy mortality of *A. fasciculatus* was observed as well as 100% mortality of the test insects. The incubated samples were found insect free. It was learnt later that the fumigated lot was passed for shipment as the quality was found excellent compared to the unfumigated lots.

Fumigation of expeller copra cake

Four hundred bags with a volume of 48 m^3/m. were fumigated using a dosage of 500 gm/m^3 with an exposure of 70 hrs at 26.7°C. The results shown in Tables 20 and 21 indicate that there was 100% mortality of *T. castaneum*, *S. paniceum*, *Necrobia rufipes* and *cryptolestes* sp. in the samples sieved after fumigation. The residue as formic acid in a composite sample was 2.3 ppm as determined by the detector tube method, described earlier. No live insects were seen in the incubated samples drawn after fumigation. Heavy infestation was observed in the "before fumigation" sample.

CONCLUSION

A total of 27 field bioassay results, on analysis showed the mean CT product of 154 $\pm$ 42 mg. hrs/L. could be used as a working CT product to determine approximate concentrations of EF in the field.

Dosages of 400 g/cu.m may be required as a blanket dosage for most commodities at an exposure period of 72 hrs. as at 300 g/cu.m survivers were seen at the top. It is imperative that good gas proof sheets are used for better fumigant retention as EF has a vapour pressure of 312 mm Hg at 30°C.

No fire hazard is likely if proper precautions are taken like no-smoking or sparks from electrical short-circuits etc., even internal heating of commodity to nearly 40-50°C met with in the fumigation of wet tapioca chips did not "spark off" an explosion.

For bag stacks the best method of application appears to be the use of branched tubings with properly aligned manifolds for proper fumigant distribution with empty gunny sacking being kept at the exit points to take care of the liquid splash. A hand compressor sprayer could be used as a pressure source. The filling of this can be done in the open to avoid vapour build up in enclosed spaces. For room fumigation the shallow unglazed baked clay pans used could aid rapid evaporation of the liquid due to the porosity and extended surface area for quick evaporation. Liquid soaking of the commodities could thus be avoided and any formic acid produced by hydrolysis of EF would remain in the pans.

Normal precautions of establishing good cross ventilation before degassing appears to be adequate. At the end of the exposure period concentrations around 16-30 mg/L might remain and these would dissipate quickly due to the high vapour pressure and diffusion characteristics of the compound. The smell is pleasant and well tolerated compared to the halogenated hydrocarbon fumigants (ethylene dibromide, ethylene dichloride, carbon tetrachloride etc.).

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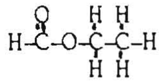
TABLE 1

Physical Properties and specifications of Ethyl Formate
(Ethyl Methanoate, Formic Ether; $HCOOC_2H_5$)

Structural formula		
Molecular weight(calc.)	...	74.08
Boiling point at 760 torr	...	54.1°C
Vapour pressure at 20°C	...	200 torr(mm Hg.)
Freezing point	...	-80°C
Density of liquid at 20°C	...	0.917 g/ml
Specific gravity 20/20°C	...	0.924
Density of gas at 25°C	...	1 g/Litre
Vapour density (Air=1)	...	2.56
Specific heat at 32°C	...	0.478 cal/g(°C)
Auto/ignition temp. in air	...	455°C
Explosive limits in air(approx.)	...	2.8%-16.5% by vol.
Solubility in water at 25°C	...	14.5% by wt.

Specifications:

Ethyl formate(min)	...	98% by wt.
Acidity(max)	...	0.1% by wt. as formic acid.
Odour:	...	aromatic, non-residual
Suspended	...	Substantially free
Distillation range: Initial B.P.	(min)	52.0°C
98%	(max)	54.5°C

Ethyl formate is miscible with alcohol and ether and gradually hydrolyses with water to formic acid and ethanol (5 days). Anhydrous sodium sulphate and calcium chloride minimise this.

TABLE 2

Toxicity of Ethyl formate to the Life Stages of *S. oryzae* and *T.castaneum*

Species		Stage	LD_{50}(Mg/L) Value	95% limits		LD_{95}(Mg/L) Value	95% limits		c x t (mg.hrs/L)
S. oryzae:		Egg	6.2	5.6,	6.8	11.8	10.0,	14.0	283
		Larvae							
Age	(	5 days	9.0	8.5,	9.6	16.0	14.8,	17.4	384
	(	9 days	10.4	9.6,	11.2	21.1	19.0,	23.5	506
	(	13 days	15.7	14.6,	16.8	30.3	27.1,	33.9	727
Age	(	16 days (pre-pupa)	20.0	19.01	21.1	44.2	39.6,	49.3	1060
	(	24 days (pupae)	13.7	13.2,	14.2	46.3	44.0,	48.8	1110
		Adults 2-3 week old	14.3	13.4	15.3	24.5	19.2	30.6	588
T.castaneum:									
		Egg	2.5	2.3,	2.9	6.4	5.3,	7.8	154
		Larva	8.8	7.4,	10.5	24.6	17.4,	30.6	590
		Pupa	18.2	16.6,	20.1	27.6	23.5,	32.4	662
		Adults 2-3 week old	19.5	18.8	20.3	26.8	22.3	32.2	643

TABLE 3

Effective Dose of Ethyl formate in Foods

Commodity	LD_{100} (actual dose: mg/L)	Sorption ratio commodity dose: LD_{95} dose
Paddy	100	3.1
Rice	75	2.3
Wheat	75	2.3
Bengal gram	50	1.6
Green gram	30	1.0
Cow pea	75	2.3
Peas	50	1.6
Bengal gram splits	150	4.7
Groundnut kernel	75	2.3
Cashew kernel	75	2.3
Almond kernel	75	2.3
Copra	150	4.7
Raisins	50	1.6
Dates	50	1.6
Walnuts	200	6.2
Turmeric	50	1.6
Ginger	100	3.1
Cumin	75	2.3
Tobacco	75	2.3
Tamarind pulp	75	2.3
Green gram splits	200	6.2
Pepper	200	6.2
Coriander	100	3.1
Rice bran	500	15.6
Copra cake	400	12.5
Copra cake powder	400	12.5
Semolina (wheat)	400	12.5

TABLE 4

Stratification of Ethyl formate in Wheat Denoted by Knock-down (k.d.) Time of T.castaneum Adults:(Hrs.)

Dose Mg/L	Knock-down time		
	Top (Surface)	Middle (0.75m)	Bottom (1.5m)
100	4.8	8.0	No mortality even after 1 week
200	1.1	3.0	-do-
300	1.0	1.0	8

TABLE 5

Fumigation of seed paddy with Ethyl formate, Methyl formate and Ethyl formate+Ethylene dibromide

Bin No.	Variety	Quantity quintals (100 kg)	Fumigant	Dosage g/m^3	Remarks
1	Madhu	5	EF+EDB (1.5:1 w/w)	700 280 EDB 420 EF	S.cerealella controlled
2	Madhu	5	EF	356	S.cerealella living
3	Madhu	4	EF	712	Good control
4	Vani	2	EF+Methyl formate (MF) (1:1 v/v)	300ml	Partial control
5	Vani	5	MF	600ml (840 mg/L)	Not controlled

TABLE 6

Penetration of Ethyl formate into Flexible packs

Package type	Amount Penetrating (mg)	Mortality of T. castaneum adults (%)	Remarks
1 HDPE 200 (gauge)	99.6	98	
2 HDPE 300 (gauge)	90.1	98	
3 LDPE 300 (gauge)	115.0	100	Quantities of
4 .MSAQ Cellophane	114.2	98	over 115 mg.
5 MST cellophane	95.9	98	are associated
6 Aluminium foil			with 100% effect.
laminate	80.9	90	Exposure of 72 hrs
7 Polycel	115	100	may help

1 HDPE : High density polyethylene
3 LDPE : Low density polyethylene
4 MSAQ : Moisture proof, sealable, anchored, opaque
5 MST : Moisture proof, sealable, transparent
6 60 gm paper /0.02mm foil/ 150 LDPE
7 Polycel : Polyethylene-cellophane laminate
LDPE 150 (gauge) / Cellophane 300 gm.

TABLE 7

Determination of Ethyl formate using Blue Litmus Paper: Reflectance Measurements

Ethyl formate ppm	mg/L	Reflectance %	Remarks
0	0	37	
50	0.15	39.5	Curve linear
100*	0.30	43	between 100-500
250	0.75	44	ppm
500	1.50	45	

* TLV

TABLE 8

Gas Chromatography of Ethyl Formate: Standard Curve

Ethyl formate (μgm)	Peak height (cms)
1.14	0.67
5.70	3.05
11.40	5.70
17.40	9.70
22.80	12.40

TABLE 9

Detector tube method for Determination of Ethyl formate and Formic acid

Ethyl formate		Formic acid	
mg (x)	Band length:cms (Y_p)	μg	Band length (cms)
0.25	1.4	1	0.15
0.50	1.7	2	0.2
1.0	2.3	4	0.4
2.0	3.4	8	0.8
3.0	4.6	12	1.2
4.0	5.7		
5.0	6.8		

Regression equation:

$$Y_p = 3.7+1.13\ (x-2.25)$$

TABLE 10

Residue of Ethyl formate in Fumigated Foods

Commodity	Ethyl formate (ppm)	Permissible Levels (ppm) (FEDERAL REGISTER, 1979)
Cardamom	12.75	100
Cashewnut	1.17	15
Cow pea	0.27	100
Bengal gram	1.62	100
Bengal gram splits	Not detected	100
Groundnut seeds	0.49	100
Green gram	4.62	100
Raisins	39.32	250
Rice (polished)	9.45	100
Sorghum	6.32	100
Turmeric rhizomes (dried)	8.65	100
Cumin	7.70	100
Wheat	0.88	100

TABLE 11

Estimation of Formic acid with Blue Litmus Paper

Formic acid: μ gm (X)	Y_p	Reflectance: % (Y)	Regression Equation
0	43	41	
62.5	44	44	
125	45	47	Y_p=46.4+0.0184 (x-187.5)
250	47.6	49	
500	52.0	51	

TABLE 12

Residue of Formic acid in Ethyl formate Fumigated Foods: Blue Litmus Paper Strip Method

Commodity	Reflectance %	Formic acid μ gm	Residue ppm
Bengal gram	48.	250	2.5
Bengal gram splits	41	0	0
Cashew nuts	44.5	80	0.8
Cow pea	45	120	1.2
Cumin	45	120	1.2
Groundnut seeds	47	220	2.2
Field beans	40	0	0
Turmeric	47.5	250	2.5
Wheat	44.5	80	0.8

TABLE 13

Concentration of Ethyl formate, c.t. products and Mortality of *T.castaneum* Adults in Probes

Exposure: Hrs	Concentration of Ethyl formate (mg/L)		
	Top	Middle	Bottom
1	20	84	80
19	54.4	76	80
27	52.8	66.4	66.4
42	40.0	41.6	46.4
72	25.6	26.6	26.2
Integrated c.t. Product: mg.hrs/L	2573	3919	4054
Mortality of *T.castaneum* adults (%)	100	100	100

TABLE 14

Bioassay of Ethyl formate concentrations in the wheat bag stack at the end of 19 hrs. of exposure. Temperature: 32-35°C

Location	Knock-down time:Hrs.	Conc. of Ethyl formate: mg/L	c.t. product mg.hrs/L
Top	2.5	54.4	136
Middle	1.5	76.0	114
Bottom	1.5	80.0	120
			123(mean)

TABLE 15

Effect of Ethyl formate Fumigation on the Life Stages of Stored Product Insects in Wheat

Date of Screening	Location of Sampling and insects			Life Stages
	Top	Middle	Bottom	
18.9.78 immediately after degassing	Tribolium:1) Rhyzopertha:1) Psocids)	*Nil	Tribolium:1 Rhyzopertha:1	Adults
25. 9.78	Nil	Nil	Nil	Pupae
4.10.78	Cryptolestes 2	Nil	Nil	Larvae
13.10.78	Cryptolestes 3	Nil	Nil	Larvae
17.10.78	Nil	Nil	Nil	Eggs

* From two damaged bags on top of the stack.

TABLE 16

Fumigation of 26 bags of Turmeric with Ethyl formate under a balloon cloth: Gas concentrations and Insect Mortality - Temperature 32°C Dose: 300 g/m^3

Exposure Hrs.	Gas concn. mg/L Top	Bottom	(Interferometer)
5	Beyond scale	Beyond scale	
21	68.8	60.5	
50	37.6	38.0	
Integrated c.t. product mg.hrs/L	2690	2570	
Mortality of test insects T.castaneum	100	100	

The control of insects was 100% even in the incubated samples.

TABLE 17

Fumigation of Field Beans and Coriander

Field beans: 320 bags (100 kg each)

Coriander: 60 bags (40 kg each)

Stack volume: 65 cu.m; one side against a wall

Gas proof sheet: HDPE Woven polyethylene laminated with LDPE

Fumigant distribution: in mud pans, 6 nos. on top of the stack equally distributed before draping cover.

Dosage: 300 gm/cu.m (19.5 kg)

Exposure: 48 hrs

Gas sampling: only from middle probe

Integrated c.t. product: 2394 mg. hrs/L

100% mortality of test insects (T.castaneum adults) and immature stages in incubated samples.

TABLE 18

Gas concentrations and Mortality of Insects in Fumigating Arecanuts with Ethyl formate

Exposure: (hrs)	Location(mg/L)			Bioassay:(k.d. time Hrs)		
	Top	Middle	Bottom	Top	Middle	Bottom
5	26.4	27.2	34.9			
21	38.2	40	45.0	5	4	2.5
47	42.6	44.6	49.0	c.t:191	160	112
68	37.4	37.0	40			
Integrated c.t. product mg.hrs/L	2636	2710	3006			
Mortality of test insects T.castaneum adults (%)	50	100	98			

TABLE 19

Bio-assay of Gas Concentrations and Mortality of Test Insects in Fumigating Tapioca chips

Exposure (Hrs)	Bio-assay: Knock down time (k.d.)		
	Top	Middle	Bottom
19	5	5	12
43	40	40	17
Mortality of T.castaneum (probes)	100	100	100

TABLE 20

Fumigation of Copra Cake with Ethyl formate

(a) Gas Concentration (Interferometer): mg/L			
Exposure(Hrs)	Top	Middle	Bottom
3.7	44	37	8
22.5	80	54	36
45.5	77	61	48
69.8	65	59	55
Integrated c.t. product mg.hrs/L	5017	3989	3147

TABLE 21

Bio-assay using T.castaneum Adults: Copra Cake Fumigation

Exposure (Hrs)		Knock-down time (Hrs)	Concn. (mg/L)	c.t. product mg. hrs/L
22.5	Top	1.25	80	100
	Middle	2.75	54	148
	Bottom	24	36	864(90%* mortality)
45.5	Top	1.3	77	100
	Middle	2.25	61	137
	Bottom	2.5	48	120

* Defective sampling.

FUMIGATION TRIALS WITH A MIXTURE OF METHYL BROMIDE AND CARBON DIOXIDE IN LARGER TYPE SILO BINS

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ABSTRACT

Tests with mixtures of methyl bromide and carbon dioxide for the control of stored product pests were conducted in silo bins at three sites. Very high methyl bromide concentrations and complete kill of insects were obtained when the CO_2 was applied as a gas. Where the CO_2 was applied as snow, and where it was applied as dry ice, the methyl bromide concentrations were much lower, and there were distinct areas where very low concentrations and some insect survival occurred.

It was concluded that air was trapped under the heavy gases, and that there may still be a need for recirculation to disperse methyl bromide more uniformly and thus ensure successful fumigation.

1 INTRODUCTION

The development by Calderon and Carmi (1973) of a method to fumigate grain successfully with methyl bromide in vertical silo bins without recirculation systems, by applying it as a mixture with CO_2, has aroused considerable interest in the South African grain industry, because (i) a relatively cheap fumigant can be used, (ii) treatment can be applied to silos that lack recirculation systems, and (iii) the grain need not be turned, resulting in lower running costs for the silo, a saving of time, and less breakage of grain. However, Calderon and Carmi tested their method in bins of only 240 and 400 m^3 capacity, while many South African bins have internal volumes of over 5000m^3. Furthermore, these authors conducted their tests in bins containing wheat, while the main South African crop is maize. In this respect it is interesting to note that Philips (1957) found little difference between wheat and maize in their resistance to airflow, but hardly any gravity penetration of methyl bromide into either commodity. Calderon and Carmi's main finding was that CO_2 assisted penetration of methyl bromide to the bottom of high vertical bins containing wheat. In contrast, Smit, Nolte and Brunnekreft (1959) obtained a complete kill below 15.2 m and no significant kill in the upper 1.5 m of maize in a bin of 27.4 m height where methyl bromide alone was injected with compressed air at points 0.0; 9.1 and 18.3 m below the grain surface. Thus it seemed justified to evaluate Calderon and Carmi's method under local conditions. Three tests were conducted, the first in a relatively small silo bin of about $1000m^3$ capacity at Settlers, and the other two in silo bins of over $5000m^3$ at Driefontein and Bloekomspruit respectively. The results of these trials are presented here.

2 PROCEDURE

2.1 The Settlers Trial

The Settlers grain silo consists of two adjacent rows of nine cylindrical flat-topped concrete bins with star bins between them. The silo is equipped with a recirculation system for fumigation with methyl bromide and the bins are considered to be reasonably gastight (but see discussion). No test for gastightness was carried out and no sealing was done. The cylindrical bin in which the test was done (see Table 1) contained sorghum seed (m.c 12-12.6% wet basis), naturally infested with various pest species (Table 2). Gas sampling tubes were placed as shown in Fig. 1. Carbon dioxide was applied as a gas from pressurised steel cylinders into the head space, and methyl bromide as a liquid on the grain surface. During application, care was taken not to exceed a pressure of 2.5 kPa. in the headspace.

During fumigation, gas samples were taken at intervals, and the concentration of methyl bromide was determined subsequently in the laboratory by GLC.

Two weeks after gas introduction, grain samples from the bin were again screened for live insects. The samples were then incubated for seven weeks at 26°C and 70% RH and checked for adult insects that developed from surviving eggs and pupae.

2.2 The Driefontein Trial

The Driefontein silo consists of free-standing concrete bins with conical concrete roofs. The dimensions and contents of the bins are given in Table 1. These bins are self-emptying through three grain outlets in the bin floor. The empty test bin was superficially sealed by applying putty to obvious crevices at the grain outlets and the inspection hatches in the wall near the bottom of the bin. The manhole and grain inlet were similarly sealed after the bin was filled with maize. No test for gastightness was carried out. Maize, infested with *Tribolium castaneum* and *Cryptolestes* sp., was transferred into it from another bin. Infestation levels and grain temperatures in the first bin are summarised in Table 3. During filling, the grain flow was stopped from time to time and cages containing food and all life stages of various pest species were placed in the test bin as shown in Fig. 2. The pest species concerned are listed in Table 4. Gas sampling tubes were placed at the same time, also shown in Fig. 2. The topmost cages were about 1 m below the grain surface, which was about 4.5 m below the bin roof, as insufficient maize was available to fill the bin to capacity. The grain inlet and manhole were then sealed and CO_2 and methyl bromide were applied (Table 1). The pressure inside the bin was monitored on water manometers connected to the gas sampling tubes, and also in the headspace. Gas samples were taken 1h, 24h and 48h after treatment and the gas concentrations (CO_2 and CH_3Br) were determined in the laboratory by GLC. After 7 days, emptying of the bin was started so that the insect cages could be retrieved. This was completed in about a week and the mortality of each species in each cage was then determined. While the bin was being emptied, two 25-kg samples were taken each day from the conveyor belt and examined for surviving insects from the natural infestation. A total of 10 such samples were taken.

The average mortality of all five species of the insects that were exposed in the cages was calculated and categorized into five groups. This data was analysed by Symap computer programs (Dougenick and Sheehan, 1975) and correlated with the distribution of methyl bromide gas on two vertical planes through the centre of the bin (N-S and E-W).

2.3 The Bloekomspruit Trial

The bins at the Bloekomspruit silo are almost identical to those at Driefontein (Table 1). The test bin here was prepared in the same way as the Driefontein bin except that there were ten levels in the grain bulk at which insect cages were placed (Fig. 3) and the cages contained only malathion resistant strain of *T. castaneum* and food. Five gas sampling tubes were placed at every alternative level on a N-S line across the bin (Fig. 3). Temperature cables were placed at a few points in the upper half of the grain bulk.

CO_2 and methyl bromide were applied as shown in Table 1.

At intervals after gas application the concentration of methyl bromide was determined, using a Miran 1A infrared gas analyzer. Gas readings were again grouped into 5 categories and subjected to Symap analyses to determine the probable gas distribution on the N-S vertical plane. The last readings were taken 110h (4.5 days) after the gases were applied. Emptying was then started and continued haphazardly over 5 weeks before completed and all insect cages were retrieved. Insect mortalities were only then determined.

3 RESULTS AND DISCUSSION

3.1 Settlers

The concentrations of methyl bromide gas at the top, middle and bottom of the bin at different intervals after application are shown in Fig. 4. For most of the time, the gas concentrations increased with depth, contrary to

Calderon and Carmi's results, where the highest methyl bromide concentrations were always found near the grain surface. Our results during this test seem to agree with that of Smit *et al.* who obtained the highest kill of test insects in the bottom part of the bin. It is unlikely, however, that the differing of the contents of the various bins contributed to these contradicting results. In our test, the bin contained tightly packed sorghum with about 37% interkernel space (Jay, 1971) which must be very similar to the interkernel space of the wheat in the Israeli bins. (35.8 to 36.2% for tightly packed wheat of different varieties and moisture contents - Jones, 1943). The bin used by Smit *et al.*, on the other hand, contained flat white maize with perhaps 45 to 50% interkernel space. Smit *et al.* concluded that the heavy gas apparently sank through the grain too quickly to give a satisfactory kill at the top of the bin and in a cone-shaped space below the manhole. In their test, no CO_2 was used as a carrier for methyl bromide. In our test, comparatively high methyl bromide concentrations were found generally. At the top, where concentrations were lowest, the calculated CT-product over 192 hours was 4649g h m^{-3}, compared to 1890 (over 120h) and 2489g h m^{-3} (over 96h) recorded by Calderon and Carmi at Rehovot and Brurim respectively (average concentrations 24.2, 15.7 and 25.9 g m^{-3} methyl bromide respectively).

No surviving insects were found after fumigation, but, as will be seen later, this is not conclusive evidence that some insects might not have survived the treatment in certain parts of the grain bulk in spite of the high gas concentrations recorded at the three sampling points.

The Settlers bin was not gastight at all. Tests with a halide detector lamp revealed considerable leakage of methyl bromide underneath the concrete roof to adjacent bins and also some leakage at the grain outlet.

3.2 Driefontein

The numbers of insects at different depths in the bin from which maize was transferred for the test are given in Table 3. The heaviest infestation occurred at the grain surface. However, during transfer these insects would undoubtedly have been dispersed. The highest temperatures occurred in the centre of the grain bulk. but warmer and cooler grain would also have become mixed during the transfer operation. On the whole, the grain temperature was comparatively high for maize (which is normally around 20°C), no doubt as a result of the insect infestation.

The pressures inside the bin during treatment are given in Table 5. The build-up of pressure during this time indicates that the bin was quite gastight especially if the size of the bin and the moderate gas flow rate of 6.8 kg/min (ca. 3.7 m^3/min.) are taken into consideration.

In Table 6 the concentrations of methyl bromide and CO_2 are shown at each sampling point, 1h, 24h and 48h after application. Methyl bromide concentrations were generally much lower than at Settlers, and at some points very little gas was measured. In contrast with the Settlers result, methyl bromide concentrations tended to be lower towards the bottom of the bin. The 24-h readings were generally poorer than the 48-h readings, which indicated that it took some time for the gas to disperse through the grain. However, insect mortalities (Table 4) indicates that uniform dispersion was not reached even after 7 days, as survival occurred in some cages. The Symap printouts (Fig. 5) identify the centre of the bin as the main problem area, and to a lesser extent also the bottom. On the other hand, no survivors from the natural infestation were found in the 25-kg samples of grain that were screened after fumigation, and this suggests that some surviving insects could have gone undetected at Settlers where the numbers of naturally infesting insects were much lower than at Driefontein.

3.3 Bloekomspruit

At Bloekomspruit, methyl bromide concentrations (Table 7) closely resembled those at Driefontein (Table 6), except at the bottom of the silo where very high concentrations were measured. In this respect there is similarity with the Settlers results. The Symap printouts show that low concentrations occurred in the centre top half of the grain, and also in the entire lower half except for 3 m at the base, where a very high concentration was maintained for the full fumigation time. It was also evident from these figures that the concentration in the top half of the silo declined with time, while that in the lower half increased.

When the Symap printout of mortalities at Driefontein was studied, it was thought that the gas distribution might be influenced by an upsurge of relatively warm air at the centre of the bin in response to a cold, heavy layer of carbon dioxide and methyl bromide. Temperature probes were therefore used at Bloekomspruit to establish the role of gas temperatures, but the figures obtained (Table 8) do not appear to fit the gas distribution patterns in Fig. 6.

Generally, methyl bromide concentrations were high enough for a long enough time to give complete mortality of caged *T. castaneum* (Table 9), but low mortalities at the centre at 14 and 17 m below the grain surface confirm that very little methyl bromide reached these areas. Poor control was also obtained in more peripheral cages 20 m down, an area previously identified as a problem area.

4 CONCLUSIONS

It would seem that the results of the Israeli tests and the Settlers test are insufficient to conclusively demonstrate the successful application of this fumigation technique. Also, there appears to remain some doubt about the usefulness of CO_2 to act as a carrier for methyl bromide towards the bottom of the bin but this was not investigated during our tests and the probable benefits of adding CO_2 are not being contested.

The results of the Driefontein test and especially the Bloekomspruit test provide conclusive evidence of well-defined problem areas in the grain bulk where methyl bromide concentrations were too low to ensure complete kill. Apparently, some air was trapped below the heavy CO_2 and methyl bromide from where it appeared to escape in the form of huge, extremely slow-moving bubbles. Given sufficient time, and provided that the bin is sufficiently gastight to retain the gas long enough, methyl bromide may eventually disperse to these problem areas in sufficient quantities to ensure complete kill. However, more uniform dispersion can probably be attained much quicker by slow circulation with a fan.

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Table 1. Summary of bin dimensions, their contents, and rates of application of methyl bromide and carbon dioxide during this test series and those of previous authors.

	Settlers	Driefontein	Bloekomspruit	Rehovot*	Brurim*	Heilbron**
Bin height (m)	27.7	32.3	31.5	17	20	27.4
Bin diameter (m)	6.8	15.2	15.2	4.2	5.0	5.5
Bin volume (M^3)	$\pm$ 1000	$\pm$ 5000	$\pm$ 5000	240	400	$\pm$ 680
CO_2 Dosage (g/m^3)	250	115	150	250	250	-
CH_3Br Dosage (g/m^3)	50	50	50	50	50	64
CO_2 applied as	gas	snow	dry ice	dry ice	dry ice	-
Bins contents	sorghum	maize	maize	wheat	wheat	maize

* Calderon & Carmi, 1973

**Smit *et al.*, 1959

Table 2. The number of live insects in 10-kg samples of sorghum, taken at different depths from the bin at Settlers, before fumigation with a mixture of methyl bromide and carbon dioxide.

Depth below Grain Surface (m)	Species			
	S. zeamais	*S. granarius*	*T. castaneum*	*Cryptolestes sp.*
6.1	1	-	-	-
12.2	1	-	-	-
18.3	32	2	-	-
(27.7m)...... (Grain outlets)	248	1	10	14

Table 3. The number of live insects in 10-kg samples of maize taken at different depths, and the temperature of the grain at those points in the bin at Driefontein, before transfer to another bin and fumigation with a mixture of methyl bromide and carbon dioxide

Depth below grain surface (m)	Species		Temperature
	T. castaneum	*Cryptolestes sp.*	
0	272	1,304	28.5
3.0	4	3	24.0
6.7	7	328	25.5
10.4	1	52	31.5
14.0	3	165	31.0
17.7	2	20	28.0
Outlet valve	2	20	22.5

Table 4. The percentage mortality in insect cages at Driefontein after fumigation with a mixture of methyl bromide and carbon dioxide. Presented here, are those cases in which a complete kill of all test species was not obtained.

Sample No.	*Sitophilus granarius*	*S. zeamais*	*Tribolium spp.*	*Rhyzopertha dominica*	*Oryzaephilus surinamensis*	$\bar{x}$
B3	83.3	90.6	11.9	69.8	64.0	63.9
C3	30.8	48.8	24.8	93.1	22.2	43.9
C6	33.3	50.0	81.6	88.6	20.0	54.7
D3	46.4	57.0	5.0	88.4	24.2	44.2
D4	78.1	81.3	80.7	100	96.7	87.4
D5	59.3	61.7	91.9	100	100	82.6
D7	94.6	98.0	91.9	100	100	96.9
D7	98.0	100	98.0	100	100	99.2
D9	76.9	96.2	88.1	100	97.4	91.7
E1	48.2	41.2	31.5	100	80.9	60.4
E2	52.2	55.5	57.8	100	64.3	66.0

Table 5. The pressures (cm water) that developed at different points inside the Driefontein bin, 30 and 60 min after application of CO_2 was begun

Sampling Point No*	30 min	60 min
Headspace	12.5	17.5
1	9.7	19.7
2	9.7	20.0
3	8.0	19.7
4	7.0	16.2
5	9.6	20.0
6	8.4	19.5
7	8.4	19.5
8	7.7	17.0
9	9.5	20.0

* See Fig. 2 for position of sampling points

Table 6. Gas concentrations at 9 positions in the Driefontein bin, 1 h, 24 h and 48 h after the gases were applied

Sampling point No.*	$CH_3Br(g/m^3)$			CO_2(%)		
	1 h	24 h	48 h	1 h	24 h	48 h
1	1.8	0.4	tr	0.3	0.6	2.3
2	1.2	tr	0.2	0.7	1.1	5.9
3	-	8.9	15.7	-	1.1	2.2
4	-	0.9	11.8	tr	0.4	3.1
5	-	0.6	2.0	0.4	2.2	4.7
6	-	11.8	17.1	16.0	1.8	3.2
			13.6			3.2
7	0.8	tr	0.3	0.3	3.4	5.3
8	-	16.8	19.3	5.8	2.8	3.3
9	-	-	21.4	10.4	-	3.0

tr = trace

* See Fig. 1 for position of sampling points

Table 7. The concentration of methyl bromide at Bloekomspruit (g/m^3) at different sampling points in the grain bulk and at various intervals after application of the gas at 50 g/m^3

Metres down and sampling point No.		Hours after application of methyl bromide								
		0	6	14	19	26	37	62	86	110
5	1.......	44.5	28.5	10.4	7.7	4.8	4.7	4.4	3.1	2.3
	2.......	8.7	8.8	12.6	12.8	9.8	8.6	6.1	3.6	2.1
	3.......	0.5	0	2.6	0.2	0.2	0	0.2	0.9	1.7
	4.......	82.0	25.0	13.0	16.8	13.2	8.5	7.2	5.5	4.1
	5.......	25.0	2.5	2.7	2.7	2.1	1.0	2.4	2.1	1.1
11	1........	0.8	0	0.1	0.7	0.7	0.8	1.5	1.4	0.8
	2.......	50.0	30.0	16.4	18.8	11.6	7.7	7.1	5.8	5.0
	3.......	1.0	0	0.1	0.2	0	0	0.1	0.5	5.6
	4.......	19.2	15.2	13.8	1.2	10.8	4.8	8.8	7.2	5.7
	5.......	0.2	0	0.1	1.0	0.7	0.8	2.3	2.2	1.8
17	1.......	0.2	0	0	0.9	1.3	0.7	1.4	1.9	1.7
	2.......	0.3	0	0	0.7	2.3	1.4	4.2	2.4	1.9
	3.......	0	0	0	0.3	0	1.1	0.6	0.4	2.0
	4.......	0.2	0	0.3	1.2	0.6	1.0	3.5	3.4	3.1
	5.......	0.6	24.8	17.2	13.8	10.4	3.1	6.5	4.4	3.2

Table 7 (continued)

Metres down and sampling point No.		Hours after application of methyl bromide								
		0	6	14	19	26	37	62	86	110
23	1.......	0.1	0	0.4	1.5	0.1	2.0	2.6	2.1	1.7
	2.......	0.1	0.4	6.6	6.2	4.8	1.6	2.3	1.9	0.7
	3.......	0.1	0	4.3	8.6	10.8	3.5	12.0	5.8	4.0
	4.......	0	0	5.7	7.8	7.2	2.5	5.5	4.2	4.5
	5.......	3.7	0.1	4.5	4.3	2.3	1.6	4.3	3.6	3.1
29	1.......	14.2	82.0	88.0	64.0	55.0	55.0	55.0	45.0	28.0
	2.......	110.0	110.0	110.0	110.0	110.0	110.0	98.0	90.0	72.0
	3.......	110.0	110.0	110.0	110.0	110.0	110.0	107.0	90.0	79.0
	4.......	110.0	110.0	110.0	110.0	110.0	110.0	110.0	86.0	71.0
	5.......	18.0	110.0	108.0	86.0	72.0	75.0	52.0	29.0	21.0

Table 8. The Grain temperatures (^{o}C) measured at Bloekomspruit after application of the gases

Metres below Grain surface	Hours after application of methyl bromide											
	0	3	4	8	11	18	23	30	41	64	88	110
10............	9	27	27	29	25	22	2	10	30	13	9	9
5.............	8	27	27	27	25	20	2	10	28	14	9	9
2.............	6	24	24	23	23	18	1	9	26	12	9	9
1 (north side)	5	22	24	22	24	17	10	6	14	-2	-3	-2
1 (south side)	5	20	23	21	24	16	1	9	24	11	8	6

Table 9. Mortalities of *T. castaneum* exposed in cages at Bloekomspruit. Exposure: 110 h.

Metres down	South		Centre	North		West		East	
	1	2	3	4	5	6	7	8	9
5.........	100	100	100	100	100	100	100	100	100
8.........	100	100	100	100	100	100	100	100	100
11.........	100	100	95.0	100	100	100	100	97.0	100
14.........	100	100	4.3	100	100	100	100	100	100
17.........	100	100	9.0	100	100	69.0	100	100	100
20.........	100	100	100	100	100	100	8.3	9.3	7.3
23.........	100	100	100	100	100	100	100	100	100
26.........	100	100	100	100	100	100	100	100	100
29........	100	100	100	100	100	100	100	100	100

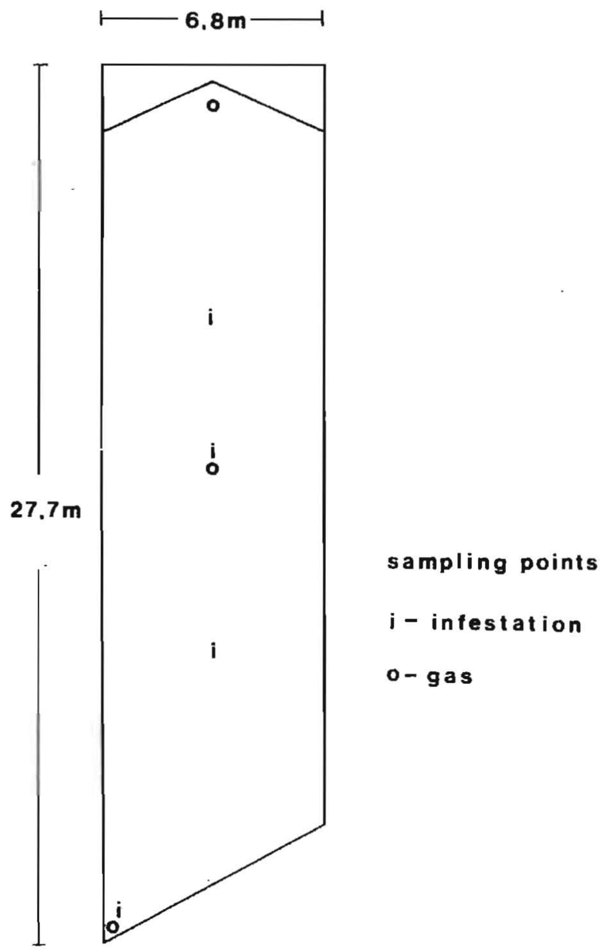


Fig. 1 The scheme of the Settlers test.

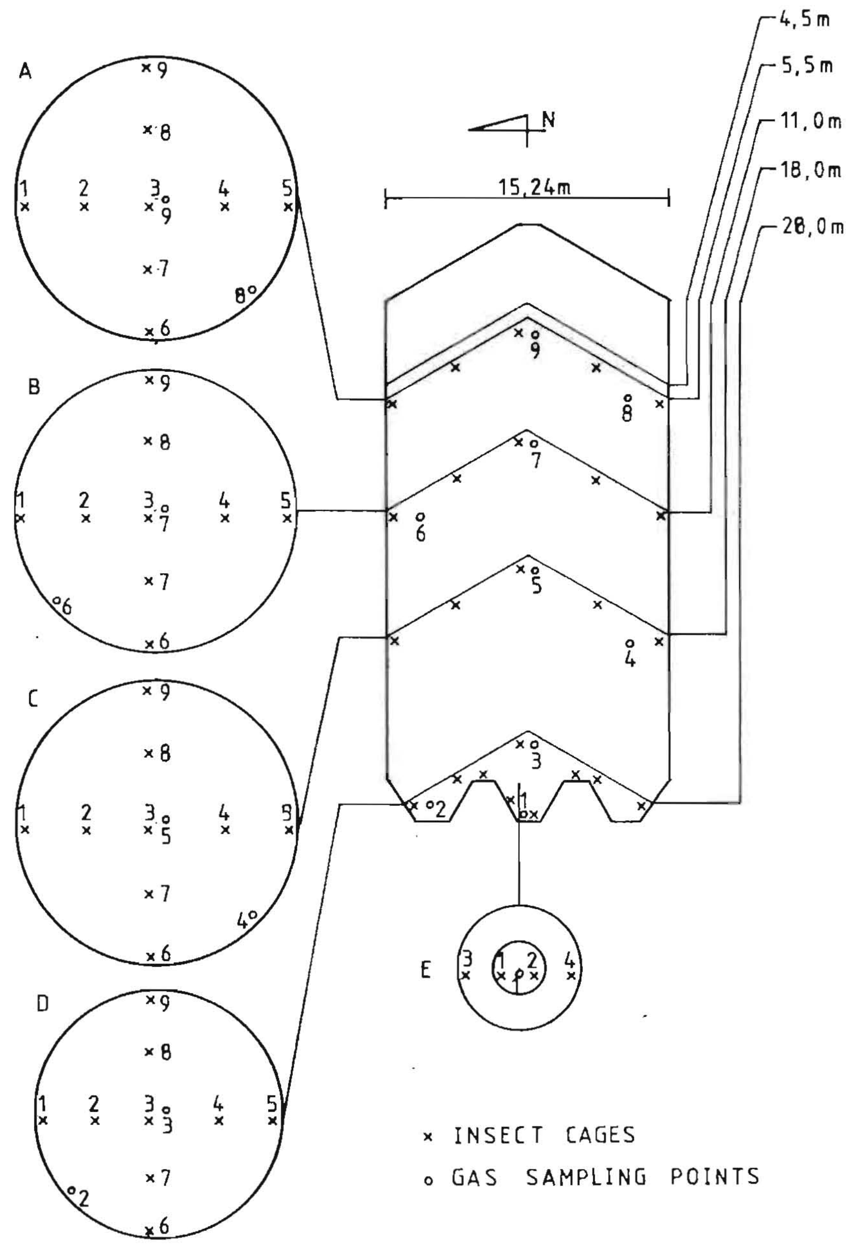


FIG. 2 THE SCHEME OF THE DRIEFONTEIN TEST.

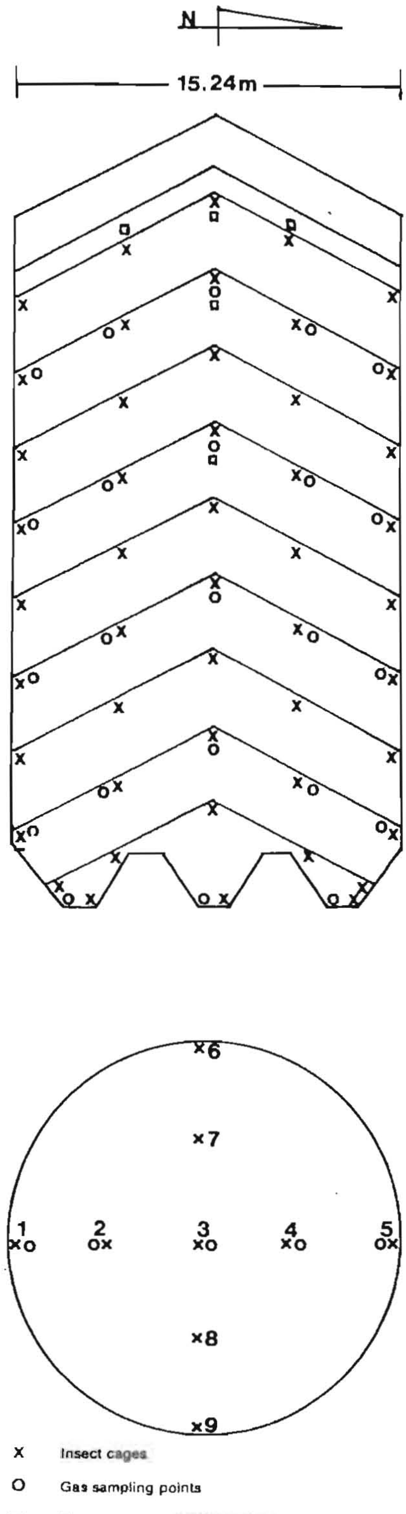


Fig. 3 The scheme of the Bloekomspruit test.

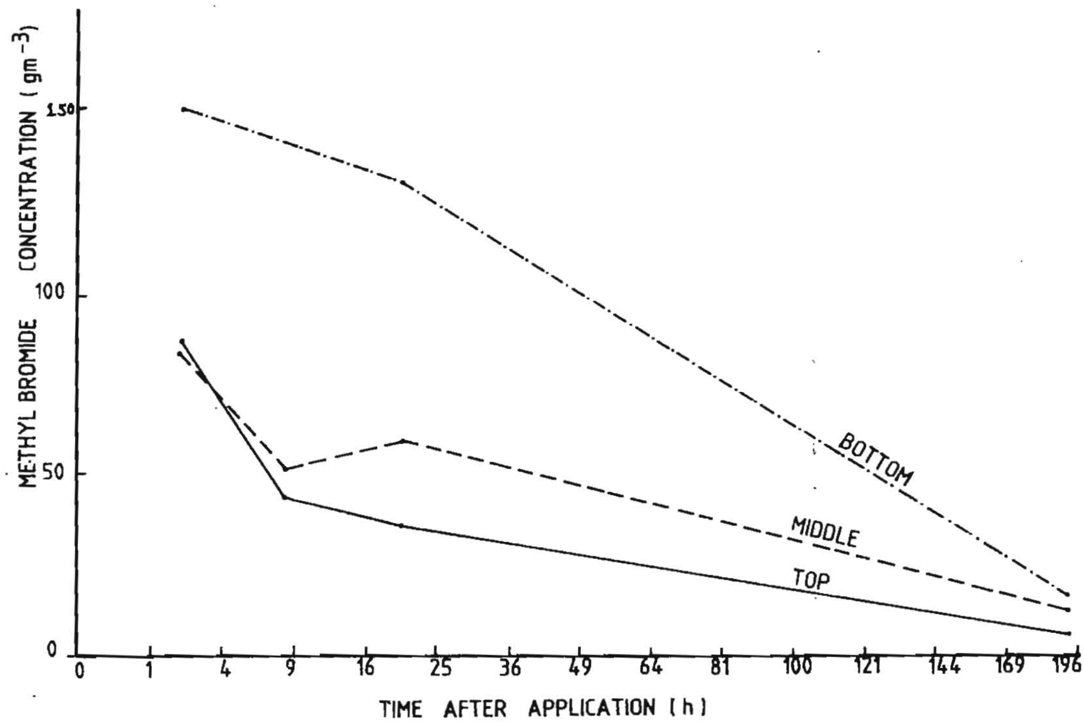


Fig. 4 The concentration of methyl-bromide at the top, middle and bottome of the Settlers bin with increase in time.

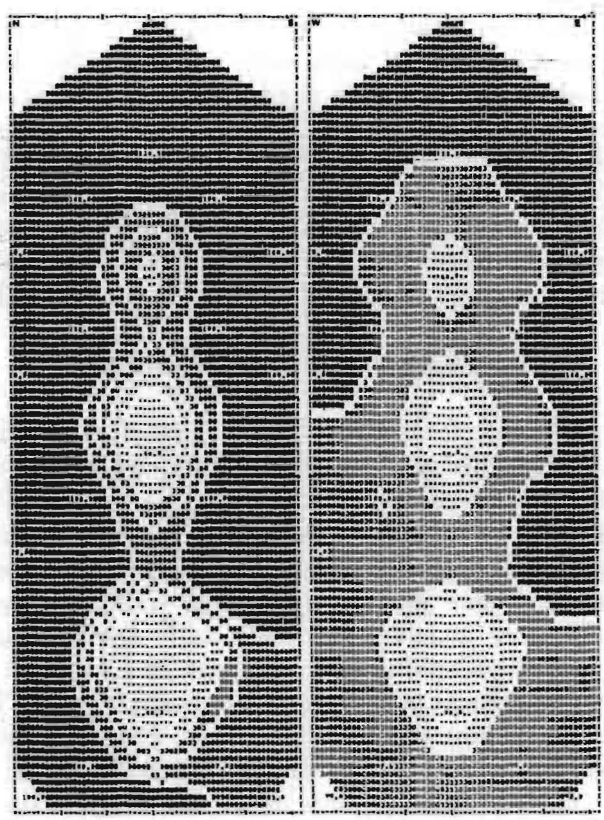
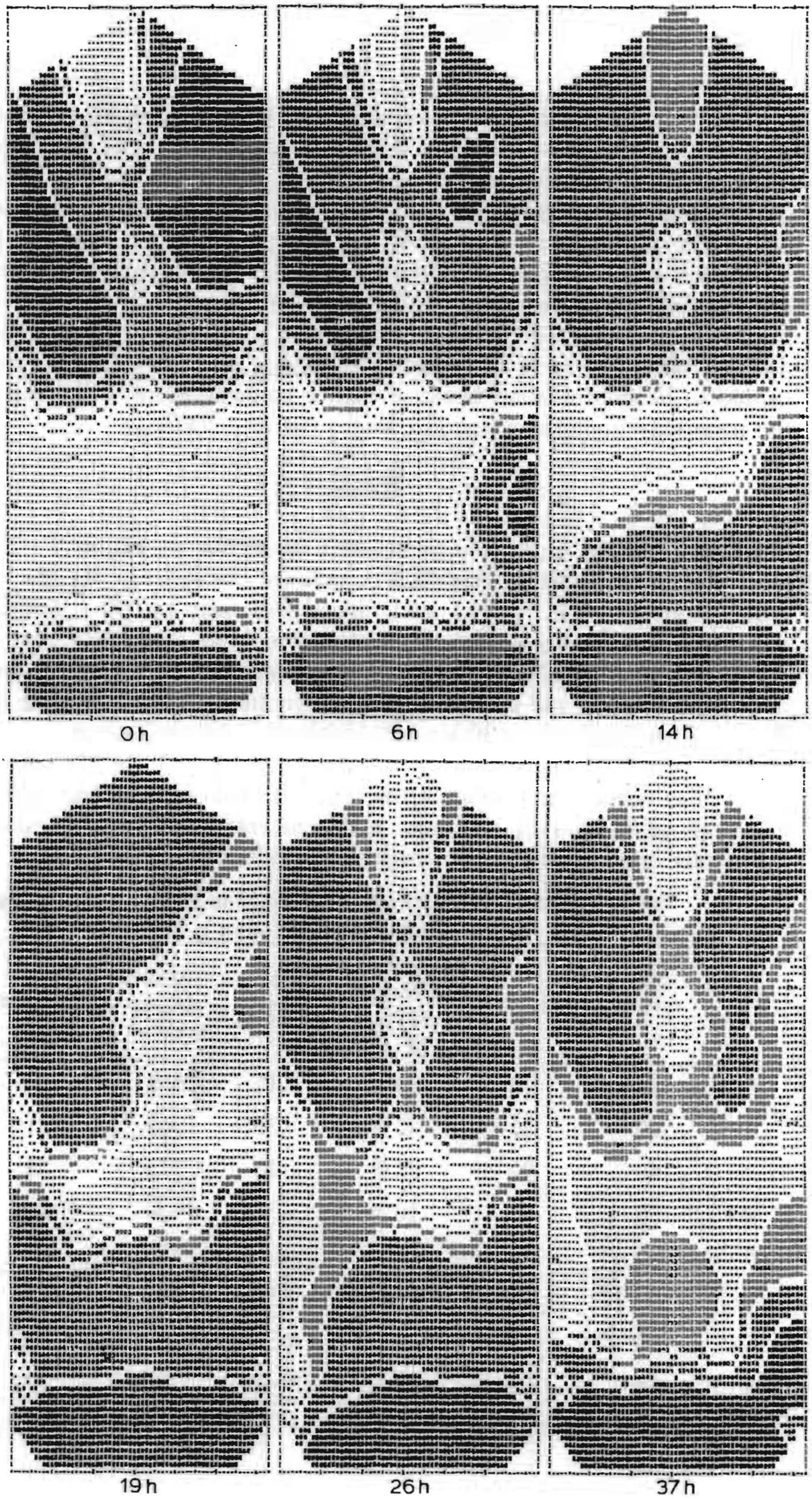


Fig. 5 The distribution of methyl bromide in the Driefontein silo as derived form insect mortalities after 7 days exposure. Darker shades represent higher concentrations (a) on the N-S plane (b) on E-W plane.


0h
6h
14h
19h
26h
37h

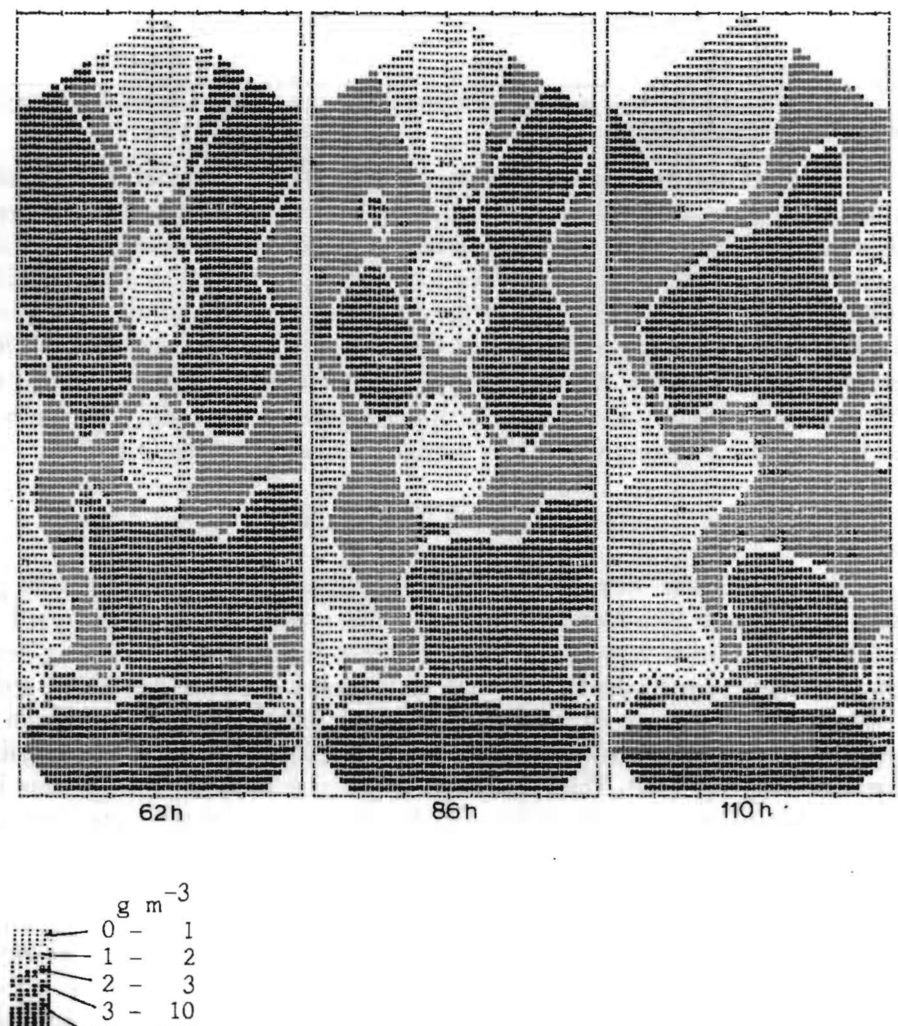


Fig. 6 The distribution of methyl bromide at intervals after application to the Bloekomspruit silo.

THE USE OF CONTROLLED AIR TO INCREASE THE EFFECTIVENESS OF FUMIGATION OF STATIONARY GRAIN STORAGES

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ABSTRACT

Fumigant recirculation was first used in the early 1920's. Nearly all fumigants have been studied for their effectiveness when applied in this manner. These studies have shown that the less sorptive fumigants are the best to use with recirculation. Many techniques are used to move the air fumigant mixture through stationary grain but the most commonly used and effective one is the recirculation push method. Fan operation time and the number of air changes required varies with each chemical employed and is dependent on the release time of the fumigant. The low air flow fumigation method was developed and patented for use with aluminium or magnesium phosphide. This system results in even penetration and distribution of hydrogen phosphide (PH_3) in grain masses of any depth or configuration. There is no danger of ignition from pressure or build-up of high concentrations of PH_3 when the system is used properly.

INTRODUCTION

Recirculation, or the use of forced air to control and move fumigants through stationary bulk grain or a farinaceous mass, has been used for over fifty years. Moffett (1927) patented a recirculation method for use with hydrocyanic acid gas. During the 1930's DEGESCH, in Frankfurt, Germany, used recirculation for methyl bromide. Phillips (1955,1957) further developed the system so that it became commercially accepted, installed, and used in many areas of the Southern U.S. Shedd (1953) and others developed information on system design and on accurately measuring the air flows within the grain mass, so that the fumigant movement, behaviour, and distribution prior to the actual fumigation could be predicted.

The controlled air or recirculation technique has been used with nearly all fumigants, including hydrocyanic acid gas, chloropicrin, ethylene oxide and carbon dioxide, and with liquid fumigants containing ethylene dibromide, ethylene dichloride, carbon disulfide, carbon tetrachloride, methyl bromide, and Phostoxin^{R} (aluminum phosphide).

Flammable or ignitable chemicals must be avoided when this recirculation technique is employed. Highly sorptive fumigants, such as hydrocyanic acid, chloropicrin, ethylene dibromide, and ethylene dichloride are not suitable for this technique. Those chemicals with low sorptive characteristics, such as methyl bromide, the liquid fumigants containing carbon bisulfide and carbon tetrachloride, and aluminum or magnesium phosphide can be recirculated with a high degree of success. Equipment design and the method of application varies with each of these chemicals.

CONVENTIONAL TECHNIQUES FOR THE USE OF CONTROLLED AIR TO MOVE FUMIGANTS

The use of controlled air for fumigants is the vehicle of conveyance for the gas and can be moved or stopped at will. Techniques such as recirculation, forced distribution or single pass, whether pushed or pulled, are simply different means of moving a controlled amount of air through a mass of grain of any depth or configuration. The fumigant is introduced by various means into the moving air, resulting in the distribution of the air/gas mixture throughout the area to be treated. The air in any of these systems can be pulled from the top to the bottom of the bin or pushed from the bottom to the top. When pulling air down through the grain, the air will tend to bend away from the walls towards the aeration ducts and negative pressures are created around the bottom perimeter of the storage which will further dilute the gas concentration in that area. Pushing the air into the bottom and up tends to move the air radically away from the ducts to the opposite walls with better distribution in the bottom perimeter and between the ducts than when the air is pulled. The overhead becomes the weak point where the fumigant concentration can be easily controlled.

Single pass fumigations can be accomplished without the use of a return pipe, but the timing of the chemical release into the air stream tends to become complicated and erratic results may be expected. Pulling the air down through the bin has some advantages since the release of the chemical can be accomplished in a controlled manner.

Recirculation using the push method is the best choice in most cases. Changing the air/gas mixture within the load from one and a half to three times provides even distribution which is characteristic of recirculation systems. Irregular warehouse loading may cause the need for six air changes in some areas to attain a single change in others. Additional blower time beyond six air changes can result in high rates of sorption of the chemical and excessive gas leakage.

The air/gas distribution is totally determined by the aeration ducts installed in the bottom or sides of the storage bins. The return pipe from the top of the bin to the blower has little to do with distribution. Its dual purpose is to return the overhead gas concentration to the system which otherwise would be wasted to the outside atmosphere and to satisfy the blower requirements. Calculations and measuring the anticipated air change can be accomplished by several methods. These air changes are timed to the particular fumigant being used. As an example, using a 0.64cm o.d. polyethylene tube, a 90.7kg cylinder of methyl bromide will empty in about 30min. Thus, the o.025m^3/min./tonne standard is used which results in a 20min. air change.

THE LOW AIR FLOW FUMIGATION METHOD

Aluminium phosphide tablets have a peak-off or maximum release of hydrogen phosphide in 19 to 30h., depending on temperature and humidity of the grain bulk. Therefore, the very slow air flow of an air change in 8 to 24h. is effective and the need for a high horsepower fan, large diameter piping and duct system are drastically reduced. When compared with the system described above for use with methyl bromide the Degesch patented Low Air Flow Fumigation Method (COOK, 1980), originally developed for use with Phostoxin accomplishes circulation needs for the aluminium/magnesium phosphides. Two types of application methods which are referred to as the "J-System" and the "J-Probe" are used. The J-System indicates hydrogen phosphide usage with an air change in 8 to 12h. The return pipe and blower can be installed into an existing aeration system or mounted on the side wall and the bottom aeration ducts can be sized to the required lower air flow. The basic principles of aeration are adhered to by keeping velocities in the pipe to 610m/min. or less.

The J-Probe employs an air displacement within 24h. which has been tested on tall silos with side wall recirculation and in ship holds loaded with bulk grain. This method has been used successfully in 37m high silos and ships filled with grain to a 15m depth. The J-Probe violates the usual principle of proper aeration by using high air velocities in small pipes. This system is being improved so that this method of distribution becomes more efficient. Table 1 presents a comparison of equipment needed for conventional aeration and recirculation with equipment required for use with the J-System.

TABLE 1

COMPARATIVE EQUIPMENT FOR A LARGE STORAGE BIN *

	PIPE ** DIAMETER (cm)	BLOWER H.P. (K.W.)	STATIC PRESSURE (PASCAL)	AIR CHANGE TIME
AERATION AND CONDITIONING	152	32 (24)	872	2.5min
RECIRCULATION METHYL BROMIDE	71	4.8 (3.6)	374	20min
J-SYSTEM PHOSTOXIN	15	0.33 (0.25)	62	8 to 12 hours

** Air velocity in the pipe restricted to 610m/min.
* Typical 3,000 to 5,000T./bin.

When deep grain depths are fumigated with aluminium phosphide by probing or broadcasting with or without plastic covering over the surface, 2000 to 4000 ppm of hydrogen phosphide (PH_3) can occur at the application or generation points. With PH_3 penetrating these grain depths at approximately 3m./day, in 3 to 5 days only 50ppm or less will be recorded at the furthest point of penetration. With the Low Air Flow Method very even distribution will occur throughout the grain mass at any depth. PH_3 Concentrations of 400 to a maximum of 1000ppm will be recorded at the point of generation and not less than 300 to 800ppm will be detected throughout the bin within 8 to 24h. after application. Table 2 presents results of the effectiveness of the J-System when compared to the conventional method of applying PH_3 in a grain bulk.

TABLE 2

COMPARATIVE RESULTS USING PHOSTOXIN TABLETS (6.6g PH_3/tonne) FUMIGATION IN GRAIN DEPTHS OF 9 to 15m.

METHOD OF APPLICATION	TIME OF EFFECTIVE PENETRATION	TOTAL EXPOSURE	RECORDED PH_3 CONCENTRATION (ppm)		
			OVERHEAD	MIDWAY	BOTTOM
1.5m Probe or Broadcast	3 to 5 days	8 to 12 days	2500	300	30
J-System	8 to 12 hours	3 to 5 days	340	330	310

If Phostoxin is properly applied, ignition from the high concentration of 1.79% (17,900 ppm) PH_3 cannot be attained. Since the J-System rarely creates more than 5 to 10cm total static pressure, measured as water, and the J-Probe

23 to 28cm, explosions from high negative pressures of 457cm cannot occur. In field studies where no standing water or condensation were present, we have found it difficult to develop PH_3 concentrations of more than 4000 to 6000 ppm.

PROCEDURES FOR USING THE LOW AIR FLOW METHOD

With a properly designed system there are no problems with application and gas distribution. The Low Air Flow Method is no panacea and it is most important to realize its capabilities and limitations. After the storage facility is sealed for fumigation and the blowers, recirculation pipe, and other equipment, are checked for proper performance, the following procedures are followed:

1. Phostoxin, preferably 3g tablets, are broadcast over the grain surface at the proper labelled dosage for the amount of grain to be treated. We do not probe the tablets into the grain nor do we cover the surface with film except for special applications.
2. Within several hours after application as determined by the temperature and humidity of the grain the blowers are activated. The air/gas mixture will be drawn from the overhead, through the return pipe down to the fan and up into the grain mass via the aeration system.
3. The gas concentration moving up through the grain mass will be slightly less than that recorded in the overhead. Since the upward air flow exceeds the ability of the gas to penetrate downwards, the last portion of the grain mass treated will be in the upper areas of the bin.
4. Distribution will be attained in 8 to 24h. After 24 to 36h. most of the aluminium phosphide tablets reduced to ash, the fans are turned off and usually no further distribution will be needed for the remaining 3 to 5 day exposure.
5. High PH_3 readings may be recorded throughout the bin within 8h. and the fans could be turned off. These readings can be misleading as the concentration will drop very rapidly within 12 to 18h. and the proper PH_3 concentrations will not be attained and maintained in the bottom two-thirds of the storage facility.
6. In very tight welded steel or concrete storages PH_3 concentrations of 30 to 50ppm may still be recorded well beyond the exposure period of 5 days. If there are no storage problems, personnel hazards, or a need to move the grain, we recommend not aerating the grain or moving it for as long as practical.
7. After the desired exposure period the treated grain can be aerated with the use of the existing system or by the same method used for other fumigants. Do not re-enter the area until it is determined by gas analyses that it is safe.

8. Distribution of the gas is not adversely affected by lower temperatures, but there will be a slower PH_3 release from the formulation.
9. The piping from the fan exhaust through and to the aeration system must be tight since the blowers must be run 24 to 30h. while the PH_3 is being released. Excessive leakage will result in loss of gas causing a possible fumigation failure and hazard to personnel.
10. Tight, welded steel or concrete storage facilities lend themselves best to these methods of fumigation with resultant reduced dosages and costs. Even so, we have installed hundreds of these systems in the less tightly sealed corrugated sheet metal buildings. However, if the storage cannot be sealed properly or where excess leakage is anticipated in the exterior aerating system, the Low Air Flow Method should not be considered.
11. In general, dosages are set to customer standards as requirements vary from country to country. The needs and requirements in the United States range from a knock down control for a quick load out to long-time storage requirements which result in almost nil infestation.
12. The normal systems which have been developed for Phostoxin application cannot be used with methyl bromide since this fumigant requires higher air flows than are produced by the J-System.

CONCLUSIONS

The Low Air Flow Method is a technique for attaining and maintaining an even distribution of an insecticidal concentration of PH_3 in a grain mass in a minimum of time. It has been proven effective in all types of storages such as country elevators, terminal elevators and in ships holds and in both vertical and horizontal structures and shows promise for the effective application of PH_3 in storages for insect control.

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PHOSPHINE FUMIGATIONS IN SILO BINS

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ABSTRACT

Wheat in tall, sealed silo bins (2,800 capacity each) was first fumigated commercially using phosphine under slow forced recirculation in 1979 in Queensland, Australia. The phosphine-generating formulation was applied to the grain surface. In most cases existing infestation was eliminated and the grain subsequently stored insect-free. The two instances where infestation was detected after treatment may have been caused by insects entering after the bins had been unsealed.

Trials were carried out using enhanced natural convection as an alternative to forced recirculation, thus avoiding possible explosion hazard through use of fans with phosphine. A large external duct, 'thermosiphon' was rigged between the bin base and apex. This was painted black to enhance the heating of the duct by the sun. The convection so produced gave good distribution of phosphine within five (5) days of application, compared with fifteen (15) days or more without the duct.

Both the forced recirculation system and the thermosiphon overcame the problems of the poor dispersion of phosphine, usually encountered in tall bins after surface application of the formulation.

USE OF PHOSPHINE

Phosphine has been widely used as a grain fumigant in Queensland for many years. In a variety of situations, it is the fumigant of preference, being cheap and easy to apply, readily removed when required by ventilation and leaving little residue on the grain. Until recently, grain stored in large silo bins was routinely treated by adding aluminium phosphide tablets to the grain stream as it was conveyed to or entered the storage bin. The phosphine was generated from the tablets at many points in the bulk. In theory, this created an even concentration phosphine throughout the storage. In practice, this was not necessarily so (Banks and Annis, in press).

This paper details some trials carried out by the State Wheat Board in Queensland to improve the distribution of phosphine obtained in silo bins compared with that given by admixture of tablets during loading and to develop ways of fumigating grain in silo bins with phosphine without the need for turning or other grain movement. It was also important to avoid the mixing of the spent preparation residues with the grain. These residues may contain appreciable quantites of unreleased phosphine and can be objectionable to workers handling grain treated by direct admixture.

The experiments described below both entail some form of recirculation to distribute phosphine through the grain mass. The phosphine was released from preparations laid on the grain surface. It was recognized that, with this form of application, natural convection is often inadequate to distribute the phosphine in tall, narrow bins (Banks and Annis, in press), but otherwise it has several advantages over admixture. These include speed of application and the ability to remove spent preparations without contaminating the grain.

The first forced recirculation treatments with phosphine were carried out in late 1979. At the time we were unaware that a very similar system was also under trial in the U.S.A. (Cook, 1980).

TREATMENTS UNDER FORCED RECIRCULATION

Methods

Treatments were carried out in concrete silo bins of 12.2 m. diameter with 29 m. high cylindrical walls, a concrete conical roof and flat base. The bins had a nominal capacity of 2,800 tonnes of wheat each. All bins were sealed so that they gave a pressure decay time of greater than 7 mins for 1500–750 Pa. decay when filled and were fitted with appropriate fans and ductwork. A tube (grain 'extractor barrel', 450 mm. diam.) led from an opening in the base of the bin wall to the centre of the bin floor. This was fitted so that an auger could be introduced for unloading, but was used as part of the gas distribution system, in these treatments.

The bins were equipped for recirculation using unslotted PVC drainage tubing (helically wire-reinforced, 50 mm. diam.) as the external duct running from close to the apex of the bin, via a blower Model 3B (Dawn: Melbourne) with 0.4 Kw. motor, to an opening in the blanking plate covering the extractor barrel outlet. The blower produced a recirculation rate of about one air change per day in the bin. The recirulation was carried out drawing the gases downward through the grain mass and returning through the external duct to the headspace.

Nine bins, containing 25,100 tonnes of wheat in total, were treated with 'Phostoxin' tablets at a rate equivalent to 0.5 gPH_3/t^{-1}. The peak of the grain bulk was levelled off. The tablets were laid in a polyethylene sheet on the level area in such a way that no tablets touched. After laying out the tablets, the apex hatch was sealed and recirulation commenced immediately.

Results

Phosphine concentration

Phosphine concentrations were measured by Drager tube at four points in the bin: in the headspace and at three points around the base of each bin (Table 1). Recirculation was discontinued after five (5) days. Phosphine concentrations were below 0.3 ppm. at all points sampled after about two months from dosing.

TABLE 1

Typical Phosphine Concentration (ppm) in a Silo Bin Under Forced Recirculation

Sampling Point	After 3-5 Days	After 11 Days
Headspace	525	100
At base by inlet for recirculation duct	475	100
At 90^{o} to this point at base	150	100
At 180^{o} to this point at base	100	100

Effectiveness of the treatment

Insects (*Tribolium castaneum* Herbst) were detected in two of the nine treated bins $4\frac{1}{2}$ months after dosing. All were known to be infested before treatment. The bins had been unsealed and inspected at 2-3 week intervals two months after dosing. There is a significant chance that the insects entered the bin through the unsealed hatches after the treatment. Their presence is thus not evidence that the treatment was unsuccessful.

All bins were retreated by the same method after five months and the three bins still containing grain were again treated after a further five months. No insects were detected on outloading nor reported by consignees receiving wheat from the treated bin.

Further use of forced recirculation

The technique was considered commercially successful, despite the occurrence of infestation on two occasions after treatment. In the 1980/81 season it was used in a further three bins. A second treatment was required in one of these bins. *Cryptolestes spp* were noted at the grain surface after more than eight months storage. Use of forced recirculation of phosphine has now been discontinued because of fears of possible explosive hazard triggered by the reduced pressures created in the recirculation fans. (Monro 1969, p.251 specifically recommends that phosphine not be subjected to recirculation).

It is hoped that the studies on phosphine explosibility currently underway in Australia (Green et al., in press) may permit development of procedures for recirculation of phosphine which are safe and thus clearing the way for use of the technique again.

THERMOSIPHON DISTRIBUTION OF PHOSPHINE

Two trials have been carried out recently to determine if natural convection can be used instead of forced distribution to disperse phosphine in tall silo bins after surface application. The use of natural convection avoids the possible explosion hazards created by fans, noted above, since the pressure differences involved are very small and are created gradually, not suddenly, as with the start-up of a fan.

Methods

The trials were conducted at Toobeah and Meandarra, Queensland in sealed concrete silo bins similar to those used for forced phosphine recirculation. At both sites, one bin was equipped with an external duct of black-painted galvanized iron (375 mm. diam.) running up the cylindrical wall of the bin. The duct was connected with flexible PVC ducting to the bin at the base through the extractor barrel plate and close to the apex through an inspection hatch. The vertical section of the duct was positioned so as to catch the sun thus heating the gases within the duct and providing gas circulation through the updraught so created. At both sites another similar bin was left unmodified and used as a 'control' bin.

Phosphine was applied as aluminium phosphine tablets (Phostoxin) to the grain surface at a rate of about 0.75 gPH_3/t^{-1} in each bin.

Gas distribution

In the control bins phosphine dispersion was slow. In both cases phosphine reached all parts of the bin sampled only after many days. In the bins equipped with the thermosiphon system, distribution was rapid. Table 2 illustrates the different rate of phosphine dispersion observed in the two types of systems. Banks and Annis (in press) have given average phosphine concentrations observed with the unmodified bin in the Meandarra trial.

TABLE 2.

Time Taken to Reach 100ppm. Phosphine at all Points Sampled

	Unmodified	With Thermosiphon
Toobeah	Not achieved after 19 days	5 days
Meandarra	15 days	5 days

The gas concentration readings showed that, with a thermosiphon system, there was a sufficient rate of circulation to provide a fairly uniform distribution of gas within two days of closing. Without the ductwork phosphine dispersed slowly downward from the surface for many days with very high concentrations (greater than 1000ppm.) maintained in the headspace and low or negligible concentrations towards the base of the bin.

The details of these trials will be presented in full elsewhere.

DISCUSSION

This paper has outlined results of trials with two systems for assisting the distribution of phosphine in tall silo bins. Both appear to be practically successful. The thermosiphon system appears well suited to isolated storage sites where maintenance of machinery may be a problem, and avoids the possible danger from use of fans with phosphine. However, if this danger can be overcome, forced recirculation may also be useful. It is easily regulated and not subject to vagaries of the weather that would influence the natural convection system. It may also achieve an even distribution more rapidly, thus possibly permitting a shorter exposure period.

ACKNOWLEDGEMENTS

The thermosiphon system for phosphine recirculation was suggested by Dr. H.J. Banks. Trials testing the system were carried out in collaboration with Dr. H.J. Banks and Mr. J.R. Wiseman of CSIRO Division of Entomology, Canberra.

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SAFETY: GRAIN DUST CONTROL IN SEALED STORAGES AND GAS CLEARING TECHNIQUES

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ABSTRACT

Introduction of a new technique in grain storage and handling may bring new, unfamiliar problems relating to safety. The use of sealed storages is one such technique. The very nature of the process, aiming to contain gases within a structure, also creates the potential for problems associated with build-up of airborne dust, retention of residual fumigant in workspaces and stresses in the storage structure which may be altered by sealing. Specific measures are required to ensure these cause no hazard or discomfort to workers. Sealed Vertical Silo bins pose few direct safety problems as workers are not normally required to work in them. They must be fitted with efficient dust extraction systems to remove dust raised during inloading and with a simple aeration system to remove gases so that personnel can gain access to the grain without hazard. Pressure relief valves must be fitted to ensure structurally dangerous air pressures are not generated, notably during outloading, in the bin.

The operational safety problems raised by sealing horizontal stores are much greater as it is often necessary for personnel to work within the store. The structure itself may be able to stand less air pressure. Particular attention is thus required to both the provision of a method of ventilation and to pressure relief systems.

It is necessary to monitor gas and dust concentrations to check that they are within acceptable limits. Exhaust gases from grain moving equipment may also build up unless properly vented.

These problems may be minimised by providing forced (or efficient natural) ventilation during grain handling or after fumigation. The ventilation systems must be designed to be sealable when not in use.

THE FLAMMABILITY LIMIT OF PURE PHOSPHINE-AIR MIXTURES AT ATMOSPHERIC PRESSURE

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Abstract: The flammability limit of pure phosphine in air at 1037 ± 14 mb total pressure with 0.39% v/v water vapour was found to be 2.1% v/v phosphine at 10°C, falling to 1.85% v/v at 50°C. The limit value increased slightly with increasing water vapour concentration. The data is discussed in terms of the mechanism and kinetics of oxidation of phosphine. It is predicted on mechanistic grounds that most gases found in grain storages will increase the flammability limit, compared with that for simple phosphine-oxygen mixtures. Ammonia may be an exception possibly lowering the limit as it could interfere with the reaction by reducing the rate of termination. It is concluded that, under conditions normally found in grain storages, temperatures of up to 80°C do not substantially increase the risk from intrinsic phosphine flammability over that at 10°C. The value of the explosion limit concentration determined in this study is consistent with the currently accepted value of 1.79% v/v in air.

INTRODUCTION

Phosphine is often used as a fumigant against insects infesting stored durable commodities such as grain. It has many of the properties desirable for a fumigant (e.g. high penetrant ability, low sorption on foodstuffs, very low residue formation). However it has one major disadvantage: it is flammable and even explosive in mixtures with air at some concentrations and pressures.

It is remarkable that the fire and explosion risks associated with phosphine are still poorly defined. This is despite a long period of use of phosphine as a fumigant, the heavy reliance on the material for pest control in many situations and the recognition that any accident in which phosphine is involved, particularly in fires or explosions, could severely affect the acceptability of the material for use.

Though it can be said that the fumigant has been used safely for treating grain in the past, recent changes in application methods for phosphine require a further assessment of the hazard. In particular phosphine-generating formulations are now sometimes applied onto the grain surface rather than distributed evenly throughout a grain bulk (e.g. Friemal, in press; Cook, in press). This method has the potential for producing much higher concentrations of the gas in the headspace of storages than normally encountered hitherto. Also forced recirculation of phosphine is being advocated (Cook, 1980), although in the past it has been recommended strongly that this not be done because of possible explosion hazard under reduced pressures (Monro, 1969 (p.251)).

The study presented here provides data on the flammability (or explosion) limits of pure phosphine-air mixtures at about 1 atm total pressure over the range of temperatures and relative humidities encountered in grain storages. The data is fundamental to assessment of flammability and explosion risks during the use of phosphine, as it provides a guide to the flammability of phosphine-air mixtures without complication from gaseous trace impurities, such as may be generated during decomposition of phosphine-releasing preparations. No reliable data has hitherto been available on the variation of the limits over the range of temperatures encountered in grain storages. The often-quoted figure of 1.79% v/v for the flammability limit of phosphine (Monro, 1969) in air is derived from a test carried out at 20°C and unspecified relative humidity with gas derived from a sachet-type preparation of aluminium phosphide (Anon., 1936). The data of Schantarovitsch (1937) and Trautz and Gabler (1929) can be extrapolated to suggest that the flammability limit might lie within the range of phosphine concentration encountered in fumigations at high temperatures (e.g. 50°C). This study was initiated specifically to check on this point since, if it proved to be correct, some procedures currently under consideration could be hazardous.

The mechanism and kinetics of gaseous oxidation of phosphine, summarised in Gmelin (1965), have received much study from Labillardière (1817) to the present. It was shown by van't Hoff (1884) that particular phosphine-oxygen mixtures were flammable or explosive below a certain pressure but were stable above the upper flammability limit (terms used here are explained in Fig. 1). This upper flammability limit can be described in terms of the minimum phosphine concentration in air or oxygen which is flammable at a fixed pressure (Fig. 1, point B'). Dalton and Hinshelwood (1929)

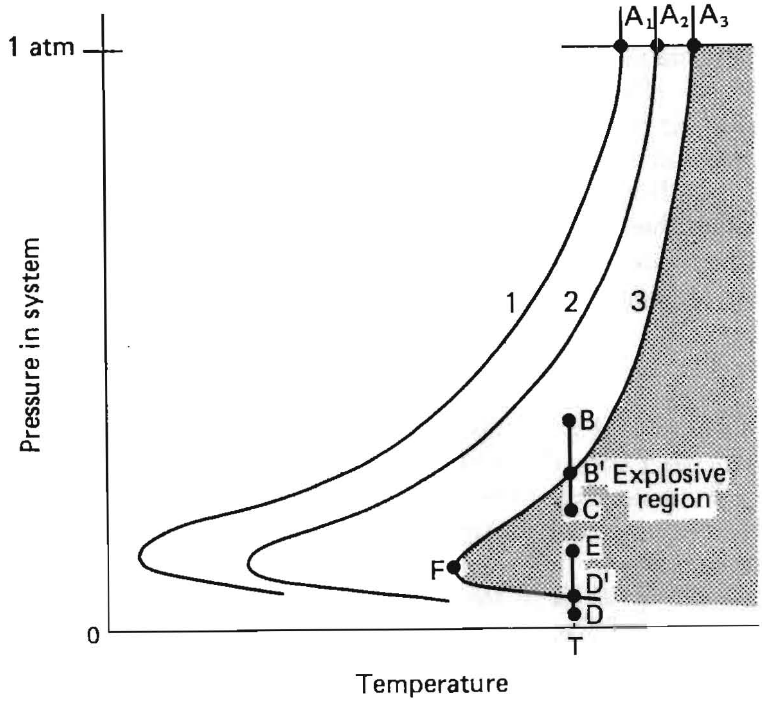

Fig. 1. Explanation of terms relating to phosphine explosibility, used in this paper. Curves 1, 2, and 3 define the boundary between the explosive and stable regions of phosphine-air mixtures for three decreasing phosphine-oxygen ratios for particular temperature system pressure combinations (adapted from curves of Schantarovitsch, 1937). Points A_1, A_2 and A_3 give the temperature at which a particular phosphine-oxygen ratio is flammable at 1 atm pressure. At temperature T and composition corresponding to curve 3, lowering the system pressure from B to C brings the system within the explosive region. Point B' defines the upper or second explosion or flammability limit. Similarly, raising the pressure from D to E produces explosive conditions, with point D' being the lower or first flammability limit. Point F, the absolute flammability limit, defines the maximum temperature at which the composition corresponding to curve 3 is stable, irrespective of system pressure.

found that there was a second critical value (Fig. 1, point D') for pressure below which a flammable mixture became stable again. The second critical value is very low (< 5 kPa) and is unlikely to be of significance in grain storage practice. With decreasing phosphine-oxygen ratios, the upper critical pressure value falls rapidly and the lower one rises, but at a slower rate (Fig. 1). These two values converge at a particular phosphine-oxygen ratio (Fig. 1, point F). This composition, equivalent to about 0.11% v/v phosphine in air at 15°C, the highest temperature studied (Dalton, 1930;

Schantarovitsch, 1937), defines the absolute flammability limit. Mixtures at less than this phosphine-oxygen ratio are not flammable irrespective of pressure.

We assume here that it is unnecessary to restrict the phosphine concentration in grain stores to below this absolute limit since mixtures at this composition are only flammable at low pressure (approx. 10 kPa, estimated from Schantarovitsch (1937) with allowance for the presence of nitrogen in air). However, to avoid possible explosion risks, we assume that the phosphine-oxygen ratio in the atmosphere of grain storages should not exceed the upper flammability limit at normal atmospheric pressure and, if reduced pressures are to be encountered, the composition of the atmosphere must not be such that it is brought into an explosive range by the reduction in pressure. The latter problem is currently under investigation. The upper flammability limit at normal atmospheric pressure is estimated here.

MECHANISM OF PHOSPHINE OXIDATION

A mechanism has been proposed (Norrish and Oldershaw, 1961) for the oxygen-phosphine reaction system under flash photolysis that is consistent with the observations of Dalton (1930), Melville and Roxburgh (1934) and others. The postulated mechanism is a chain reaction, involving free radicals in the following steps:

$O_2 + PH_2\cdot \longrightarrow HPO + OH\cdot$	Propagation
$OH\cdot + PH_3 \longrightarrow PH_2\cdot + H_2O$	
$O_2 + PH_2\cdot \longrightarrow PH + HO_2\cdot$	
$O_2 + PH \longrightarrow HPO + O$	
$O + PH_3 \longrightarrow PH_2\cdot + OH\cdot$	Branching
$O + O_2 + M \longrightarrow O_3 + M$	Termination
Radical + Wall $\longrightarrow$ Compound	
$HPO + O_2 \longrightarrow HPO_3$	Secondary Reactions
$HPO_3 + H_2O \longrightarrow H_3PO_4$	

The condition for flammability or explosion occurs when the rate of branching exceeds the rate of termination. Similar mechanisms can be expected for any process producing free radicals in a phosphine-oxygen or -air mixture. The initiation process studied in this work, spark ignition, gives a localised source of radicals and can be taken as similar to that which might occur in grain storages (electrostatic discharge, electrical faults) at atmospheric pressure.

In this reaction scheme there is only one branching reaction. Consequently the rate of branching, r_b, can be defined as

$$r_b = k_1 . P_{PH_3} . P_O \tag{1}$$

where k_1 is the foreward rate constant and P_{PH3} and P_O are the partial pressures of phosphine and oxygen atoms. Since it has been shown by Dalton (1930), that wall effects are negligible at the upper explosion limit, the termination rate, r_t, is given by

$$r_t = \sum_M k_2{}^M . P_O . P_{O_2} . P_M \tag{2}$$

where P_{O2} and P_M are the partial pressures of oxygen molecules and the third body M, and $k_2{}^M$ is the rate constant for this termolecular collision involving M. The flammability limit, where the rate of branching is equal to the termination rate, is therefore defined as

$$k_1 . P_{PH_3} = \sum_M k_2{}^M . P_{O_2} . P_M \tag{3}$$

The Equation (3) is analogous to that derived for the upper limit by Norris and Oldershaw (1961). At the limit the concentration of radicals is generally small compared with the stable species present and thus termination by collision between two radicals may be ignored. For damp air, Equation (3) may be then written as

$$k_1 . P_{PH_3} = k_2{}^{O_2} . P_{O_2} . P_{O_2} + k_2{}^{N_2} . P_{N_2} . P_{O_2} + k_2{}^{PH_3} . P_{PH_3} . P_{O_2} + k_2{}^{H_2O} . P_{H_2O} . P_{O_2} \tag{4}$$

where P_i is the partial pressure of the species i. The temperature dependence of the rate constant, $k_2{}^i$, is given by

$$k_2{}^i = r_i \mathrm{e}^{E_2{}^i/T} \tag{5}$$

where r_i is the termolecular collision rate of species i, and for k_1

$$k_1 = r_b \mathrm{e}^{E_1/T} \tag{6}$$

where T is the absolute temperature and E_n the activation energy for the nth process.

The presence of gases which cannot give rise to additional radicals, thus enhancing the branching reaction rate, will inhibit the reaction by increasing the terminating termolecular reaction rate.

EXPERIMENTAL

A schematic diagram of the apparatus used for the determination of flammability limits is shown in Fig. 2. The explosion chamber and mixing apparatus were placed inside an environmental chamber which controlled the temperature and water content of the experiment. An explosion chamber of approximately 8L capacity was filled with a mixture of air and water vapour of known composition from the controlled environment chamber. After closing the valves to the environmental chamber and mixing apparatus, the temperature of the explosion chamber was raised to the experimental temperature. This procedure permitted observations to be made over a range of relative humidities and water concentrations.

Phosphine was generated by the action of water on phosphonium iodide. This method is known to give phosphine of high purity (Fluck and Novobilsky, 1969). The gas was stored over water in a glass bell jar. The phosphine was transferred by a pneumatically operated syringe from the storage jar to the explosion chamber. Mixing was helped by a low velocity recirculating fan in the explosion chamber. Preliminary experiments had shown that the gases were well mixed ($\pm$ 5%) after about 1 minute. The temperature and pressure of the experiment were sensed by a thermocouple and pressure transducer in the explosion chamber. A high capacity spark was used to ignite the mixture to ensure that the limit was not dependent on the ignition energy supplied. If the mixture did not ignite additional phosphine was added, the mixture was then sampled by syringe for gas composition analysis, the spark was applied again and the process repeated until flame propagation occurred. Ignition was indicated by a white mist rolling through the explosion chamber. The rate of temperature and pressure rise observed and the delay in ignition were used as qualitative measures of how close the mixture strength was to the flammability limit. Ignition near the limit occurred only after a long delay, up to two minutes, and with a

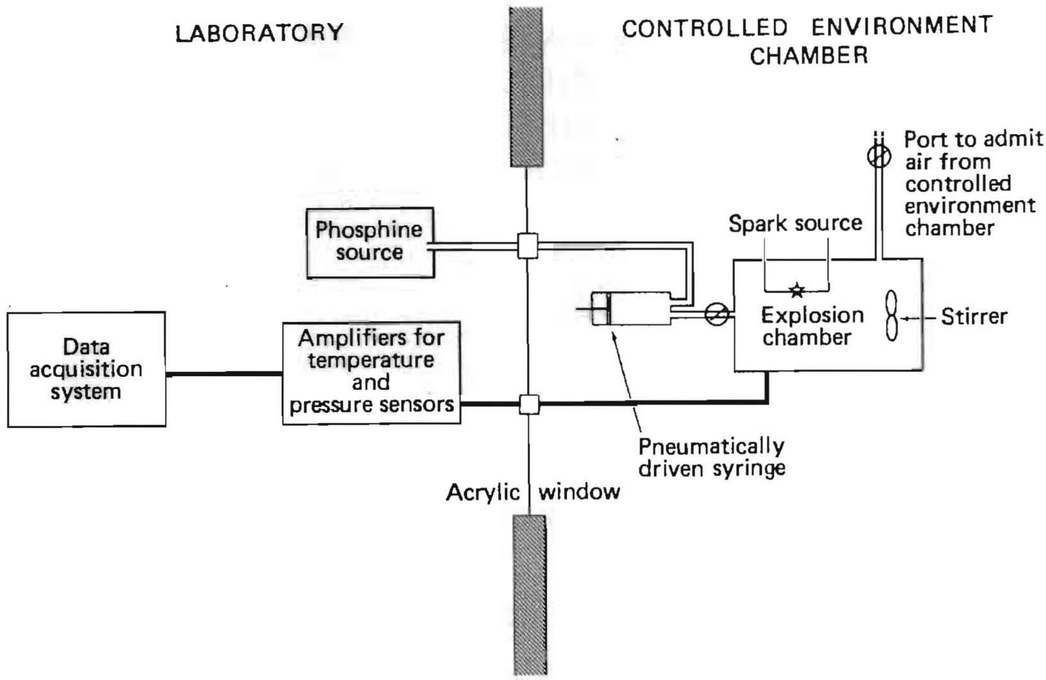

Fig. 2. Schematic diagram of apparatus used to determine the flammability limit.

small rise in temperature and pressure, while an explosion well above the limit gave a rapid and strong reaction.

Gas samples for analysis were taken from the region surrounding the spark electrode prior to applying the spark to the gas mixture. The gas samples were analysed for nitrogen, oxygen and phosphine. The water concentration was not estimated directly, but was controlled by adjusting the water content in the air of the environmental chamber and thus the water concentration in the air admitted to the explosion chamber prior to introduction of the phosphine.

RESULTS AND DISCUSSION

Determination of the flammability limit

The compositions of the gas mixtures in experiments showing flame propagation, together with those having the highest phosphine concentrations that did not result in flame propagation, are shown in Table 1.

The phosphine-oxygen ratios investigated that just did or just did not give an explosion are shown in Fig. 3 as a function of temperature at each of three water concentrations. The flammability limit lies between the points of explosion and no explosion, at each temperature. Although the gas analysis system will give concentrations to an accuracy of 1%, local variations within the explosion chamber lead to an estimated uncertainty of about 5%. The error bars given in Fig. 3 represent this 5% uncertainty.

Calculation of the forward rate constant for oxidation

Equation (4), the equation for the flammability limit, may be written in an alternative form involving the collision frequencies relative to those of oxygen for the termolecular reaction:

$$k_1 . P_{PH_3} = k_2^{O_2} . P_{O_2}(P_{O_2} + aP_{PH_3} + bP_{H_2O}) + k_2^{N_2} . P_{O_2} . P_{N_2} \quad (7)$$

where a and b are the relative collision frequencies for phosphine and water respectively. Arnold and Comes (1979) have accurately measured the parameters required for calculation of the reaction rates $k_2^{O_2}$ and $k_2^{N_2}$ according to Equation (5). These parameter values, given in Table 2, have been used to calculate these rate constants and thus the forward rate constant, k_1, according to Equation (7). The rates for other third bodies have not been explicitly measured although relative collision frequencies at 300K can be inferred for some species. The available data for chemical species of interest in phosphine flammability are collated in Table 3.

In calculating k_1, a value of a = 2.5 has been assumed. This value is similar to that given by Dalton (1930) and Kassel (1932). Values of k_1 and b for this work were estimated from the variation of ln k_1 against $1/T$. The best overall fit was obtained with a value of b = 3 (Fig. 4), giving the rate constant k_1 value and temperature dependence shown in Table 4. The maximum and minimum deviations are also plotted in Fig. 4. The phosphine-oxygen ratio for the calculated limit is shown in Fig. 3 using these values.

Comparison with literature data on flammability

The value of k_1 determined in this work with those obtained by similar calculations using data in the literature are compared in Table 4. The values of k_1 at 300K and the likely uncertainty

TABLE 1.

Composition of near limit mixtures of phosphine, air and water vapour.

T°C	Total Pressure (mb)	Mole fraction				% r.h.	Occurrence of Explosion	Quality of Explosion
		N_2	O_2	PH_3	H_2O			
12.4	1036	0.78	0.20	0.0188	0.0036	25	No	
12.3	1039	0.77	0.20	0.0253	0.0036	25	Yes	Violent (some way beyond limit)
11.4	1049	0.79	0.21	0.0204	0.0040	30	No	
11.3	1048	0.77	0.21	0.0214	0.0040	30	Yes	Very mild (close to limit)
28.9	1043	0.77	0.21	0.0173	0.0040	11	No	
28.9	1043	0.77	0.21	0.0209	0.0040	11	Yes	Very mild (close to limit)
50.2	1047	0.77	0.20	0.0180	0.0039	3	No	
50.0	1048	0.77	0.20	0.0200	0.0039	3	Yes	Mild
19.6	1032	0.78	0.19	0.0197	0.0037	16	No	
20.9	1033	0.77	0.20	0.0184	0.0037	15	Yes	Very mild (very close to limit)
40.7	1035	0.78	0.20	0.0155	0.0040	5	No	
40.8	1035	0.77	0.20	0.0186	0.0040	5	Yes	Mild
25.2	1031	0.77	0.21	0.0189	0.0040	13	No	
24.7	1031	0.77	0.21	0.0215	0.0040	13	Yes	Strong
20.3	1023	0.76	0.20	0.0205	0.0187	80	No	
20.3	1023	0.75	0.20	0.0293	0.0187	80	Yes	Very severe (some way beyond limit)
29.8	1028	0.76	0.20	0.0156	0.0206	50	No	
29.9	1025	0.76	0.20	0.0176	0.0206	50	Yes	Mild
40.2	1039	0.76	0.21	0.0198	0.0197	27	No	
39.6	1037	0.76	0.21	0.0188	0.0197	27	Yes	Mild
49.4	1047	0.76	0.20	0.0159	0.0211	17	No	
50.3	1049	0.76	0.20	0.0183	0.0211	17	Yes	Very mild
29.9	1027	0.74	0.20	0.0184	0.0390	94	No	
29.8	1027	0.74	0.20	0.0201	0.0390	94	Yes	Very mild
40.0	1039	0.74	0.20	0.0190	0.0408	56	No	
40.2	1039	0.74	0.19	0.0207	0.0408	55	Yes	Strong
49.9	1051	0.74	0.20	0.0209	0.0370	31	No	
49.8	1049	0.75	0.20	0.0193	0.0370	31	Yes	Mild

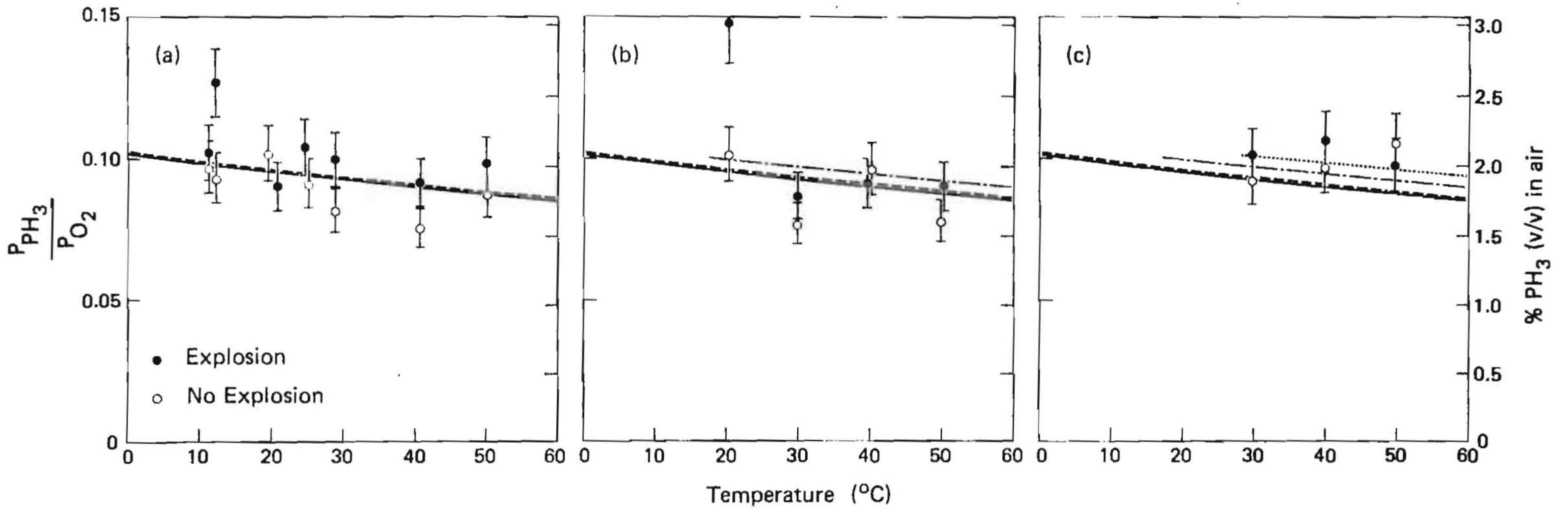

Fig. 3. Variation in flammability of phosphine-air mixtures with temperature at about (a) 0.4%, (b) 2% and (c) 4% v/v water vapour with calculated trends for the dry mixture (———) and with 0.39 (-----), 2.0 (—-—) and 3.9 (............)% water vapour.

TABLE 2.

Reaction rates for the formation of ozone (Arnold and Comes, 1979).

Gas (M)	r_2^M cm^6 $molec.^{-2}$ s^{-1}	E_2^M K^{-1}
O_2	$(6.75 \pm 0.43) \times 10^{-35}$	635 ± 18
N_2	$(1.82 \pm 0.23) \times 10^{-35}$	995 ± 37

TABLE 3.

Collision frequencies relative to oxygen at 300K estimated from various data sources

Gas (M)	Frequency relative to oxygen	Reference
O_2	1	
N_2	0.88	Baulch *et al.* (1972)
	0.85	Arnold and Comes (1979)
PH_3	2.7	Dalton (1930)
	2.1	Kassel (1932)
	0.5	Trautz and Gabler (1929)
	4	Schantarovitsch (1937)
H_2O	8.8	Baulch *et al.* (1972)
	4	Trautz and Gabler (1929)
	3	This work
CO_2	3.8	Baulch *et al.* (1972)
O	3	Baulch *et al.* (1972)
O_3	3.0	Baulch *et al.* (1972)

factor are also given.

It is notable that the rate constants predicted from earlier work are generally some two orders of magnitude lower at 300K than we find here. There are a number of possible reasons for this difference. Most factors tend to provide a lower apparent value of k_1. The literature data was obtained for relatively high phosphine concentrations and often with pressures below 8 kPa. The constants a and b will be temperature dependent. The early work was mainly concerned with phosphine-oxygen ratios above 0.1 and the

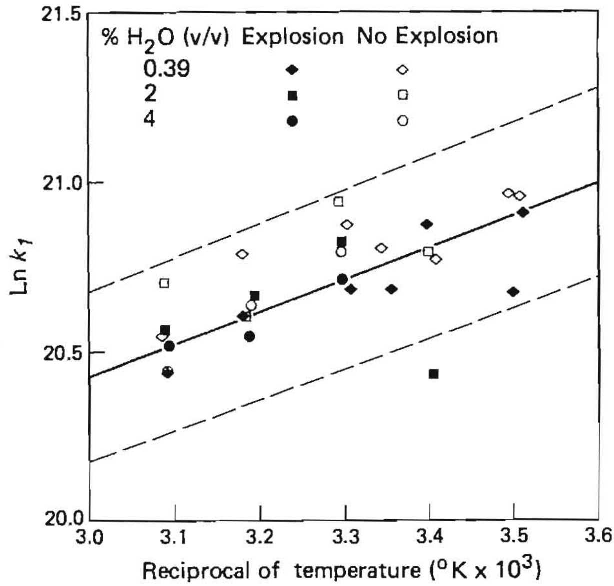

Fig. 4. Dependence of the rate constant for the branching reaction on temperature.

reported limits were those observed by reducing the pressure in the system until explosion occured. These limits are indicative of the auto-ignition point and will depend on the initiation process and are unlikely to be lower than the spark-induced flammability limits.

There is a possibility that another reaction becomes important at higher pressures. The initiation process involves the formation of an H atom. It is possible for this radical to cause branching through the following reaction

$$O_2 + H^\bullet \dashrightarrow O + OH^\bullet .$$

This reaction is not very likely at low pressures (Dalton, 1930) as the hydrogen radical diffuses to the wall too quickly. However at higher concentrations of phosphine and higher total pressures this reaction is possible. The hydrogen atom concentration is dependent on the dissociation of phosphine to give $PH_2^\bullet$ and $H^\bullet$. The use of a spark ignition source or photolysis facilitates this process, leading to higher hydrogen atom concentrations, produced locally in the former case and throughout the system in the latter. The additional term to the rate of branching would be

$$k_3 . k_4 . P_{PH_3} . P_{O_2} \tag{8}$$

where k_3 is the forward rate constant for the above reaction and

k_4 is the rate constant of the dissociation. This term is the same form as the termolecular termination involving phosphine. The net result would be an additional variation in the value of a with pressure and temperature.

The reasons for the differences between the flammability observed in sparkand auto-ignition experiments is currently under investigation.

Effect of other gases on flammability limit

The presence of water vapour reduces the flammability of the mixture. Although the data is consistent with the expected termolecular termination, additional branching reactions involving water could take place:

$$H_2O + O \dashrightarrow OH^{\cdot} + OH^{\cdot}$$

$$PH + H_2O \dashrightarrow PH_2^{\cdot} + OH^{\cdot}$$

The first reaction is unlikely to be important as the rate constant at 300K is some 10 magnitudes (Baulch *et al.*, 1972) smaller than the reaction of O with $PH_2^{\cdot}$. We do not have sufficient data to assess conclusively the possibility that the second reaction occurs. The rate constant for this reaction has a complex dependence on the rates of propagation of the chain reaction, but since the flammability limit falls with increasing $[H_2O]$, these reactions are unlikely to be significant.

Commercial phosphine-releasing agents release other gases in addition to phosphine. In general these gases would tend to reduce the flammability of a phosphine air mixture due to the species acting as an inert gas in the termolecular collision process. However, products which release ammonia could lower the flammability limit via the reaction (Olszyna and Heicklen, 1972):

$$NH_3 + O \dashrightarrow NH_2^{\cdot} + OH^{\cdot},$$

a reaction analogous to the branching reaction of the reaction scheme of Norrish and Oldershaw (1961).

Implications for phosphine fumigation

The data presented in Fig. 3 shows that there is only a slight effect of temperature on the explosion limit for phosphine in air. By extrapolation, it can be expected that the explosion limit will be > 1.5% v/v even at 100°C. Limited extrapolation of this kind is reasonable as the kinetic model used here is well founded and the observations show a linear trend when transformed as for Fig. 4.

TABLE 4.

Estimated value and temperature dependence of k_1, the rate constant for the branching reaction.

Ignition source	k_1 $cm^3\ molec^{-1}\ s^{-1}$	k_1 at 300K $cm^3\ molec^{-1}\ s^{-1}$	Uncertainty factor	Reference
Spark	$(5.9 \pm 0.6) \times 10^{-15} \exp\left(\frac{950 \pm 50}{T}\right)$	1.4×10^{-13}	10	This work
Auto-ignition under reducing pressure	$7.2 \times 10^{-11} \exp\left(\frac{-2590}{T}\right)$	1.3×10^{-15}	200	Schantarovitsch (1937)
	$2.5 \times 10^{-11} \exp\left(\frac{-2800}{T}\right)$	2.2×10^{-15}	200	Trautz and Gabler (1929)
		4.4×10^{-15} ; 1.49×10^{-15}	10	Dalton (1930)
		4.6×10^{-15}	200	Kassel (1932)

TABLE 5.

The explosion limits for phosphine-air mixtures at 1 atm pressure and 0.39% v/v water vapour at various temperatures

Temperature (°C)	Phosphine concentration % v/v	g m^{-3}
10	2.10	31.9
20	2.04	31.0
30	1.98	30.1
40	1.92	29.1
50	1.85	28.2
60*	1.80	22.3
70*	1.74	26.4
80*	1.68	25.5

* extrapolated values on the basis of the relationship shown in Fig. 4.

In most fumigations, as now carried out, the concentration of phosphine is unlikely to exceed the flammability limit values given in Table 5, except possibly in the vicinity of a phosphine-generating preparation. However in fumigation carried out where the added preparations are concentrated in a restricted space, such as the headspace of a well-filled vertical cell, it may be possible to exceed those levels in a significant volume of the storage, with the possible hazard from ignition. Clearly such situations are to be avoided either by restricting the total quantity added or by ensuring by some means that there is an adequate rate of gas dispersion from the region in which the preparation is placed so that build-up of a hazardous concentration is prevented.

The upper flammability limit, measured here, refers to that in a free space, such as a storage headspace or in ductwork. In a filled grain storage, much of the storage atmosphere is within the bulk. The upper limit is not sensitive to the size of the reaction vessel in which it is measured, at least down to less than 4 mm for the smallest dimension of the vessel (Dalton 1930). This scale is similar to the distance between some grains. Thus it is possible that the flammability limit in grain will not differ substantially from that in a free space. However, inasmuch as wall effects become significant in very small spaces and the temperature rise during propagation of a flame is inhibited by grain, the upper limit within grain will not be lower than in free space and the free space value can thus be used as a conservative estimate for the total storage atmosphere for safety assessments.

This study has dealt with the limits of flammability of pure phosphine-air mixtures. Further work is in hand to determine the sensitivity of the upper explosion limit to total pressure and to determine experimentally the influence, if any, that gaseous trace components (e.g. diphosphine), generated from phosphine-generating formulations, may have on the limit.

ACKNOWLEDGEMENTS

This study was financed by the Bulk Grain Handling Authorities through the Australian Wheat Board and carried out under the auspices of the Coordinating Committee on Silo Sealants. We are grateful to Dr D.J. Webley of the Australian Wheat Board for his assistance and encouragement in this project and to Dr C. Reichmuth of the Institut fur Vorratsschutz, Berlin, for locating the original work citing the explosion limit of 1.79%. Drs A.R. Gilby and T.E.Bellas criticised the draft manuscript.

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TECHNIQUES FOR ANALYSING FUMIGANTS AT ULTRA-LOW CONCENTRATION LEVELS

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INTRODUCTION

Contamination of atmospheres with toxic gases has created problems of great concern to environmental and health authorities. New techniques have revealed the presence of materials that were previously unsuspected and new knowledge on the harmful effects of toxic compounds has pointed up hazards that were not known to exist. The advent of new industrial processes with the consequent proliferation of toxic contaminants has emphasized the need for sensitive instruments and methods that can easily analyse low concentrations of chemicals in atmospheres. For toxic compounds like fumigants, where substantial quantities may be periodically injected into atmospheres and where the general public is already apprehensive of the dangers from pesticides, the need is urgent.

Analysis of gases from systems where a high degree of dilution occurs (as in contaminated atmospheric air) has the inherent problem of separating the small amounts of the contaminants from massive quantities of other compounds i.e. nitrogen and oxygen. The contaminants then must be retained in a way that will be suitable for analysis by some appropriate technique.

A method for concentrating such diluted gases has been developed and used to measure, by gas chromatography, very low concentrations of fumigants (Dumas 1978, 1982, Dumas and Bond 1981). Low levels of the residual gas desorbed from fumigated grain have been thus collected and analysed. The method is also useful for low levels of contaminating gases in atmospheric air.

The procedures for collecting and concentrating fumigants, along with methods for analysing ultra low levels of the gases, are described here.

CONCENTRATING GASES FROM DILUTE SAMPLES

To collect the contaminating gas from the atmosphere a trapping device somewhat similar to the column in a gas chromatograph was used.

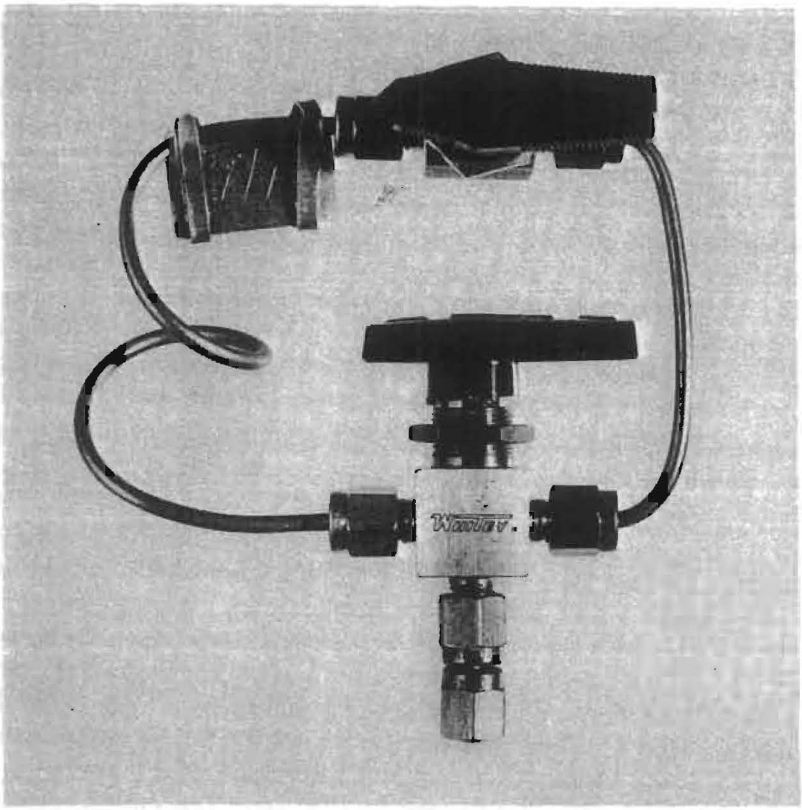

Fig. 1. Trapping device designed for collection and analysis of samples by gas chromatography.

A short length of stainless steel tubing was packed with appropriate sorptive materials and fitted with three way valves plus connections for incorporation in the gas flow system of a GLC. Selection of the sorptive packing material was achieved by cooling the trap with dry ice while it was installed in the GLC and determining the ability of the packing to retain the test gas.

Atmospheric contaminants were collected by passing known volumes of the diluted samples through the trap while it was held in a small foam insulated box filled with dry ice. For analysis the trap was then inserted in the GLC, allowed to warm up to column temperature and the stopcocks were then opened to allow carrier gas to move the sample through the column to the detector.

When contaminated air was under test a syringe was used to obtain and inject the desired quantity. For collection of gas that desorbed from fumigated commodities an appropriate amount of the commodity was retained in a gas-tight container for a specified period of time and then a measured volume of nitrogen was used to flush the desorbed gas from the container and into the trap.

This method was found to be suitable and reliable for collection of ultra-low concentrations of methyl bromide and phosphine, both from samples in air and samples desorbed from fumigated cereals (Dumas 1980, 1982). When wheat treated with phosphine or methyl bromide was analysed for desorbing residues of the gases, levels down to ppt (10^{-12}g) could be detected and measured. Further investigation of the procedure showed that low levels of gases could also be collected at ambient temperatures with a satisfactory degree of accuracy. Consequently the versatility of the method was expanded and its utility for application under field conditions increased.

ANALYSIS OF COLLECTED GAS SAMPLES

Three detectors were found to have the required sensitivity for analysing the low levels of fumigants that were collected by the trapping technique. A flame ionization detector was used for methyl bromide analysis and an alkali flame ionization detector for phosphine. A photoionization detector in a portable GLC (Photovac 10A10) operating at ambient temperature was found to be effectual for analysis of four fumigants under field conditions at levels down to ppb (10^{-9}g), (Bond and Dumas 1982, Dumas and Bond 1982). This detector was also found to be useful for the detection and analysis of low concentrations of formaldehyde (Dumas 1982). Analysis were accomplished by direct injection of 1 ml samples of the gas-air mixtures into the instrument. For lower concentrations a special valve can be supplied with this instrument to incorporate the trapping technique described above and thus further increase its analytical capability.

Details of the above methods have been published in the journals cited below. While the potential capacity for analysing ultra-low levels of gases has been advanced by using the principles and instrumentation described here considerable scope for future development still remains. Several facets of modern technology might be exploited to accommodate the increasing needs for sensitive and precise analytical methods for fumigants.

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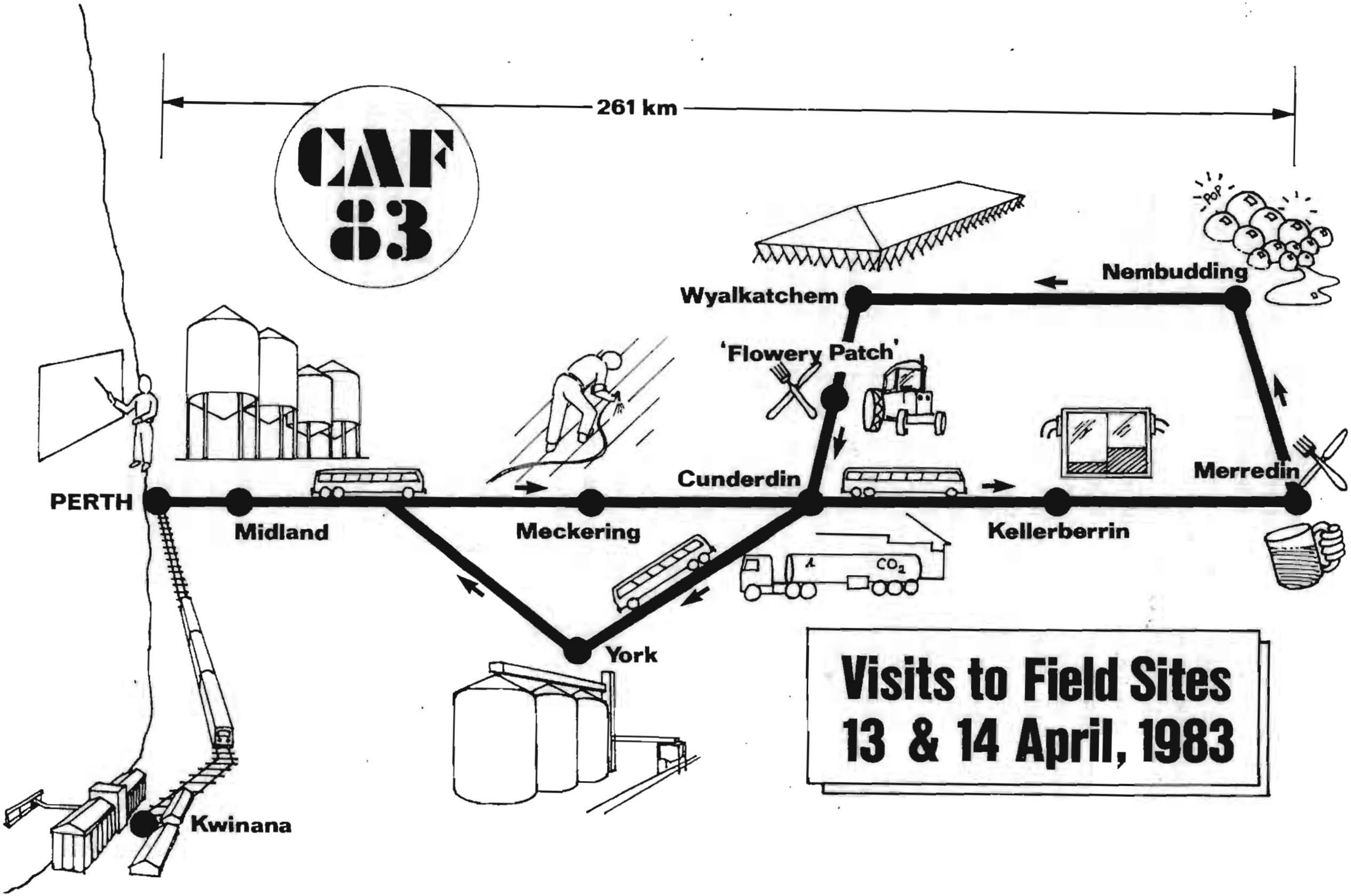
PRACTICAL DEMONSTRATION TOUR OF COUNTRY SITES

The sites visited for the practical demonstrations were selected to provide sequential story of Controlled Atmosphere for the sealing process through to purging with carbon dioxide.

Midland	Farm Silo Expo - the smallest of storages sealed.
Meckering	An average Central Storage System grain store set up to provide an exploded view of progression through the sealing process.
Kellerberrin	Conducting a pressure test on a sealed storage.
Merredin	Overnight stop and viewing a large storage and grain handling equipment.
Membudding	Leak detection.
Wyalkatchem	A less cost storage to cater for above average production.
"Flowery Patch"	A typical broad acre farm. Lunch stop.
Cunderdin	Purging with CO_2
York	Vertical steel storage, sealed during construction.
Kwinana	The largest of the storages sealed and fitted for Controlled Atmosphere.


261 km
CAF 83
Wyalkatchem
Nembudding
POP
'Flowery Patch'
Cunderdin
Merredin
PERTH
Midland
Meckering
Kellerberrin
CO_2
York
Kwinana
Visits to Field Sites
13 & 14 April, 1983

MIDLAND EXPO - ON-FARM TYPE STORAGES USED FOR DEMONSTRATION PURPOSES

This location is the site of 13 on-farm type storages (1 fibreglass the rest steel), each of about 16 tonnes wheat capacity, and established with the co-operation of resident silo manufacturers, to demonstrate to farmers the effectiveness of sealing their seed and feed grain storages when introducing a fumigant (usually phosphine) for total insect control. The demonstrations included the introduction of generated colored smoke - to simulate a fumigant - into both sealed and unsealed silos. This dramatically showed the loss of fumigant to be expected from an unsealed storage and the retention of the gas in a sealed storage. The influence of the weather conditions - sun and wind - and the chimney stack effect of sucking the smoke out of the unsealed storage - was clearly shown as was the internal pressure variations caused by natural heating and cooling. A pressure of 200 pascals was introduced into a sealed silo by use of a standard type vacuum cleaner and the time noted for decay to half life to determine the effectiveness of the seal. The pressure relief vents attached to each silo were explained. For details of on-farm silo sealing refer to Papers 45 and 46.

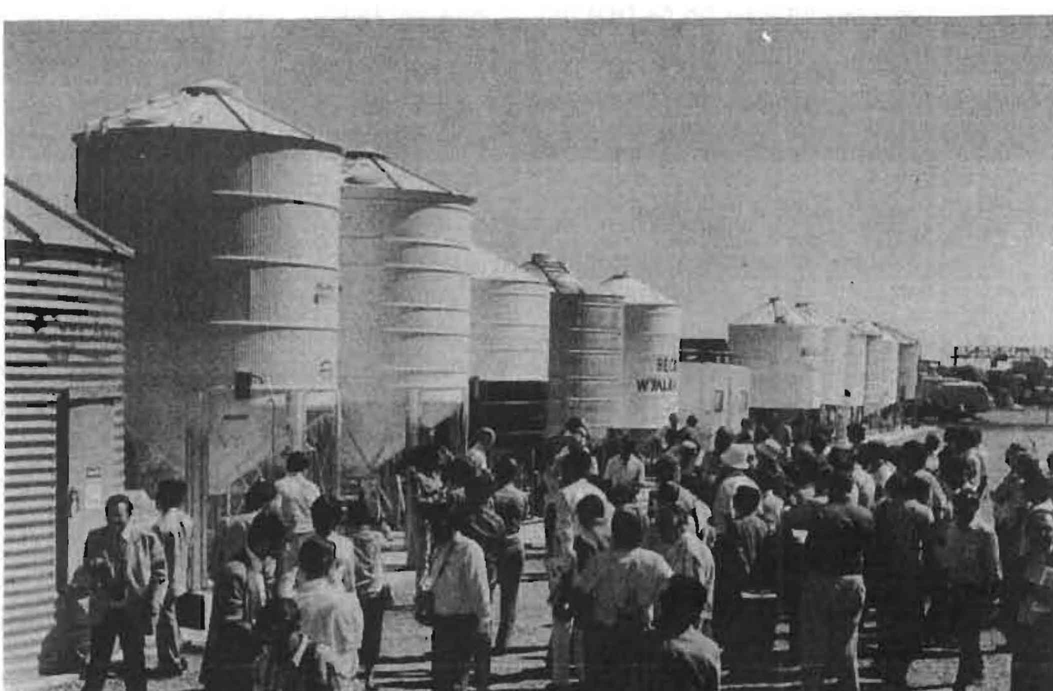


Photo 1. General View of "Midland Expo".

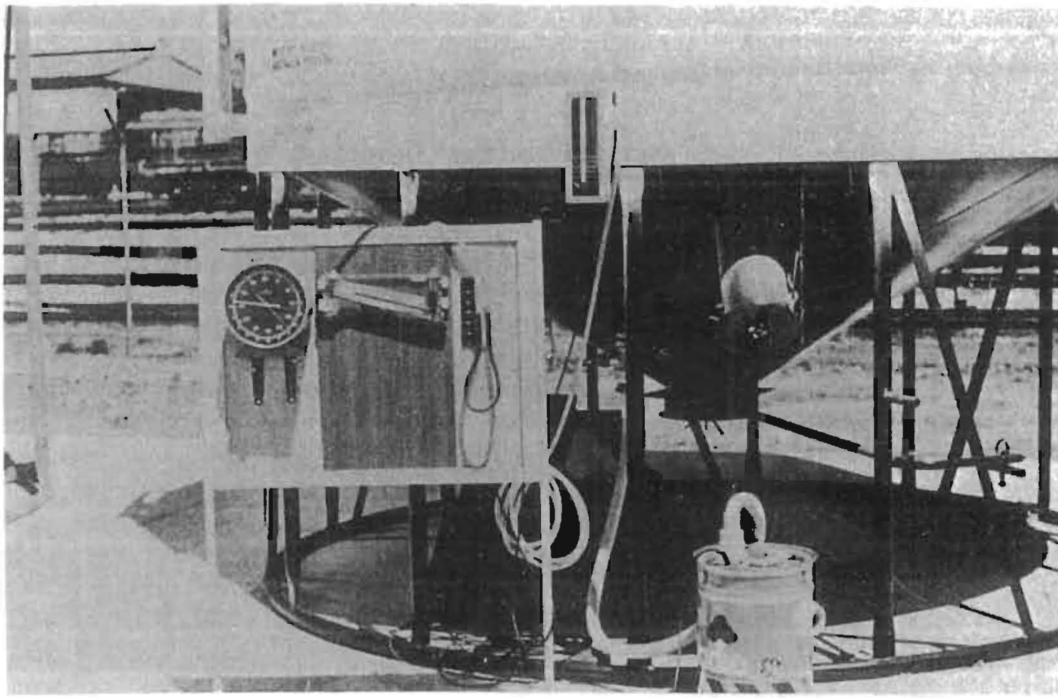


Photo 2. Sealed Farm Silo showing manometer and pressure relief vent.

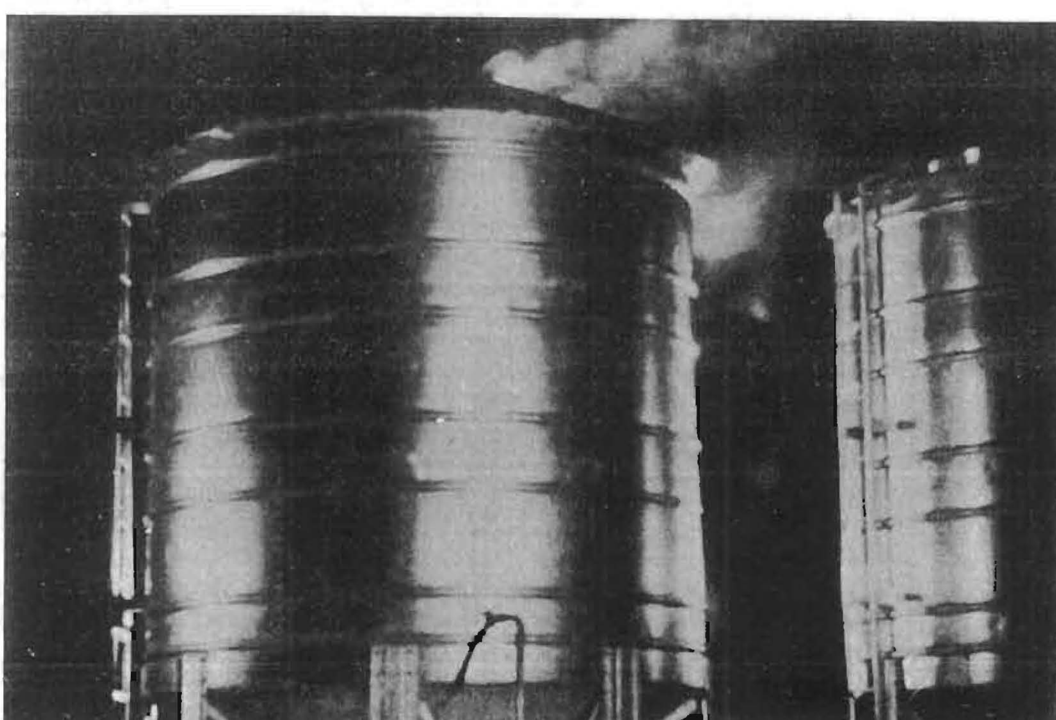


Photo 3. Leakage of fumigant (smoke) from an unsealed farm silo.

MECKERING : HORIZONTAL STORAGE IN PROCESS OF BEING SEALED

The storage visited was a standard 'C' type horizontal silo of 22,000 tonnes wheat capacity, constructed with concrete walls and floors and having a clear-span trussed roof of corrugated galvanised iron cladding. Gable-end walls and side curtain-walls are also of corrugated iron. Of particular interest was the application of rigid polyurethane foam over the internal laps of the steel sheeting and along the gaps formed between the curtain and gable-end walls and the roof and concrete walls of the building. Foam was also applied along the roof ridge areas, around the internal perimeter of doorways, around the fans in each of the end gable walls and the entry point for the overhead conveyors. The spray application of the internal wall sealant was also demonstrated. Other features pointed out were the methods of external sealing of the roof cladding sheets, attention to the concrete floor, door sealing and the final top coat of a heat reflectant material sprayed over all external surfaces. For details of sealing procedures refer to Papers 11, 12 and 13.

Photo 4. Sealing in progress on the end wall of a horizontal silo at Meckering. Note all lap ends of the corrugated iron sheets used to clad the gable-end wall are individually sealed, as are surface cracks evident in the concrete wall.

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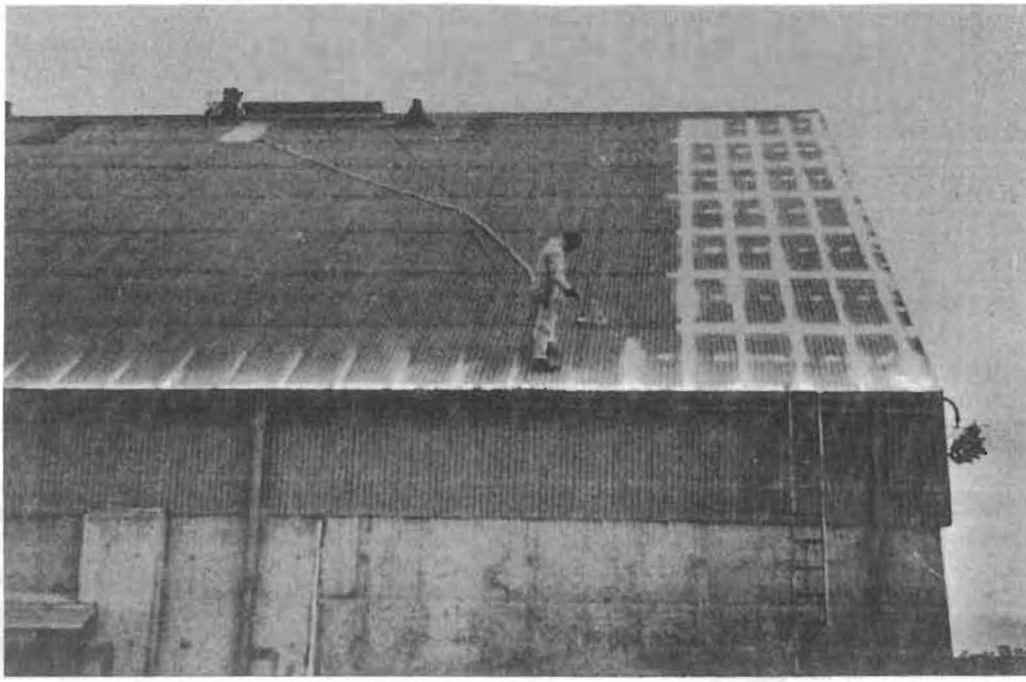

Photo 5. Spray sealing of all lap ends of corrugated iron roof sheets on horizontal silo at Meckering.

Photo 6. Application of final coat of reflectant material over previously sealed roof and wall at Meckering.

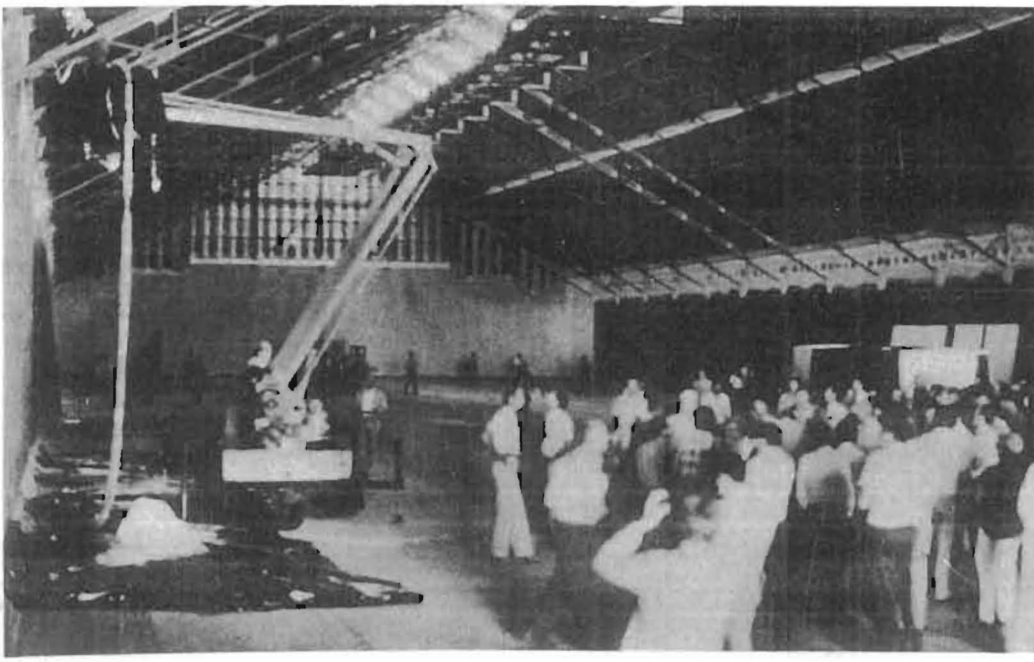

Photo 7. Interior view of Meckering horizontal storage showing applications of polyurethane foam to the roof/wall joints.

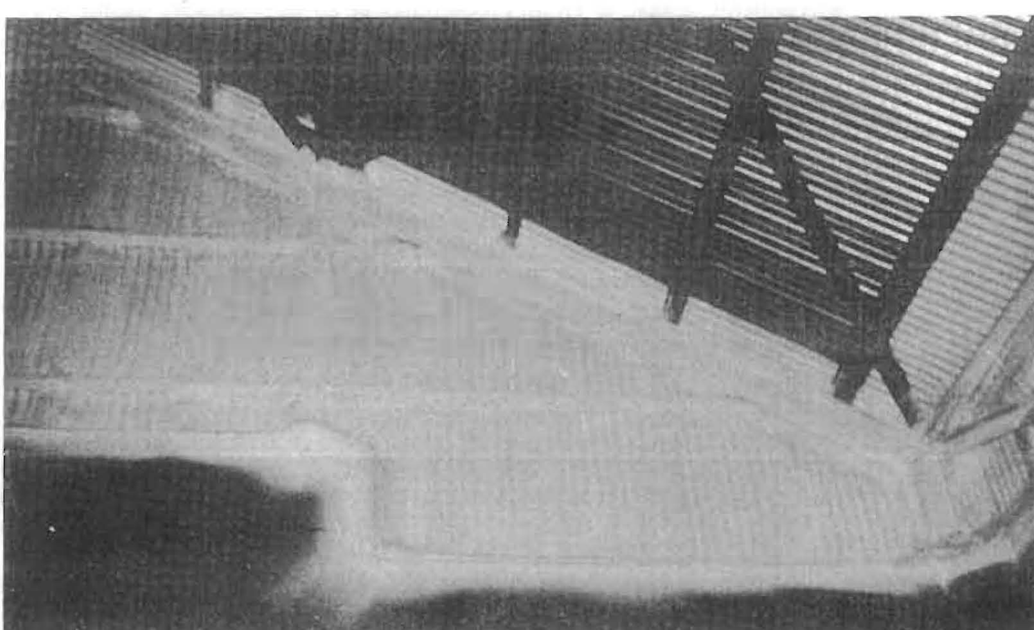

Photo 8. View of a gable end wall at the Meckering horizontal Silo after foaming of all joints and spray sealing of the finishing coat.

Photo 9. Application of foam inside a horizontal storage.

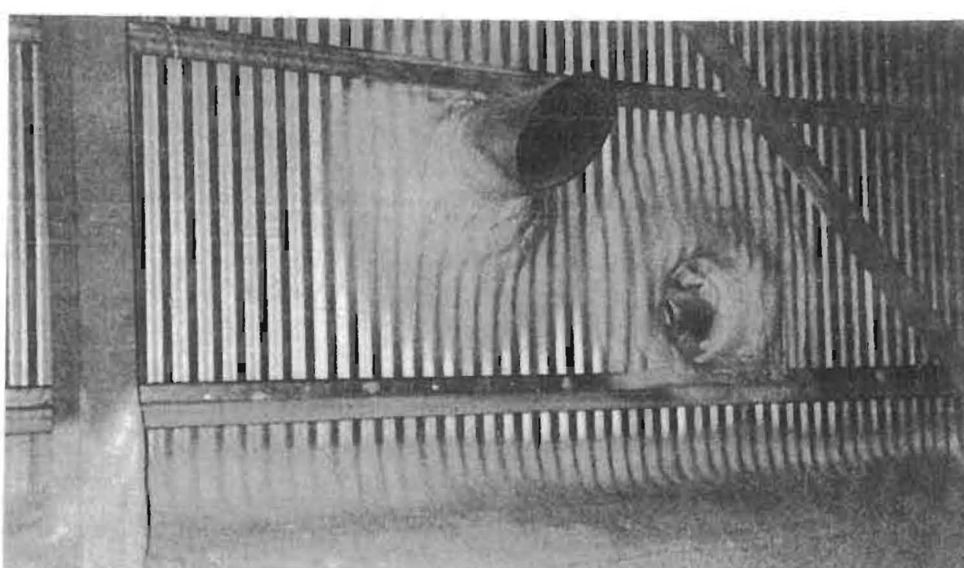

Photo 10. Foam application along an inside wall ledge and around the fan and pressure release duct fitted into a gable and wall.

Photo 11. Sealing inside the roof ridge line of a horizontal storage.

KELLERBERRIN : PRESSURE TEST ON A SEALED STORAGE

The demonstration at this site was of a pressure test of a completely sealed storage - a standard 'A' type horizontal storage of 27,500 tonnes wheat capacity. Before starting the test, the naturally induced pressure inside the storage, shown by the positive pressure on the intake side of the pressure relief vent, was pointed out. This positive pressure is noted during the warming of the internal air spaces by the sun and a reverse negative pressure will occur during the cooling down phase.

One gable end fan was started and a pressure of 200 pascals introduced into the storage, noting the increasing pressure on a manometer and by the imbalance of the light oil in the chambers of the pressure relief vent. After turning off the fan, and resealing the fan aperture, the retention of pressure was clearly demonstrated through an almost negligible decay noted on both the manometer and pressure relief vent over a period of 30 minutes.

At the conclusion of this test a main silo entry door was opened to physically show by sound and feel the force of the pressurised internal air escaping to the external atmosphere. This force was strong enough to blow a piece of heavy cloth material at right angles to the door opening. For details of pressure effects on grain storages refer to Papers 16 and 21.

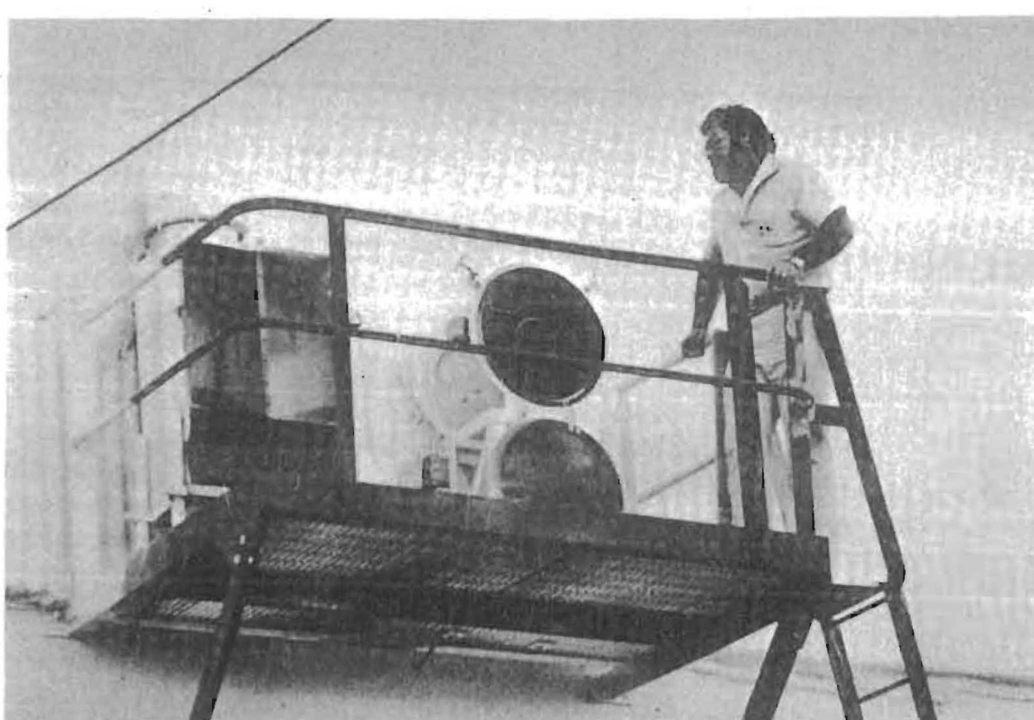

Photo 12. Pressure relief vent and fan built into gable end wall of horizontal storage at Kellerberrin. The fan cover is opened while pressurising the storage and is sealed for the pressure decay and leak detection test.

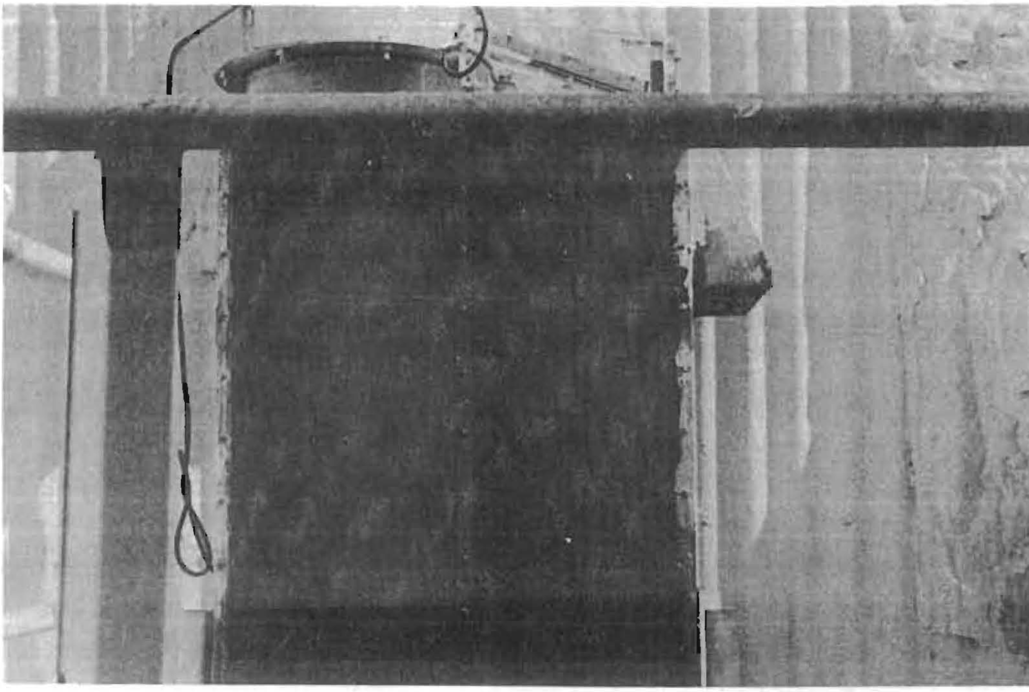

Photo 13. Kellerberrin storage under pressure indicated by difference in fluid level in the pressure vent.

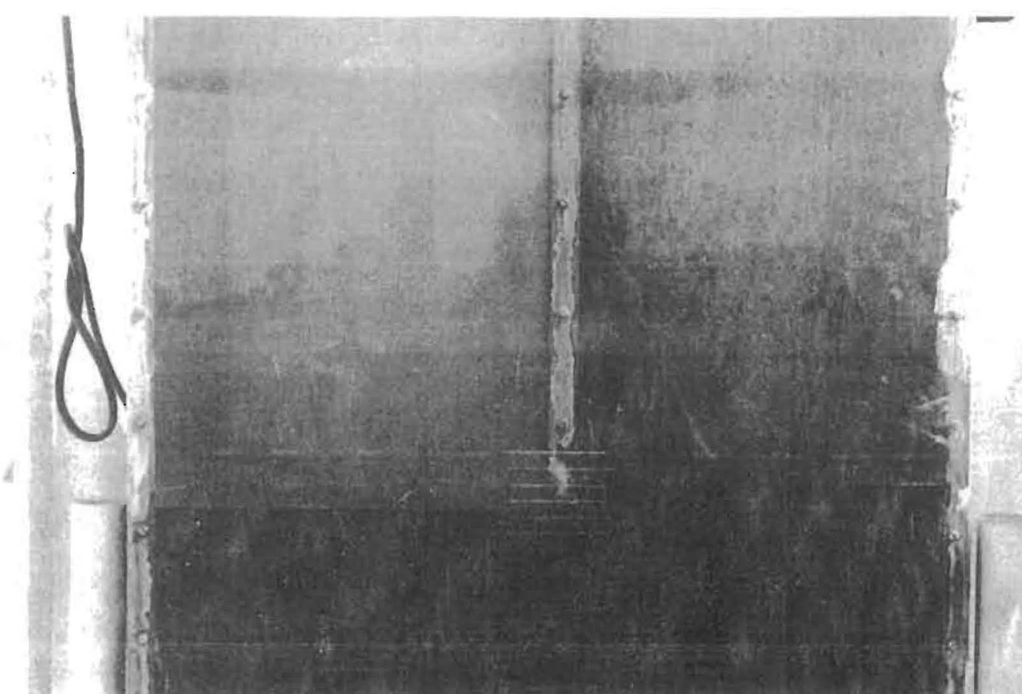

Photo 14. Close-up of a typical pressure relief vent fitted to a horizontal storage.

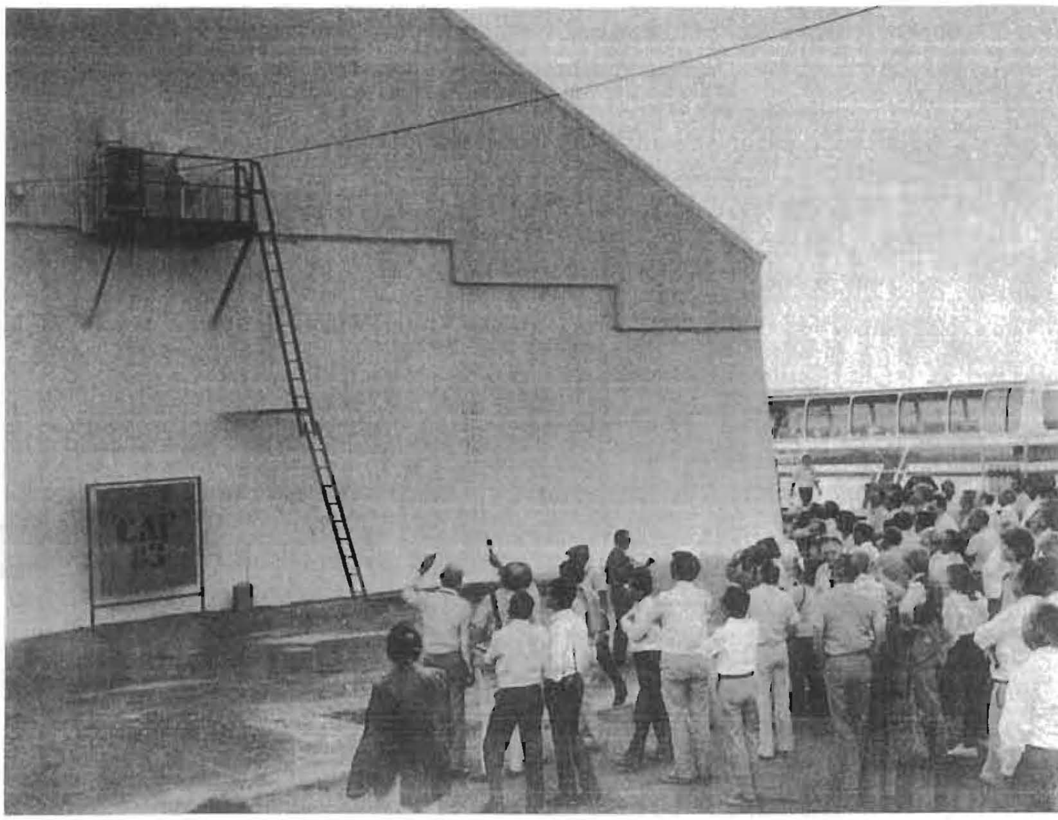

Photo 15. Symposium delegates inspecting pressure testing trial at Kellerberrin.

MERREDIN : STATIC DISPLAY OF COMPANY EQUIPMENT

Various items of grain handling and maintenance equipment were displayed inside an 'E' type horizontal storage (wheat capacity 233,000 tonnes constructed of galvanised iron on inverted Y frame timber wall supports with bitumen floor and corrugated iron roof on trusses mounted on steel columns). The displays included:

a A standard country mobile elevator fitted with spray equipment used for the application of a grain protectant to the grain stream at intake into the older types of storages in the C.B.H. system. These machines are virtually unchanged from the original design developed from a bag elevator in the early 1930's. They incorporate a bucket belt encased in a light sheet steel barrel and driven by a direct drive belt from an 8 horsepower diesel engine. The handling capacity of grain delivered by the farmers motor truck through a self emptying hopper direct to the elevator boot is 60 tonnes per hour. The machine is moved along the storage to progressively fill it through steel chuting leading from the elevator head to the centre of the storage. The elevator is also used to retrieve grain from inside the storage by means of an attached mechanical scoop known as a Clarke Shovel.

b Lobstar - a machine designed to compliment the country elevator for the receival and retrieval of grain in an open bulkhead situation. Screw Augers on the front of the machine under the delivery hopper moves the grain to a central belt conveyor and delivers it to a mechanical thrower to direct the grain to the stack being filled. Retrieval is by removal of the hopper and driving the auger front of the machine into the grain stack for self recovery and loading via the conveyor into motor trucks under the end chute.

c Front-end Loader - These machines are based on an industrial 60 horse-power tractor fitted with a hydraulically operated bucket with a capacity of 1.6 tonnes of grain. They are used to retrieve grain from the horizontal grain storages for delivery to an elevator pit and so out to the transport system. Each machine is capable of outloading from an average storage at a rate of 100 tonnes per hour. A feature of the tractor is the slick tread driving tyres used to minimise damage to storage floors and to spilled grain which otherwise occurs with conventional lug grip tyres.

d Other Items of Equipment used Throughout the System were Displayed as - Trailer mounted high reach work platform: Pest Control specialised wash down unit: Pest Control standard mobile unit with spray equipment, pumps and samples of materials used (herbicides, rodenticides, chemical protectants and fumigants): Standard country truck fitted with crane and clam-shell bucket: Mobile mechanical maintenance workshop unit: High reach portable scaffolding: Sampling equipment for on site testing of grain quality: Safety equipment and photographic displays of the Company over the past 50 years.

The display site was also used as the venue for a social barbecue function held during the overnight stop at Merredin and has been described as "The World's Biggest Grill Room".

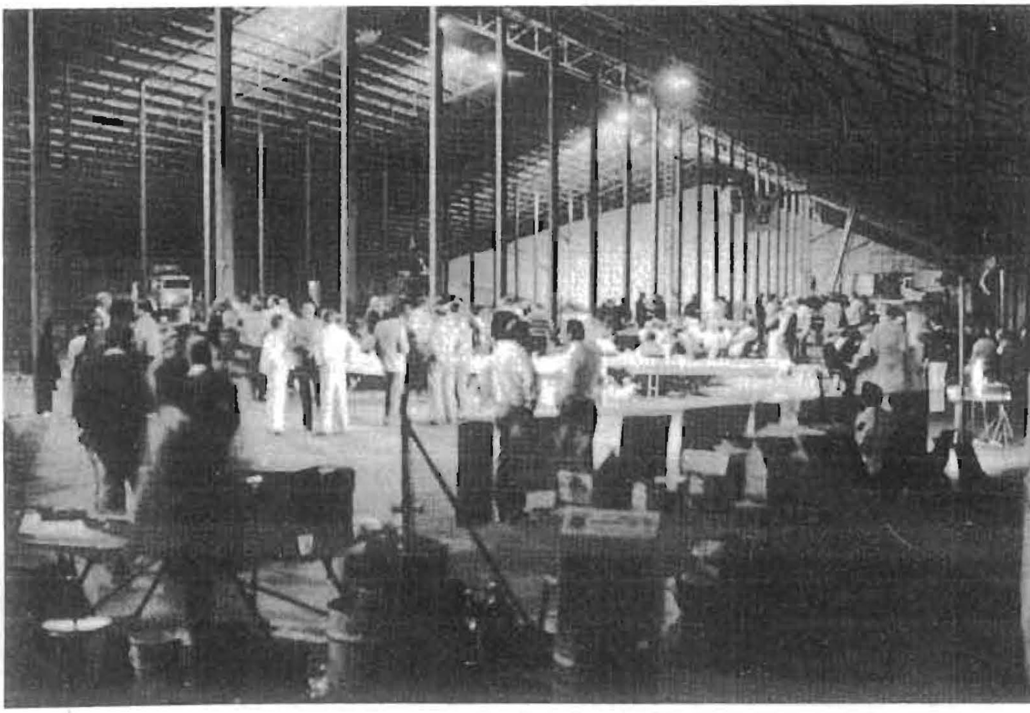

Photo 16. Inside "The World's Biggest Grill Room" - the venue of a social barbeque evening held for Symposium delegates inside a large horizontal storage at Merredin. In the background is a static display of various grain handling machinery and equipment fronting a stack of some 100,000 tonnes of wheat.

NEMBUDDING - SOAP BUBBLE LEAK DETECTION TEST

A field test of leak detection in a sealed and pressurised grain storage - 'A' type horizontal storage of 21,800 tonnes wheat capacity - was demonstrated at Nembudding. This storage was pressurised by using the gable end fans and a weak detergent solution was sprayed over areas of the roof and walls where leaks had been previously detected. The resultant bubbles appearing over the faulty areas enabled the leaks to be pin-pointed and marked for future repairs to the seal. Although other methods of leak detection by use of sophisticated sound, heat and colour measuring equipment have been tried, the simplicity and effectiveness of the "soap bubble" test was shown to be the most viable for our conditions and circumstances. For details of leak detection methods refer to Paper 22.

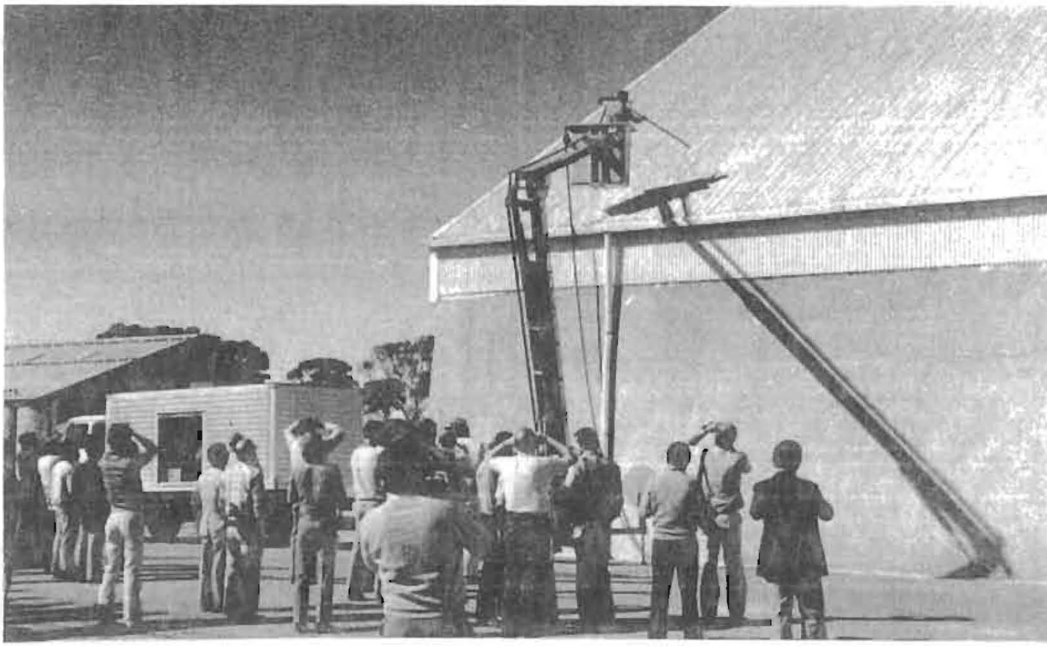

Photo 17. Symposium delegates at a "leak detection" test at Nembudding. After pressurising the storage, a weak detergent solution is sprayed over external surfaces. Escaping air through leaks form bubbles which are then marked for later attention to the seal.

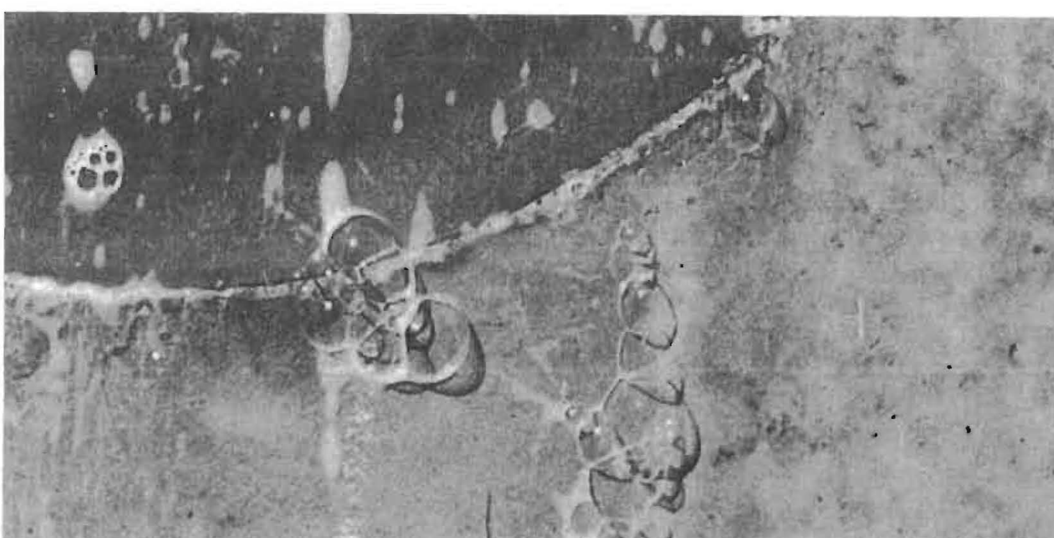

Photo 18. Close-up view of soap bubbles forming in leak areas through a concrete wall.

WYALKATCHEM - SEALED OPEN BULKHEAD EMERGENCY STORAGE.

A sheeted and sealed open bulkhead type storage was demonstrated at this site. The storage consists of a slightly cambered (for drainage) bitumen pad laid on open ground with portable steel frames erected around the perimeter. Grain is loaded into the storage using conventional mobile elevators (or a lobstar and thrower) and the stack is covered with vinyl sheeting anchored with a series of cables. Each sheet is sewn and heat sealed to the next and the covering is then sealed to side sheets placed down the inside of the walls and laid back along the floor under the grain to form a sealed envelope. Small ports are built into the top sheets to allow for sampling and the introduction of a fumigant should this become necessary to control insect activity. This type of storage is used solely as an emergency facility to hold overflow grain in times of good seasons. Its flexibility allows it to be moved from site to site as required. For details of sealing open stacks and the 'bunker' system, refer to Papers 38 and 39.

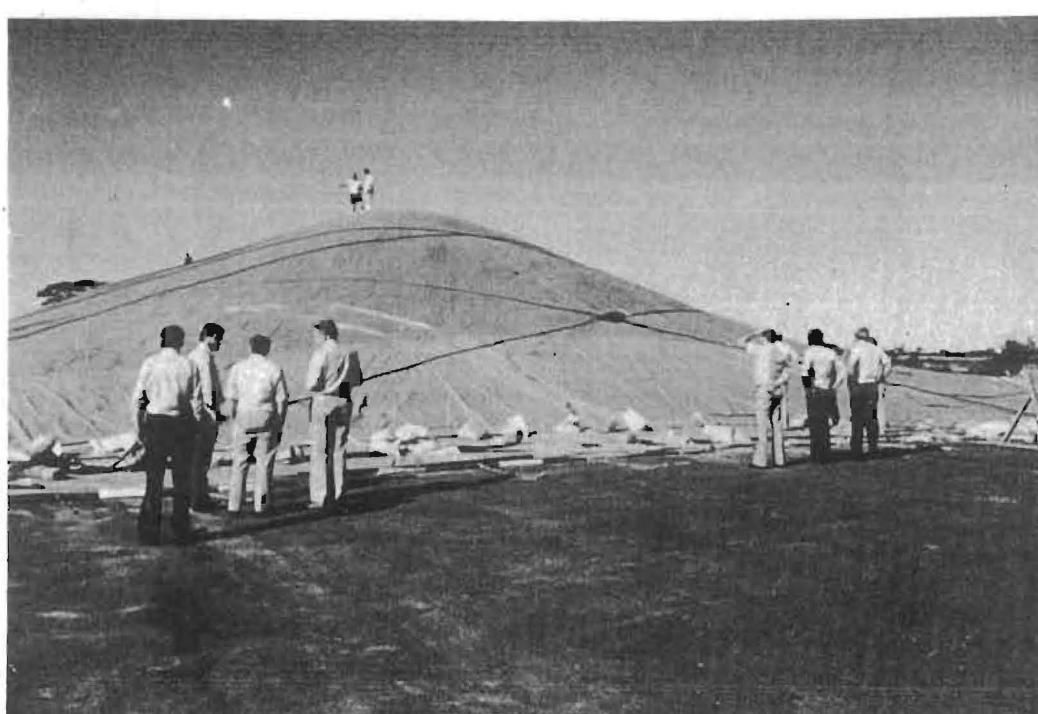

Photo 19. A covered "bunker" storage. Cables are used to tie the PVC sheets to the grain surface to prevent wind damage.

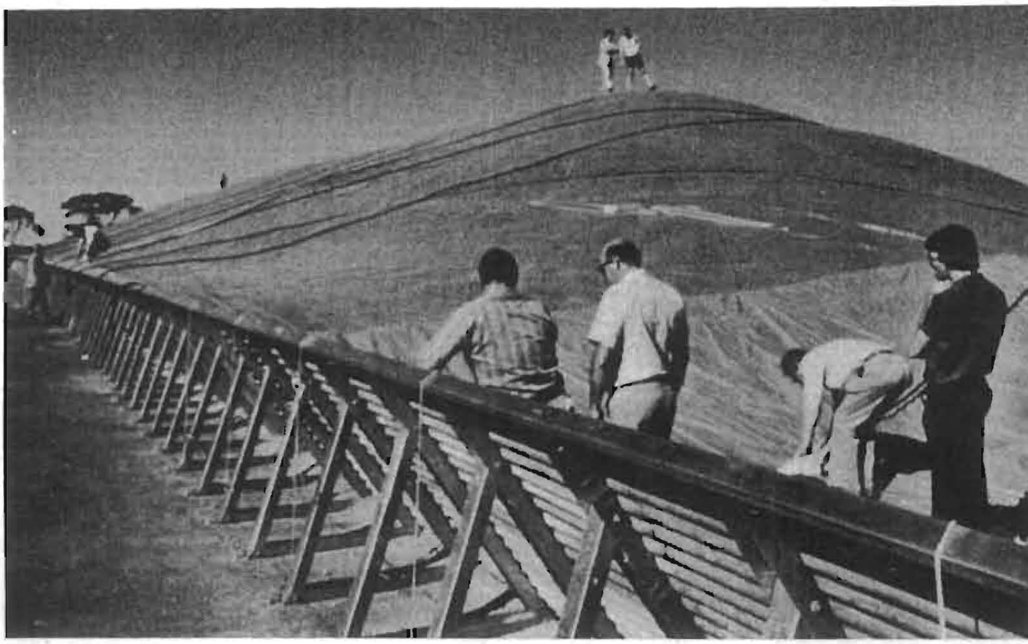

Photo 20. Bunker storage at Wyalkatchem showing the bulkhead type side walls constructed of portable steel frames.

Photo 21. Delegates visit a vintage farm machinery museum inside one of the original grain storages built at Wyalkatchem in 1933.

Photo 22. A brief luncheon interlude at the farm of C.B.H. Director Mr Jack Lundy at Cunderdin. The opportunity was taken to view displays of sheep mustering with dogs, sheep shearing and items of farm machinery used on a typical wheat and sheep farm.

CUNDERDIN : INTRODUCTION OF CARBON DIOXIDE INTO A SEALED STORAGE.

The demonstration at this sealed and fitted storage - a 'C' type horizontal silo 27,700 tonnes of wheat capacity - was the introduction of carbon dioxide gas into a full storage of grain. The liquid CO_2 was supplied to the site by a tanker vehicle and was converted to gas by a vaporiser before being pumped into the storage through perforated ducting laid along the floor. The rate of induction is up to 4 tonnes per hour and a total supply of 48 tonnes is used to give an atmosphere of approximately 75% CO_2. Recirculation of the gas within the storage is by means of ducts and fan which draw the CO_2 from the bottom of the storage and delivers it into the top head-space where it is allowed to spread and permeate back through the grain to the bottom, ensuring an even distribution of gas at all times during the period of fumigation. For details of the use of CO_2 as a fumigant, refer to Papers 5, 6, 7, 19, 25 and 28.

Photo 23. Demonstration of the introduction of CO_2 gas into a sealed storage at Cunderedin. Liquified carbon dioxide is supplied by tanker from where it is pumped through a vaporiser into the storage.

YORK : 'L' TYPE STEEL VERTICAL STORAGES.

A brief inspection of the newly constructed 'L' type vertical steel silo was made at this point. The cells are each of 5,000 tonnes wheat capacity and are fully sealed and coated with a heat reflectant material during construction. They are also fitted for the introduction and recirculation of an inert gas. For details of storage design for C.A. refer to Paper 17.

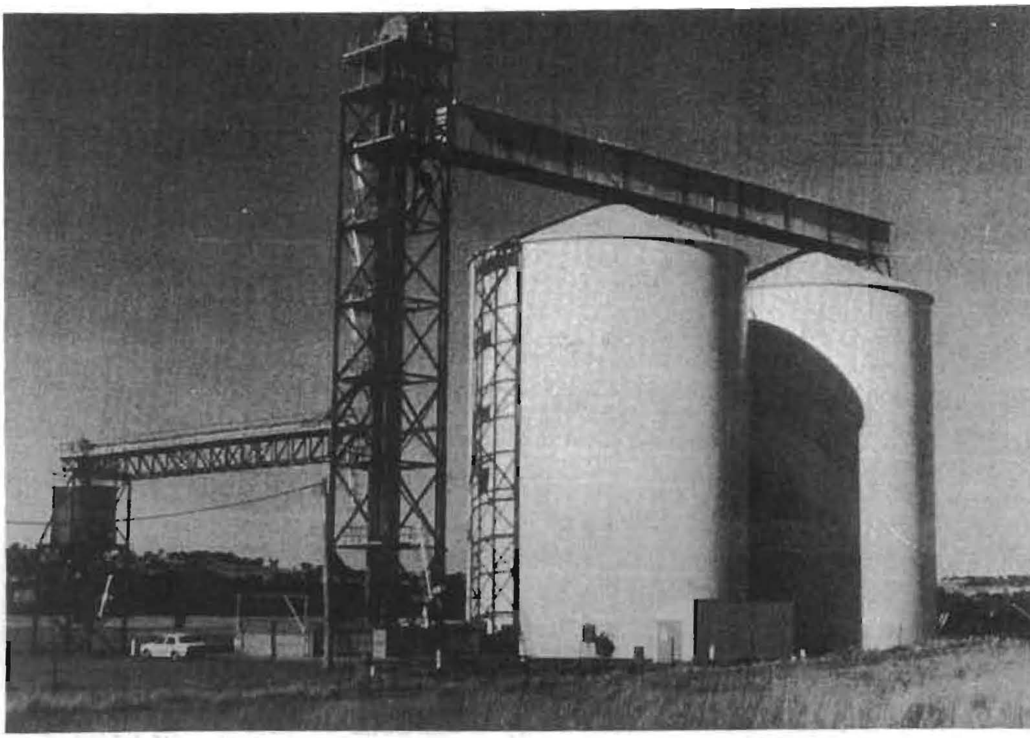

Photo 24. Twin steel vertical cells, each of 5,000 tonnes capacity with a through-put of 300 tonnes per hour at York.

KWINANA : SATURDAY 16TH APRIL 1983

By courtesy of Westrail (the State Government's rail system), a special train carried delegates from Perth through the suburbs to the grain export terminal at Kwinana. Here the largest single storage to be sealed anywhere in the world was inspected.

KWINANA : SEALING AND FITTING OF NO. 1 HORIZONTAL STORAGE.

A very large horizontal storage (wheat capacity 300,000 tonnes) at the Kwinana Grain Export Terminal, completely sealed and fitted for the introduction and recirculation of carbon dioxide, was inspected on the Saturday following the first week of the Symposium. Supplies of carbon dioxide have been arranged from a Nickel refinery, some 3 kilometres from the terminal, where it is produced as a by-product in the refining process. A scrubbing, drying and cooling plant and a pipe line has been constructed to pump the gaseous CO_2 direct to the storage as and when required. Entry is through a plenum chamber and a series of valves through progressively reduced pipes into each of the two under storage tunnels which then act as very large manifolds. A further 32 entry points are supplied along the two side walls to supplement the tunnel entries in uniform swamping of the storage with a calculated 300 to 400 tonnes of gas in a full storage.

Once the storage is filled with carbon dioxide to a concentration of about 40%, the gas will be recirculated by drawing it from the tunnels and floor areas by extractor fans built into the ducting and directing it into the head space above the grain where it will spread and permeate back through the grain to the floor and tunnel areas to be again picked up and recirculated in a continuing movement.

Release of positive pressure built up during the CO_2 swamping action, and of both positive and negative pressures induced by the prevailing weather conditions of sun and wind, is through a series of pressure relief valves connected by ducting to the head space of the storage.

Monitoring of gas concentrations and distribution within the storage is carried out during all stages of the fumigation through tubing and extractor pumps to provide samples of the internal atmosphere for testing with gas analysers. Should concentrations decline to below required levels, a maintenance phase will introduce a further supply of carbon dioxide through the pipe line (estimated 5 to 10 tonnes daily).

When the grain in the storage is required for outloading to ships, the gas clearance phase is commenced to allow safe entry into the storage. The recirculation ducting, with valves reversed, is used to expel gas in the low levels (floor and tunnels) direct to the atmosphere. Large fans built into each gable end of the storage are used, in conjunction with prevailing wind condition, to create an air current from one end of the head space to the other and clear these areas of gas to the outside atmosphere. Blower fans are also used in the tunnel areas to assist the extractor fans in the end ducting to clear this area of concentrations of gas.

When the gas analysers show all areas are safe for re-entry, the seals around the outloading conveyors are removed, entry doors opened and the storage outloaded in the normal manner. Should excessive grain dust remain in suspension in the working areas because of an absence of air movement in the sealed storage, the gable end fans and extractor fans can be used to create an artificial air flow for the removal of grain dust, so minimising any risk of a dust explosion and increasing worker comfort. For details of C.A. in a very large storage refer to Paper 21.

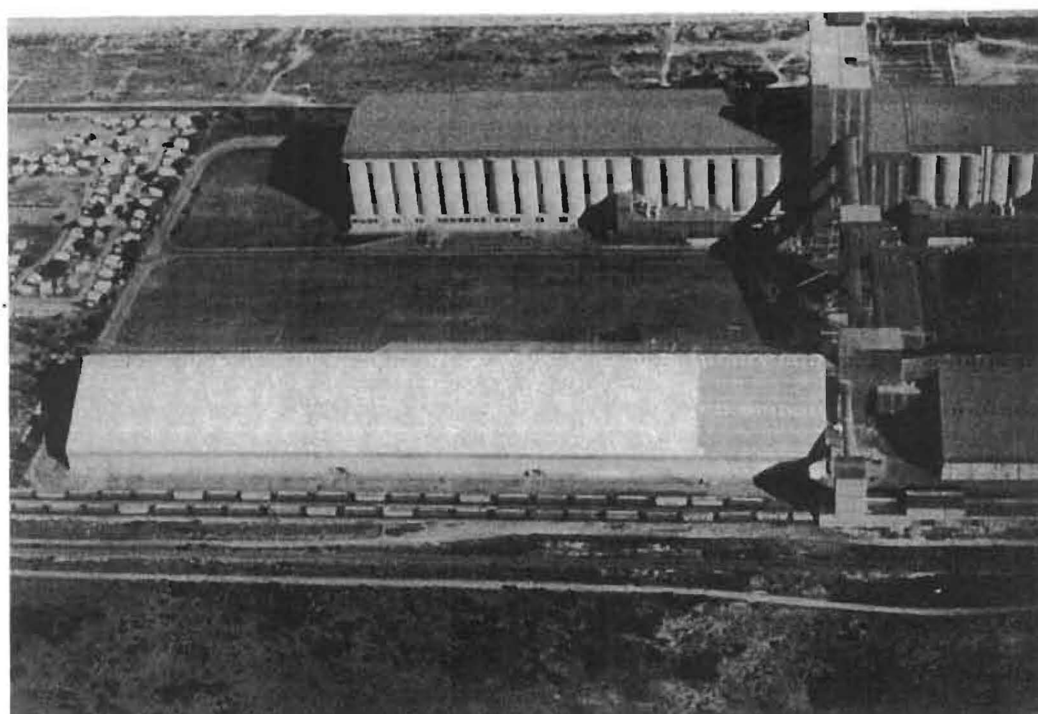

Photo 25. The Kwinana export terminal showing the No. 1 horizontal storage (foreground) during the process of sealing. This storage, of approx 300,000 tonnes wheat capacity, is the largest yet sealed. It is completely fitted with manifolds for the induction of CO_2 gas direct via a pipline from a nickel refinery some 3 kilometres distant. Gas recirculation and clearance systems are provided together with pressure relief vents and a centrally located monitoring system to automatically provide gas concentration readings throughout all areas of the storage.

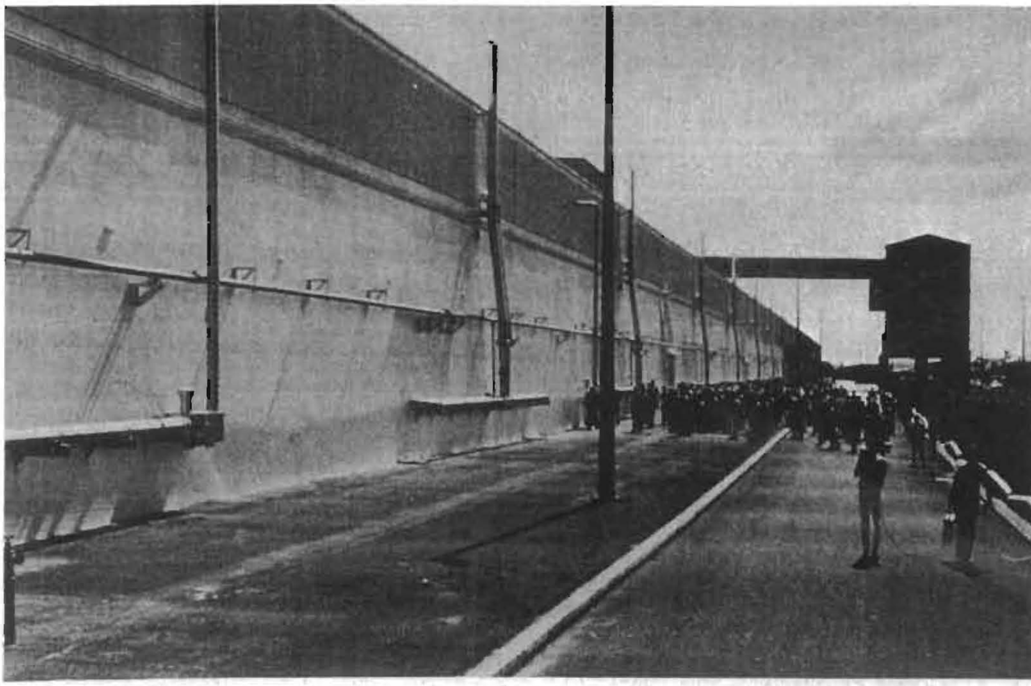

Photo 26. Delegates arriving at Kwinana to inspect the sealed and fitted No. 1 Horizontal storage.

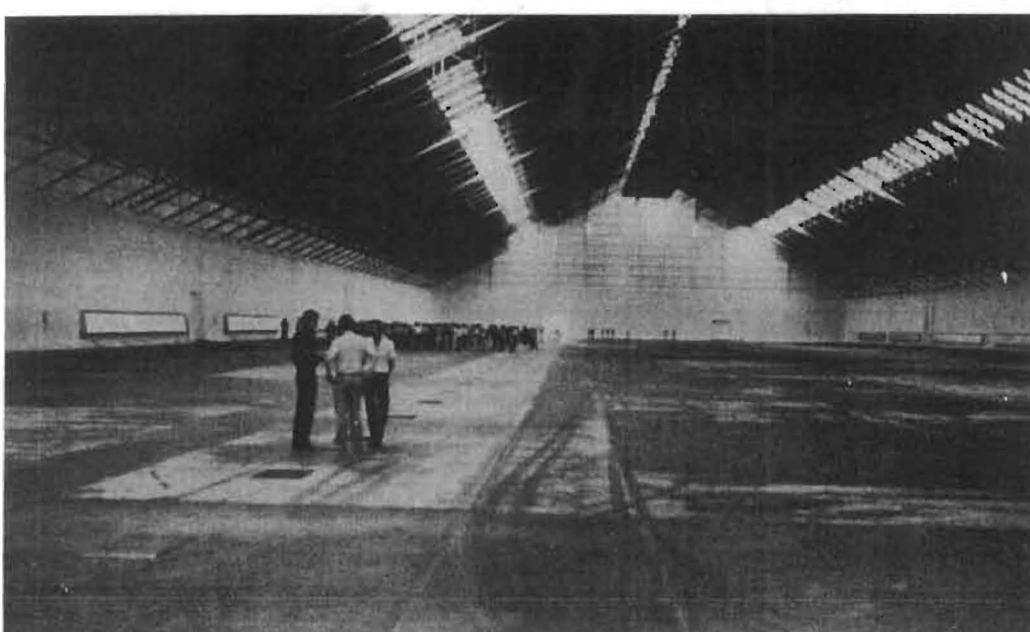

Photo 27 An interior view of the No. 1 horizontal storage at Kwinana.

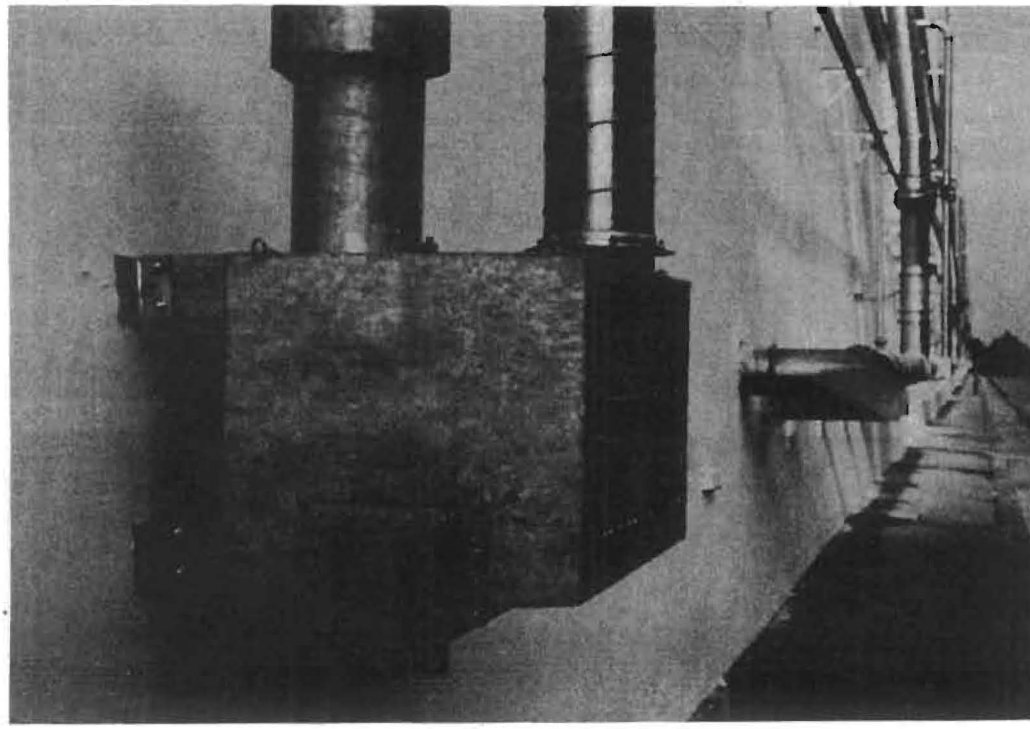

Photo 28. A close-up view of the pressure relief vent fitted at Kwinana. The spiralled ducting enters the storage throught the curtain wall. This allows excessive internal pressure to be vented through an oil bath and out to atmosphere through the short hooded outlet. Negative pressure is relieved by the reverse to this system, allowing air at atmospheric pressure to enter the silo back through the hooded duct, the oil bath and the duct leading to the headspace. The level at which pressure venting occurs may be adjusted by increasing or decreasing the level of oil in relation to the internal baffle fitted between the two chambers.

SESSION 7.

FUTUROLOGY

Papers by:

G. Love	–	Australia
E. Jay	–	United States of America
D.J. Calverley	–	United Kingdom
H.J. Banks	–	Australia

COST COMPARISONS OF DIFFERENT INSECT CONTROL MEASURES

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ABSTRACT

Since the early 1970's, bulk handling authorities have been attempting to reduce their dependence on chemical grain protectants by diversifying their pest control options. However, one factor slowing the adoption of alternative insect control methods has been their apparent high cost.

This paper reports some of the results of a major BAE study conducted between 1979 and 1981 on the economics of modifying existing grain storages for the use of alternative grain insect control measures. The costs of various fumigation/modified atmosphere control strategies such as the use of phosphine, carbon dioxide, exhaust gas and nitrogen are examined, together with costs of various other insect control strategies, including the use of chemical grain protectants.

It is shown that the costs of alternative insect control strategies vary markedly with the type of storage structure, and that these strategies as a group tend to appear expensive in relation to chemical protectants partly because a high proportion of their costs are in the form of capital costs.

It is concluded that the fumigation/modified atmosphere control strategies as a group represent the most economic approach to the elimination of dependence on grain protectants for insect control in Australia's central storage system.

INTRODUCTION

In a year of average grain production over three-quarters of Australia's wheat and one-third of its coarse grains are exported. The acceptability of Australia's grain to overseas buyers is of importance to government and to grain marketing authorities. For example, the Australian Government's Exports (Grain) Regulations require all wheat, barley, oats and sorghum exported from Australia to be inspected and shown to be free of insect infestation.

For about two decades, the bulk grain handling authorities (B.G.H.A.'s) have been able to rely almost exclusively on chemical grain protectants to ensure this requirement is met. However, grain insects have rapidly developed resistance to grain protectants. Since the early 1970's, bulk handling authorities have been attempting to reduce their dependence on chemical grain protectants by diversifying their pest control options.

Many alternative control methods have been investigated. However, one factor slowing the adoption of these methods, particularly those which would involve some modification of existing storages, has been their apparent high cost. For many of these methods, a high proportion of their costs appears in the form of capital costs. For this reason, they appear somewhat expensive in relation to a control strategy such as chemical grain protectants, for which the capital cost component is low. While costings of these different insect control measures have appeared from time to time, comparison of these costings have tended to be difficult due to the different accounting conventions and assumptions adopted.

Accordingly, and at the request of the Standing Committee on Agriculture, the Bureau of Agricultural Economics between 1979 and 1981, conducted a major study on the economics of modifying existing grain storages for the use of alternative grain insect control measures (Love *et al.*, 1983). This study formed the basis for a Working Party report presented to Standing Committee in 1981. Some results from the study were also presented at the First Australian Stored Grain Pest Control Conference in Melbourne (Love, 1981). However, limitations of space prevented a full presentation of results at that conference.

This paper presents more detailed results from the BAE study in relation to the costs of various fumigation/modified atmosphere control strategies. While the cost of these strategies will have risen somewhat in nominal terms since the study was conducted, it is considered that the relativity between these and other control strategies will not have changed greatly.

THE STATE BULK HANDLING SYSTEMS

In Australia, most grain is delivered into the central storage system at or soon after harvest. Its temperature at delivery is typically ideal for the rapid development of insect infestation. In addition, much of the grain may remain in storage for nine months or more before being outloaded. Thus, to supply grain for export in an insect-free condition requires each bulk handling authority to exercise stringent quality control measures at all stages of the grain storage, handling and transport process.

Construction of each of the State bulk handling and storage systems commenced at different times, and each system contains a different mix of vertical and horizontal storages. The size and quality of individual storages also differs. Vertical bins range in capacity from as little as 0.6 kt to as much as 10 kt, while the capacities of horizontal sheds range from around 3 kt to as much as 300 kt. As will be shown later in this paper, the costs of alternative control strategies depend to a large extent on storage type.

The capacities of individual State bulk handling systems by location and storage type, are given in Table 1. This table illustrates the preponderance of horizontal storage in New South Wales and Western Australia, and the high proportion of vertical storage in Victoria, Queensland and South Australia. From the point of view of assessing the costs of different insect control measures, however, the quality of storage is also important. Some idea of this can be gained from Table 2. The slight difference in total storage capacity between each table results from definitional differences as well as new storage added in 1980-81.

THE INSECT CONTROL SYSTEMS

The BAE study examined a number of insect control methods: chemical protectants, refrigerated and ambient aeration, and a group of fumigation/modified atmosphere strategies which used either phosphine, carbon dioxide, exhaust gas or nitrogen gas. The objective of the study was to identify which of the various alternative insect control techniques would provide the most cost effective means of reducing or, more preferably, eliminating Australia's reliance on grain protectants in the existing grain storage system.

Control measures designed to completely eliminate the use of grain protectants include refrigerated aeration and fumigation/modified atmosphere.

With refrigerated aeration, grain is cooled by the forcing through it of large quantities of artifically chilled air. The objective is to reduce grain temperatures in all parts of the grain bulk below those at which insects can breed (about $15^{o}C$). During the storage period any populations of insects present at the commencement of storage hopefully will die out. Maintaining the grain at these low temperatures should also discourage reinfestation.

With fumigation/modified atmosphere control methods, the storage is sealed to an appropriate level of gas-tightness, and an atmosphere lethal to grain insects is introduced. The objective is to kill, over a period of days or weeks, any insects present. Over the fumigation period, the induced atmosphere may degrade to a sub-lethal level. This level may, however, be sufficient to discourge reinfestation. Reinfestation is also discouraged by the physical barrier provided by the sealed fabric of the storage. If the grain is to be stored for a long period (over six months), a retreatment may have to be considered.

COST CALCULATIONS

To enable valid comparison between the various control methods, all capital costs were discounted over the lifetime of the storage modifications. The annual equivalent of the capital costs were then added to annual operating costs to arrive at a total annual cost. These total annual costs were expressed on a dollar per tonne of grain treated basis, assuming a 90 per cent fill at the beginning of the storage period and that only one parcel of grain was treated each year.

Whatever the fumigant or modified atmosphere used, a storage must first be sealed to the required level of gas-tightness. Virtually all new grain storages in Australia are now built so as to be able to be sealed or readily sealable. For older structures, however, the cost of sealing can represent a significant capital cost.

When converting one of these older storages for fumigation/modified atmosphere, the cost of sealing per se accounts for about 70 per cent of initial capital cost. The remainder consists of the cost of engineering modifications and the installation of the necessary gas introduction plumbing.

Welded steel silos are readily sealable, since their walls are usually gas-tight. The walls of many concrete bins particularly older bins, however, are often porous and cracked. These have to be coated with a sealant to be made gas-tight. Bolted steel structure have to be sealed around bolt heads and joints. All structures require sealing around doors, hatches and vents. Uncapped silos such as many of those found in South Australia present special problems. These cells must be capped before they can be sealed.

Necessary engineering modifications can include the installation of relief valves to allow for diurnal pressure variations, the fitting of infill sheeting between the walls and roof, the blanking of eaves, and the sealing of hatches and external ducting, and recirculation fans. In the cost analysis, it was assumed that the storage fabric would need to be resealed every eight years. It was also assumed that the pressure valves and gas-introduction plumbing would need to be replaced after twenty years. An allowance was made for annual repairs and maintenance. Both nitrogen and carbon dioxide were assumed to be delivered in liquid form by road tanker. The cost of carbon dioxide treatment was calculated for bins and for sheds. The cost of nitrogen treatment was calculated for bins only.*

The cost of exhaust gas (produced by the burning of LPG) was calculated on the basis of one burner per block of six 2.2 kt bins. It was

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* To be effective in excluding oxygen, nitrogen has to be maintained at an atmospheric concentration of greater than 98 per cent. This can be achieved in bins but would be difficult to achieve in a large shed. With carbon dioxide, the maintenance of such high concentrations of this gas is less critical. Hence carbon dioxide can be used in sheds. In a large shed, carbon dioxide levels are kept even horizontally because of gas density effects and are maintained so vertically by recirculation.

assumed that one burner would be able to purge one bin at a time while also maintaining an insecticidal atmosphere in those bins already purged. It was also assumed that, for horizontal sheds, one burner would be needed for each 10 kt, or part thereof, of nominal capacity. However, it was noted that if modified atmosphere storage using exhaust gas were to be introduced to a particular region, a system of rotating LPG burners would probably be possible. This could reduce the cost of this type of modified atmosphere storage.

The cost of fumigation using phosphine was calculated on the use of one 'blanket' (1,100 g phosphine equivalent) per 1.35 kt of grain.

The annual capital and operating costs for the various fumigation/modified atmosphere treatments, as calculated in the Bureau study, are presented in Table 3. These costs are summed to arrive at a total annual cost, and compared with the total annual costs of other insect control options, in Table 4.

IMPLICATIONS OF THESE COSTS

Two things are apparent from Table 4. Firstly, the costs of fumigation/modified atmosphere using phosphine, carbon dioxide or exhaust gas appear to be generally lower than the costs of refrigerated aeration or modified atmosphere storage using nitrogen. Secondly, the table illustrates clearly how the overall cost of fumigation/modified atmosphere strategies varies with the type of storage structure required to be modified. There are two reasons for this. The major one is that the cost of sealing presently-unsealed storage structures varies, being cheapest for welded steel bins and most expensive for older concrete bins (especially those which have to be capped prior to sealing).

The cost of sealing horizontal sheds appears intermediate between the two. There also appear to be economies of size in the sealing of larger horizontal sheds, as a result of their lower surface area per unit volume of usable storage capacity.*

A secondary reason (important in the case of carbon dioxide and exhaust gas) is that horizontal sheds, even fully filled, have a large amount of

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* However, it should be noted that because larger sheds are usually used for segregating several grades of grain, the degree of fill may sometimes be less than if only one grade had been stored. Therefore, on a per tonne of grain treated basis, the cost advantage of sealing larger sheds may be reduced or, in some situations, reversed.

headspace relative to fully filled bins. Consequently, more gas may be needed per tonne of grain treated.

CONCLUSIONS

From the cost data in Table 4, which relate to the modification of existing grain storages for the use of alternative grain insect control measures, the following conclusions can be drawn:

- For those control methods examined, the lowest-cost insect control strategy which has the potential to completely eliminate the need to use chemical grain protectants appears to be phosphine fumigation in welded steel bins.

- The cost of sealing existing horizontal sheds and fumigating with phosphine appears comparable, in broad terms, with the cost of those chemical grain protectants used in the eastern States of Australia.

- If a shed were to be sealed, and fumigated with carbon dioxide or exhaust gas instead of phosphine, the total cost of treatment could rise to, in some cases, about twice the cost of those chemical grain protectants used in the eastern States of Australia.

Overall, the Bureau study concluded that, on a national basis, the fumigation/modified atmosphere control strategies as a group represented the most economic approach to the elimination of dependence on grain protectants for insect control in Australia's central storage system. The cost of sealing welded steel bins and horizontal sheds, plus the cost of fumigation (assuming phosphine would be used as the principal fumigant) appeared on par with the cost of grain protectants in the eastern States. The study also concluded that in view of the high cost of sealing older-type capped and uncapped bins, for these storages, the best option appeared to be to attempt to reduce the amount of grain protectant needed by timely grain movement and/or grain cooling.

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Pest control alternatives for the central storage system. Proceedings of the First Australian Stored Grain Pest Control Conference, Melbourne, Victoria, May 1981, Sect 1, 9-13.

Love, G., Twyford-Jones, P. and Woolcock, I. (1983)
An Economic Evaluation of Alternative Grain Insect Control Measures,
Occasional Paper No. 78, Bureau of Agricultural Economics, Canberra. 115 pp.

Table 1 Storage capacity of the mainland state bulk handling authorities: 1980-81

State	Storage capacity	Location		Type		
		Country	Seaboard	Vertical	Horizontal	Other (a)
	kt	%	%	%	%	%
New South Wales	5 963	95	5	25	71	4
Victoria	3 940	73	27	53	46	1
Queensland	1 479	92	8	79	31	–
South Australia	4 080	57	43	80	13	7
Western Australia	7 909	76	24	13	59	28
Mainland States	23 371	78	22	39	50	11

(a) Includes horizontal-type emergency storages such as steel-frame sites (Western Australia), 'A' frame sites and timber framed bulkheads (New South Wales), and various types of bulkheads and sheds without fixed machinery.

Sources: AWB, Annual Report 1980-81; BGHA's (personal communications).

Table 2 Storage capacity of the Mainland State Bulk Handling Authorities: by storage type: 1979-80

Type	Capacity	Percentage of total capcity
	kt	%
Welded steel/sealed concrete silos	1 801	8.2
Horizontal sheds	8 525	38.6
Capped concrete/bolted steel silos	739	3.4
Uncapped concrete silos	2 858	12.9
Large sub-terminals (a)	1 311	5.9
Provisional/Temporary (b)	1 657	7.5
Seaboard terminals	5 197	23.5
Total	22 088	100.0

(a) Large horizontal sub-terminals (New South Wales) and transfer depots (Western Australia). Victorian and South Australian sub-terminals included in country storages.

(b) Includes timber bulkheads and some horizontal storages without machinery, but not 'A' frame sites, bunker storages or bagged grain sheds.

Source: Love (1981).

Table 3 Capital and operating costs(a) of various fumigation/modified atmosphere insect control strategies: for various types of storage

	Type of storage				
	Vertical silo			Horizontal silo	
	Welded steel	Capped concrete	Uncapped concrete	15 kt	30 kt
	$/t	$/t	$/t	$/t	$/t
Annual cost equivalent of capital costs (sealing and modification) (b)	0.13	1.42	1.98	0.79	0.52
Operating cost (c)					
- phosphine	0.04	0.04	0.04	0.04	0.04
- carbon dioxide	0.36	0.36	0.36	0.50	0.50
- exhaust gas	0.33	0.33	0.33	0.55	0.35
- nitrogen	0.67	0.67	0.67	-	-

(a) Cost of grain treated, assuming 90 per cent fill at time of treatment and that only one parcel of grain is treated each year.

(b) Includes cost of contract for maintenance of the seal to a specified standard.

(c) For one gas treatment.

Note: A real rate of interest of 5 per cent has been assumed. This rate is defined as the nominal or market rate of interest less the annual rate of inflation.

Table 4 Total annual cost(a) for selected grain insect control strategies

Storage type and Storage period	Control strategy					
			Fumigation/modified atmosphere(c)			
	Chemical protectant(b)	Refrigerated aeration	Phosphine	Carbon dioxide	Exhaust gas (d)	Nitrogen
Six months						
- welded steel bin	0.55	-	0.17	0.49	0.46	0.80
- 15 kt horizontal shed	0.55	2.39	0.83	1.29	1.34	-
- 30 kt horizontal shed	0.55	1.94	0.56	1.02	0.99	-
- Capped concrete bin	0.55	2.38	1.46	1.78	1.75	2.09
- Uncapped concrete bin	0.55	2.38	2.02	2.34	2.31	2.65
Ten months (e)						
- Welded steel bin	0.82	-	0.21	0.85	0.79	1.47
- 15 kt horizontal shed	0.82	2.60	0.87	1.79	1.89	-
- 30 kt horizontal shed	0.82	2.16	0.60	1.52	1.22	-
- Capped concrete bin	0.82	2.59	2.14	2.08	2.08	2.76
- Uncapped concrete bin	0.82	2.59	2.06	2.70	2.64	3.32

(a) Cost of grain treated, assuming 90 per cent fill at time of treatment and that only one parcel of grain is treated each year.

(b) Cost in New South Wales, Victoria and Queensland.

(c) In sealed storage.

(d) Low oxygen atmosphere produced by burning propane in air.

(e) For fumigation/modified atmosphere, one retreatment after six months' storage is assumed. For chemical protectants, a retreatment at half rate is assumed.

IMPERFECTIONS IN OUR CURRENT KNOWLEDGE OF INSECT BIOLOGY AS RELATED TO THEIR RESPONSE TO CONTROLLED ATMOSPHERES

EDWARD JAY

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ABSTRACT

Several recent review papers have commented on the fact that there is little revelance between laboratory studies and the field application of controlled atmospheres (CA) to control stored-product insects. Most of this research used either larvae or adults of one or two species of insects, usually *Tribolium* spp., to demonstrate that there is a difference in mortality when the insects are exposed to different CA compositions. We have developed generalities from this research such as "pupae are harder to control than are the other three life stages of these stored-product insects" and that "a 60% carbon dioxide (CO_2) concentration is more effective against most of these species than is a 98 to 100% CO_2 concentration". Are these generalizations true for all of the major pest species? This paper attempts to develop some order in this area by reviewing recent and chiefly unpublished research by the author. An examination of the responses within and between species and life stages and their relationship to temperature, relative humidity and CA composition of these insects is presented. Shorter exposure times are generally needed to control stored-product pests in field situations than would be expected from laboratory results. Attempts are made to explain the causes of this phenomenon, how it relates to the overall information transfer from the laboratory to actual CA field applications and how the available laboratory data can logically be applied in the field. Future research needs are described on the basis of the information contained in this paper.

INTRODUCTION

Review papers on the effects of controlled atmopheres on stored product pests were published by Bailey and Banks (1975 and 1980). In these papers the effective atmospheric gas compositions to control stored-product insects, regardless of the practical application in a field situation, were mentioned. Some authors have investigated rare atmospheres including helium (Aliniazee, 1972) but the high cost of this gas would not make its use practical. Other authors have investigated either very low relative humidities (r.h.s) such as 7 to 30%, or very high r.h.s such as 72 to 95% (Jay, *et al.*, 1971; Navarro, 1978). These Studies are justifiable from a scientific standpoint and contribute to knowledge on the mode of action of controlled atmospheres (CA).

However, it is not a common practice to store grain at extreme humidity levels, so this information is again of little value to a practical storage insect control scheme.

Bailey and Banks (1980) concluded from the available literature that there are still gaps in our knowledge of the biological action of CA, that we need additional work on dose-mortality responses to various gas mixtures with particular emphasis on tolerant stages and species to provide a basis for an exposure schedule dependent on the temperature of the bulk to be treated; and that we need information on the interaction of high carbon dioxide (CO_2) atmospheres and temperatures.

What is wrong with this overall research effort? It can be seen from the above that, after years of study on the effects of CA on stored-product insects, little data has emerged which can be translated to field studies. Several economically important species such as Sitotroga and Cryptolestes have been overlooked, and much of the research has been conducted with gas mixtures which would be economically impossible to produce in field situations.

When CA is used to control stored-product insects many factors enter into the success of the treatment. Some of these factors are shown in Figure 1 and these factors will form the basis of this paper.

The purpose of this paper is to contribute additional information to our current knowledge of storage insects in response to CA. The information reported is generally based on recent research conducted by the author with Tribolium castaneum (Herbst), Oryzaephilus surinamensis (L.), Rhizopertha dominica (F.), Sitophilus oryzae (L.), Sitophilus zeamais Motschulsky, Trogoderma glabrum (Herbst), Trogoderma variable (Ballion), and Esphestia cautella (Walker). The information presented here deals mainly with the effects of CO_2 and to some extent nitrogen (N_2) atmospheres on these insects and does not consider the effects of generated atmospheres on these pest species.

MATERIALS AND METHODS

When actually purging out a storage bin or structure to obtain various CO_2 concentrations, the oxygen (O_2) concentrations usually present are shown in Table 1.

Table 1 - Approximate O_2 concentrations found when purging with CO_2 or when mixing a given percentage of CO_2 with air. 1/

% CO_2	% O_2
45	12
60	8
75	5
90	2
99	0.3

1/ Balance of mixture will be nitrogen (N_2), argon and rare gases.

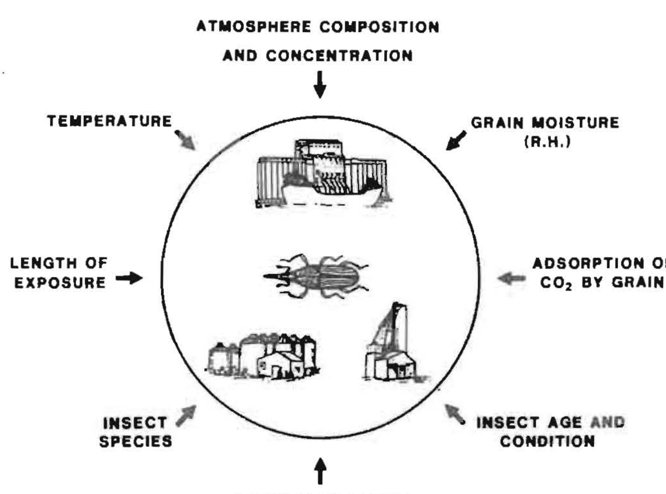

Figure 1 -- Some factors affecting the treatment with controlled atmospheres to control stored-product insects.

This paper will only be concerned with these concentrations of CO_2 at > 50% r.h. unless otherwise noted. The exposure chambers used were similar to those described by Jay and Cuff (1981). They consisted of 2.8-l glass jars that were partly submerged in laboratory water baths which could be heated or cooled to provide different temperatures for some studies. The jars were closed with metal screw-top lids fitted with 0.6 cm o.d. copper tubing that were used as gas inlet and gas exit tubes. The lids were also fitted with a neoprene stopper so a humidity sensor could be inserted. During the exposure the cages containing the insects were suspended in the chamber from a 5-cm length of steel wire hung from the underside of the neoprene stopper holding the thermometer.

The gas mixtures were released from the cylinders through two-stage regulators and flowed through a micrometering valve and flowmeter into gas washing bottles that contained the glycerol-water mixture to adjust the r.h. of the gas. Flow rates of 200 cc/min were used for the first hour and 30 cc/min for the balance of the exposure periods. The r.h. was monitored with an electric hygrometer (model 15-2001 humidity indicator and narrow range humidity sensors, Hygrodynamics, Inc.), and the temperature was recorded daily by using the dial thermometers. A Fisher-Hamilton model 29 gas partitioner equipped with dual columns was used to measure the areas under the peaks.

The effectiveness of the treatment for S. oryzae and R. dominica immature stages was determined by dividing the total number of adult insects that emerged after treatment by the total number that emerged in the control and converting this to percent reduction in emergence (RIE) (Jay, 1980a).

RESULTS AND DISCUSSION

Low temperatures

Temperature is an important factor in the use of CA for insect control, as it is in conventional fumigation. However, little research has been conducted at temperatures below 16°C on the effects of CA. Table 2 shows the effects of 2° and 16°C on mixed age (1,2,3,4 and 5 week old) immature life stages of S. oryzae and R. dominica and shows that at 2°C the cold air alone was more effective than the two CA's tested after a 1-wk exposure against S. Oryzae but not against R. dominica where the 60% CO_2 atmsophere was more effective. A 2-wk exposure at this temperature showed that the cold air and 99% N_2 was more effective than 60% CO_2 against S. oryzae but 100% RIE of R. dominica could be achieved only with the 60% CO_2 atmosphere. At 16°C the effects of the air alone on both species was diminished, whereas R. dominica appears to be the more resistant than S. oryzae at this temperature. At this temperature 60% CO_2 was more effective against both species than was 99% N_2.

High temperatures

Table 3 shows the effects of elevated temperatures on mortality of larvae of *T. glabrum* and *T. variabile* when exposed to 61% and 99% CO_2 at 33° and 38°C. Although at 33°C exposure to 99% CO_2 for 48 h produced high mortalities in both species 100% mortality was obtained only for *T. glabrum*. However, at 38°C exposure to 99% CO_2 for 24 h produced 100% mortality in both species, while 61% CO_2 was only slightly more effective at this temperature than it was at 33°C.

Table 2 - Percent reduction in emergence (RIE) of adults for immature *S. oryzae* (SO) and *R. dominica* (RD) exposed for 1 or 2 wk to two low temperatures at 55-60% r.h.

Atmosphere	SO*		RD	
	1 wk	2 wk	1 wk	2 wk
2°C				
Air	99	100	73	99
60% CO_2	96	99	89	100
99% N_2	95	100	63	96
16°C				
Air	67	89	25	54
60% CO_2	81	99	91	100
99% N_2	42	86	60	91

* From Jay, 1980a.

Table 3 Mortality (%) of T. glabrum (TG) and T. variabile (TV) when exposed to two concentrations of CO_2 at two temperatures and 50% r.h.

Species	Exposure time (h)	61% CO_2	99% CO_2
		33°C	
TG	24	22	90
TV		15	73
TG	48	25	100
TV		15	97
		38°C	
TG	24	21	100
TV		16	100
TG	48	32	100
TV		31	100

Table 4 presents results of similar exposures of the pupae of the two Trogoderma species. Again it can be seen that at 33°C 99% CO_2 was more effective than 63% CO_2 and produced nearly 100% mortality in both species after a 48-h exposure. An increase in temperature to 38°C reduced the exposure time for 100% mortality to 24 h for both species when the insects were exposed to 99% CO_2. Increasing the temperature from 33 to 38°C had little effect on the effectiveness of the atmosphere containing 63% CO_2.

Table 4 - Mortality (%) of pupae of T. glabrum (TG) and T. variabile (TV) when exposed to two concentrations of CO_2 at two temperatures and 50% r.h.

Species	Exposure time (h)	63% CO_2	99% CO_2
		33°C	
TG	24	30	92
TV		22	87
TG	48	96	100
TV		64	100
		38°C	
TG	16	13	100
TV		9	93
TG	24	44	100
TV		31	100

Effects of temperature on three life stages of the same species

Table 5 presents results of exposures of larvae, pupae and adults of O. surinamensis to 77% CO_2 at four temperatures and exposure periods of up to 10 days. One-hundred % mortality of larvae of this species was obtained after an exposure of 3 days at 16°C, after 2 days at 21° and 27°C, and after 1 day at 32°C. Adults of this species displayed similar mortalities when exposed to this CO_2 concentration. However, it took 10 days to obtain 100% mortality of pupae of O. surinamensis at 16° and 21°C but only 3 days at 27° and 32°C.

Table 5 - Time in days required to obtain 100% mortality of O. surinamensis when exposed to 77% CO_2 at different temperatures and 50% r.h.

Temperature °C	Larvae	Pupae	Adults
16	3	10	3
21	2	10	3
27	2	3	2
32	1	3	1

Species and life stage of stored-product insects

When the results from Tables 3 and 4 are compared it can be seen that pupae of T. glabrum and T. variabile are more susceptible to the effects of 61% (or 63%) and 99% CO_2 than the larvae, except for 16 h exposure at 38°C whereas Table 5 shows that pupae of O. surinamensis are much more resistant to a CO_2 treatment than are larvae and adults. These differences are found between closely related species (Trogoderma spp) and within the different life stages of the same species (O. surinamensis). However, a few comparisons are available from the existing literature on comparative studies between the different life stages of 2 species that were conducted under identical conditions.

A study directly comparing the effects of two CA's on the 4 life stages of T. castaneum and E. cautella was conducted in steel towers containing shelled peanuts (groundnuts). These tests were conducted at ca 27°C and the rh in the bulk was ca 66%. Table 6 presents results of these studies. Eggs of both species were controlled (100% mortality) in 2 days in an atmosphere containing 63% Co_2 and in 3 days in a 99% N_2 atmosphere. This table shows that it took more than 6 days to attain complete mortality of T. castaneum larvae in the CO_2 atmosphere while this level of mortality was obtained in only 2 days when larvae of this species was exposed to the N_2 atmosphere. Larvae of E. cautella exhibited a reverse reaction to these two atmospheres and were controlled in 5 days in the CO_2 atmosphere and in more than 6 days in the N_2 atmosphere. Pupae of both species were controlled more rapidly in the N_2atmosphere than in the CO_2 atmosphere while E. cautella pupae was more sensitive than T. castaneum pupae.

Table 6 - Time in days required to obtain 100% mortality of all life stages of T. castaneum (TCA) and E. cautella (EC) when exposed to two atmospheres at 27°C and 66% r.h.

Life Stage	Species	63% CO_2 Days	99% N_2 Days
Eggs	TCA	2	3
	EC	2	3
Larvae	TCA	greater than 6	2
	EC	5	greater than 6
Pupae	TCA	5	4
	EC	3	2
Adults	TCA	4	3
	EC	2	2

Adults of T. castaneum were completely controlled in 4 days in the CO_2 atmosphere and in 3 days in the N_2 atmosphere while adults of E. cautella were controlled in 2 days in both atmospheres.

Relative humidity

The response of 3 life stages of T. castaneum to 66% r.h. (from the data on peanuts reported above) and to 50% r.h. (Jay and Cuff 1981) are compared in Table 7. These r.h.'s are in the range of conventional grain storage practice. Table 7 shows that after 48 h exposure mortality of larvae and pupae is much higher at the lower r.h. while there is no significant difference between adult mortalities recorded at two r.h.'s. After 72 h, mortality of larvae and pupae remained markedly higher at 50% than at 66% r.h. but mortality of adults at 50% are close to those recorded at 66% r.h.

Table 7 - Mortality (%) of 3 life stages of T. castaneum when exposed to ca 60% CO_2 at 50% and 66% r.h. for 48 and 72 h.

Life Stage	48 h		72 h	
	50%*	66%	50%	66%
Larvae	48	20	80	53
Pupae	89	46	99	81
Adult	96	94	99	98

* From Jay and Cuff (1981).

Effect of CA composition

Jay (1971) stated that a 60% CO_2 concentration at or above 27°C was adequate for controlling most stored product insects in a 4-day exposure period. This statement was based on unpublished laboratory studies described below. However, from data already presented (ie Tables 3 and 4) it can be seen that some species and life stages respond much faster to higher CO_2 concentrations than they do to those around 60%.

Table 8 shows the time required to obtain 100% mortality of larvae, pupae, and adults of O. surinamensis exposed to four CO_2 concentrations at four temperatures. This table shows that there is a dramatic difference in the time required to attain 100% control of these life stages at 16° and 21°C when the CO_2 concentration is raised from 91 to 98%. At 91% CO_2 at these two temperatures, it takes 10 days to obtain 100% mortality; at 98% CO_2 it only takes 3 days to attain this level of control. This difference is not so pronounced at 27° and 32°C; however, time required to obtain 100% mortality drops from 2 days to 0.7 days at 32°C when the CO_2 concentration is increased from 91 to 98%.

Table 8 – Time in days to obtain 100% mortality of larvae, pupae and adults of O. surinamensis when exposed to four CO_2 concentrations at four temperatures and 50% r.h.

% CO_2	Temperature – °C			
	16	21	27	32
62	10	10	4	4
77	10	10	3	3
91	10	10	3	2
98	3	3	2	0.7

Table 9 presents data on the effects of four high CO_2 concentration atmospheres on T. glabrum and T. variabile larvae and pupae at 36°C. In most cases there is a gradual increase in mortality as the CO_2 concentration is increased from 61 to 89%. However, there is again a large increase in mortality when the CO_2 concentration is increased from 89 to 99%.

Table 9 – Mortality (%) T. glabrum and T. variabile larvae and pupae when exposed for 24 h to four CO_2 concentrations at 36°C and 50% r.h.

% CO_2	% Mortality			
	T. glabrum		T. variable	
	Larvae	Pupae	Larvae	Pupae
61*	21	44	16	31
76	37	49	23	45
89	42	61	20	67
99	100	100	100	100

* Pupae were exposed to an atmosphere containing 63% CO_2.

Jay and Cuff (1981) also presented data on the effects of 58 and 97% CO_2 atmospheres on three life stages of T. castaneum. This data is summarized in Table 10 and shows that there was less marked difference between these two atmospheres on larvae and pupae when compared to the response of the adults.

Table 10 - Mortality of three life stages of T. castaneum when exposed to two CO_2 atmospheres at 27°C and 50% r.h.

Life Stage	Exposure time (hr)	% Mortality	
		58% CO_2	97% CO_2
Larvae	72	80	99
Pupae	72	99	100
Adults	24	28	100

* From Jay and Cuff (1981).

The data from Tables 8, 9, and 10 are all based on the effect of different CO_2 concentrations on species which feed externally while Table 11 presents data on R. dominica and S. zeamais, which are internal feeders. These insects were exposed at 27°C and 43% r.h. as 1, 2, 3, 4 or 5 week-old immatures for 1, 2, 3 or 4 days to the CO_2 concentrations indicated in Table 11. The data in this table are from a statistical analysis of the individual degree of freedom of counts of adult emergence (as compared to controls exposed in air) resulting from these tests.

Table 11 - Mean number of adults that emerged from immatures exposed for 1, 2, 3 or 4 days to five atmospheres at 27°C and 43% r.h.

Atmosphere %	R. dominica	S. zeamais
Air	70.7	22.6
39 CO_2	30.4	14.9
48 CO_2	17.1	9.4
99 CO_2	14.5	8.9
60 CO_2	9.7	5.8

The data in Table 11 shows that as the CO_2 concentration is increased from 39% to 48% the number of emerging adults decreases. However, when the number of adults emerging from 99% CO_2 is compared to those emerging from those exposed to the 60% CO_2 the number emerging in the later atmosphere are less than those emerging from the former atmosphere.

These data on R. dominica and S. zeamais were used as a background for the 60% CO_2 control regime recommended by Jay (1971). This regime is inadequate for species such as T. glabrum and T. variabile which cannot be successfully controlled with 60% CO_2. Also, O. surinamensis and some life stages of T. castaneum are controlled more rapidly by CO_2 concentrations approaching 100% than they are at CO_2 concentrations of ca 60%.

Translating laboratory studies to field use

Generally, when a field test using CO_2 is conducted on infested grain higher mortality is obtained in the natural population than would be expected from laboratory studies. This can be seen in the field test reported by Jay and Pearman (1973) where a 4-day treatment at a CO_2 concentration of ca 60% of 958 kl of maize resulted in 99.9% control of a natural population of Sitophilus spp (probably most were S. zeamais). This infested maize had a higher m.c. (11 to 16% m.c. or c.a. 55 to 80% r.h.) than did the infested maize used in the laboratory tests with S. zeamais (Table 11) and a lower mean temperature of 20.7° vs 27°C for that used in the laboratory tests. This 99.9% control can be compared to 74.4% control obtained in the laboratory studies (% taken from the data used to prepare Table 11) and would not be expected based on the higher r.h.'s and lower temperature encountered in the field study.

The possible reasons for the difference between the degree of control obtained in the laboratory and that obtained in the field could be due to the technique used in the laboratory where, at the end of the exposure, the insects are immediately removed from the CA exposure container and transferred to another container for post-exposure observations in a normal atmosphere. This practice probably causes a rapid desorption of the CO_2 absorbed during the exposure. In field situations the grain may not be moved or aerated for several days or weeks after the treatment is concluded, and there would be a gradual desorption of the CO_2 from the grain into the surrounding atmosphere. The speed of this desorption would be, of course, dependent on the tightness of the storage vessel that had been treated.

Concentrations of CO_2 below 35% can be effective in providing continued control of stored-product insects. In laboratory studies on the effects of 5% to 25% CO_2 concentrations, 25 S. oryzae adults were confined on 100g of wheat for 24 h before they were exposed for periods of 15 or 30 days. These tests were conducted at 27°C and 60% rh and post-exposure readings were made for 4-weeks after the insects were removed from the tested atmospheres.

Table 12 shows that when insects were exposed for 15 days to 5% CO_2 there was a 39% RIE but only a 1% RIE was obtained with the insects exposed for 30 days to this atmosphere. Increasing the CO_2 concentration to 15% resulted in a 89% RIE after a 15-day exposure and 73% for the 30-day exposure. Exposures to the atmospheres containing 20 and 25% CO_2 resulted in almost 100% RIE for both exposure periods.

Table 12 - Mean number of adults that emerged and % reduction in emergence (RIE) of S. oryzae after a 15 or a 30 day exposure to indicated CO_2 concentrations at 27°C and 60% r.h.

Exposure: Time % CO_2	15 Days: Number of adults	15 Days: % RIE	30 Days: Number of adults	30 Days: % RIE
0.1 (Air)	720	--	1,004	--
5	436	39	998	1
10	305	50	559	44
15	79	89	274	73
20	19	97	34	97
25	10	99	4	99

The design of these tests, which only included adults at the beginning of the exposures indicates that these low CO_2 atmospheres may be controlling only eggs and/or young larvae and possibly some of the original adults at the 20 and 25% CO_2 concentrations.

These studies show that atmospheres above 10 or 15% CO_2 give a high degree of control. Certainly, these are the type atmospheres encountered in the gradual desorption of CO_2 by the grain and this gradual desorption continues to give control after the treatment has been terminated.

Condition of the insect

We have shown that insect life stage is an important factor in the length of time required for a given CA composition to produce adequate control of a species. Other factors that also play an impor-tant role is the condition of the insect and the availability of food. Table 13 presents data on the simulataneous exposure of normal and quiescent larvae of T. glabrum to four CO_2 atmosphere. The quiescent larvae were obtained by crowding them without food for 30 days prior to exposure. During a 48h exposure at 32°C the quiescent larvae were not as affected by atmospheres containing 60, 75, or 90% CO_2 as were the normal larvae but when they were exposed to 99% CO_2 they exhibited the same response (100% mortality) as the normal larvae.

Table 13 - Mortality (%) of normal and quiescent T. glabrum larvae when exposed to four CO_2 atmospheres for 48-h at 32°C and 49% r.h.

$\%CO_2$	% Mortality	
	Normal	Quiescent
60	39	16
75	47	28
90	94	52
99	100	100

CONCLUSIONS

Table 11, if presented in its entirety, would show the effects of 8 CAs (and air) on 2 species of stored product insects and could be expanded to show the individual effects on 1, 2, 3, 4 and 5 week old immatures of these insects. These studies as they now stand have statistically 479 total degrees of freedom for each species and are probably two of the most complete laboratory studies ever conducted in this area. However, this research was conducted at only one temperature and one r.h. and the effects of the atmospheres on eggs and adults was not considered. Also, the exposure times tested were not long enough to achieve data on 100% mortality. A complete laboratory study on these or any other species should include exposure at several temperatures and relative humidities and the length of exposure should be expanded until complete mortality is obtained. There is a need for data such as this for each of the economically important stored-product insects. More research is needed on the mode of action of CA on the insect at the several concentrations recommended for use in actual field situations. Adsorption and desorption of CO_2 by grain should be studied further, particularly as it relates to the immature insects feeding inside the kernel. Additional research is also needed on the effects of CA on diapausing or quiescent stages of stored-product insects. When this information is obtained it should be translated into a format to be used by industry in a practical manner to achieve effective control in the field.

The knowledge available at the present time provides some guidelines for treating grain with CO_2 in grain or oilseed storage bins or silos in a grain marketing system like the U.S.A. employs. If the grain to be treated is at or below 14% m.c. and at or above 27°C and if it does not contain Trogoderma spp. then a concentration of ca. 60% should be attained and maintained for 5 to 6 days if it is infested only with internal feeders. If external feeders are present a concentration as near to 100% CO_2 should be maintained for this period. Grain below 27°C and/or having a high m.c. or infested with Trogoderma should be treated for longer periods to obtain satisfactory control. Grain infested with species which have little or no research conducted on them, such as Sitotroga and Cryptolystes spp, should be treated in a similar manner and carefully sampled after treatment to determine the degree of control obtained.

The research necessary to eliminate the current inconsistencies and to provide a complete overview for the use of CA for stored-product insect control may have been brought up to date by this data. However, this research should be completed so that this control technique can become a viable alternative to conventional, residue-producing chemical fumigation.

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WHERE IS CA STORAGE GOING IN DEVELOPING COUNTRIES?

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ABSTRACT

The paper considers the possibilities for CA grain storage in developing countries. The efficiency of current and traditional storage systems on farms and at national level are reviewed. Traditional unimproved farm stores are found to be generally efficient with low losses; On more developed farms losses are higher, but except in underground pits and where metal containers are used, it is considered more practical to improve conventional storage practices than to attempt to introduce an alternative system. Losses in national storage systems are variable and it has proved difficult to sustain an improvement in conventional store management to contain losses. Storage practices which were satisfactory for short periods are proving unsatisfactory for long term storage. Increasing resistance to insecticides and fumigants is causing concern although simple trials using inert gas as disinfestants are proving successful. Suggestions are made for the implementation and design of CA storage at national level and for regional reserve stocks.

INTRODUCTION

The storage of grain to provide a continuous supply of food between harvests has been practised for as long as cereal grains have been cultivated. The methods of storage adopted in any particular location would, no doubt, have evolved from trial and error methods using locally available resources and the indigenous skills of the farmer. It is claimed that underground storage has been an important method for storage of grains, if not one of the principle methods, in the main cereal growing societies for upwards of 9000 years (Gilman & Boxall, 1979). It is also suggested that in Roman times airtightness in underground storage was identified as a significant factor in grain preservation, although it is probably not until the early 19th Century that this fact was generally accepted (Sigant, 1981). Clearly, therefore, some form of controlled atmosphere has been practised for several millenia in what must have been, for the period, advanced societies, but without the appendages of modern technology.

In most developed countries, underground storage is not practised today, probably because agriculture has adopted new concepts of storage containers and mechanical handling from industry which are better adapted to above ground storage. Nevertheless, modern storage structures have been used for many years to provide airtight storage on a large scale. Recent developments in using modified atmospheric gas concentrations for the control of stored products insects and undesirable microbiological development is clearly a natural progression from airtight storage.

The paper attempts to formulate possibilities for the application of controlled atmosphere storage in developing countries as a continuing process of development and progression to meet the changing circumstances of food grain storage. To do so it is necessary first to examine the situation in these countries at the present time with respect to grain production, movement and storage.

The total food grain production in developing countries is a little short of 500 million tonnes. Only a small proportion of this leaves the farm for marketing. Various estimates suggest that around 75% or about 300 million tonnes will remain on farms or within small communities for local domestic consumption; 120 million tonnes pass into national marketing systems, the great majority being operated by parastatal organisations. Food production in developing countries is generally inadequate to meet requirements and the gross import of food grains into deficit countries reached 105 million tonnes in 1981. (There were exports of about 30 million tonnes, principally from Latin America.)

Looking to the future, FAO has calculated population, production demand and per caput production trends up to AD 2000 (FAO 1981). In Africa and the Near East, demand will rise at a faster rate than production, requiring continually increased imports. In other areas, demand and production is calculated to rise in line with each other. The implication of these predictions is that developing countries will probably increase their dependence upon imports of food grains, either as direct purchases or as food aid.

The situation is, therefore; that farming communities retain about 370 million tonnes for their own consumption; 225 million tonnes of home produced and imported grains are handled, stored and marketed to both rural and urban populations, largely by national parastatal organisations; these amounts are likely to increase during this and the next decade. The need and the potential for the development of efficient and effective storage systems is very considerable.

FARMER STORAGE SYSTEMS

Farmers' storage systems are extremely diverse. They are developed largely through the interaction of the climate, the agricultural system, and the availability of local resources. Farmers in dry areas are considered to have fewer storage problems than those in wetter and more humid areas where storage must be preceded by a drying period, or the storage system must incorporate a facility to dry. Conversely, farmers in dry areas have only one crop each year (unless multicropping with irrigation) and have to store for 11 months while farmers in more humid climates generally have a range of alternative food crops, frequently have two crops each year and therefore need to store grains for only 4-5 months.

There are, however, essential common elements in all farmers' situations:

(a) Credit or investment in improvements is often expensive and there is competition for improvements and development throughout the farming system;

(b) There is also competition for the farmers' own managerial capabilities and undue emphasis on changes in one sector of the farming programme may distort attention away from other and equally important sectors;

(c) Although there are well recorded exceptions, farmers are generally given poor support by extension services in the post-harvest sector. The Service itself may be sparse, poorly organised, but generally extension workers' education and training has been directed towards crop production and crop preservation has been neglected;

(d) The difficulty of maintaining inputs into the farming system. Insecticides provides a classical example; insecticides are frequently proven to be effective for a particular situation but the cost and the organisation to pack, distribute and monitor the insecticide is too costly for a commercial operation and beyond the capability of the local resources. (McCullum Deighton, 1981).

The efficiency of farmers' storage systems has been given much consideration recently. Reliable data on farmers' storage losses are very sparse except where specific situations have been identified. Many of the estimates of physical loss are based on experimental work in laboratories or small scale field trials which are extrapolated to what are believed to be normal post-harvest practices. Frequently the figures quoted are either an upper extreme or an unqualified average. Whilst there is little justification for using this data to determine the need for remedial measures to reduce losses they have, however, drawn attention to the variety of losses that can occur and how these can be measured. Direct weight loss is the simplest, the one most amenable to measurement and the kind of loss most often quoted.

The FAO Prevention of Food Losses Programme has established that the general levels of total post harvest losses on farms of staple foods such as cereals is around 12% (Huysmans, 1982). Detailed studies have indicated that in traditional farming systems using unimproved varieties of cereals, storage losses can be as low as 3% (Greeley, 1980) and are generally in the range 5-8%. It can be argued that at this level of loss the resources needed to reduce these losses even further may be more effectively employed in other sectors of the farming system. Losses are known to be higher when so-called improvements are introduced into the traditional system, of which multicropping with irrigation and new high yielding varieties are the most significant. Nevertheless Golob (1981) found that in well managed farmers' stores the use of insecticides to prevent loss did not yield an economic return when storing hybrid high yielding varieties of maize.

Most losses so far discussed are caused by insects but losses also arise from storage at too high moisture contents which are much more difficult to quantify. Drying of crops at farm level is a most intransigent problem. Considerable resources have been expended on the development of farmers' drying systems with remarkably little result. Where it can be practised, sun drying is quick and effective, but elsewhere the most promising developments are those based on traditional air drying systems which require exposure of the crop to ambient air for several weeks or months.

Attempts have been made to improve farmers' storage including the use of plastic liners for the ubiquitous gunny bag to provide a gas tight container for fumigation by liquid fumigants, concrete structures for ventilated and airtight storage, and small flexible silos for airtight storage (O'Dowd, 1971). Rarely are these improvements acceptable or sustained once the initial enthusiasm for them has waned or the Government or donor financial support has been removed (Andrews, 1973). Connell (1974) comments that it is extremely difficult to develop a satisfactory economic argument in favour of action in attempting to solve a stored product insect pest problem. It can be very difficult to persuade a farmer to seek credit for insecticides or other inputs, particularly for grain to be marketed, if he is not to be penalised for a modest infestation or be given a bonus for maintaining grain in good condition.

For this reason there seems little prospect or indeed incentive in the forseeable future, to attempt anything more sophisticated for above ground storage systems than to ensure that the traditional systems are properly managed and modified as necessary to accommodate changes in farming systems, improvements in the flow of agricultural inputs and developing technology.

Underground or pit storage occupies a somewhat anomalous position. It is traditional in many areas (Gilman and Boxall, 1974) and whilst performance is somewhat variable it can, as airtight storage, provide an effective protection against insects and rodents over long periods. There have been a number of instances when attempts have been made to reintroduce pits in some areas, but the general impression seems to be that underground storage is on the decline, albeit slowly. It may be speculated that this is due to the greater need for drying newer varieties, the improvement of resources for building above ground stores and distributing agricultural inputs, including insecticides; and also because of an increasing antipathy towards the labour needed to build and empty pits. It is a system clearly appropriate to the drier areas where rainfall is erratic and where there may be a need to store food for several seasons as a precaution against crop failure. The depletion of oxygen is, however, alleged to be closely associated with soil moisture, which moves into the grain from the surrounding soil. Mould damage to grain adjacent to the pit sides and on the surface of the grain bulk is, therefore, to be expected. Efforts have been made to line traditional storage pits but none of the methods adopted appear to entirely exclude moisture. In Ethiopia, where underground storage is well established in some areas and where losses due to water damage can be considerable, waterproof concrete liners have been introduced into traditional pits with some success and this improvement appears to have been welcomed by local farmers (Boxall, 1973).

Metal bins have developed recently as non traditional farmers' storage structures in Southern Africa and parts of Central and Southern America. In Africa particularly, they were adapted from water tanks, the manufacture of which had developed as a significant industry. Because of this, the bins were relatively cheap compared to traditional stores. Additionally, they prevented rodent infestation and reduced exposure to insect infestation. Pre-drying of produce is essential if moisture damage is to be prevented. In well managed stores, insecticide is used and there is apparently a concept of using the bins as airtight storage (Giles 1983). However, the method and standard of manufacture are frequently inadequate to ensure air tightness. Fumigation of metal bins using phosphine is practised occasionally and appears satisfactory but there are no reports of the CT products attained.

In both improved pits and metal bins there is a possibility of improving storage conditions by using some form of controlled atmosphere at least as a disinfestation measure. The efficiency of the present systems will, however, offer significant competition to any alternatives until they are found to be inadequate or insects become resistant to the commonly available insecticides.

LARGE SCALE STORAGE

The storage functions of Government and parastatal marketing organisations include:

1. Storage of seasonal or operational stocks to meet seasonal demand and effect price stabilisation;

2. Maintenance of carry-over stocks between seasons;

3. The establishment and maintenance of strategic or longterm reserves against crop failures.

In all these operations, it is generally the marketing policy of Government which determines the scope and scale of the storage operation. Seasonal and carry-over storage involves the aggregation of locally procured produce into stores holding upwards of 1000 tonnes and storing for periods of up to twelve months for the majority of grain, but frequently for periods of up to 24 months. Heretofore, storage management has relied on good storage hygiene and the use of insecticides and fumigants to control insect infestation. Spoilage by moisture is not usually serious, largely because Government purchasing is limited to maximum moisture content standards, but the increasingly frequent reports of the incidence of toxins in stored products suggests that moisture content problems may be under-estimated.

The efficiency of these storage operations has not been given serious study, although it is known to vary widely, ranging from very good with very small losses, to very poor with almost total loss of stored commodities, at the two extremes. It is self evident that sound informed management is essential at all levels, supported by clear policies, adequate budgetary provisions for store maintenance and consumables and adequate training facilities for operational staff. Regrettably, it is all too common for some if not all of these requirements not to be met. In a survey of the use of flexible storage structures, O'Dowd and Kenneford (1983) found that unacceptable performance was largely a fault of the human factor rather than any fault in the structures themselves. An informal evaluation of the experiences of trainees at Tropical Products Institute suggests that the principal difficulty they face in applying improved storage practices is the unawareness of their immediate managers or the constraints placed on them by the unawareness of higher management or by the impedence of cumbersome and inflexible administrations.

An indeterminate proportion, but probably exceeding 95% of the grain, is stored in developing countries in bags. The stores themselves are generally of a conventional but quite variable warehouse design, ranging in size from 500 tonnes upwards. Design requirements are generally simple (Gracey and Calverley, 1979; Hayward, 1981). In spite of this, it is very difficult to ensure that stores are constructed to reasonable standards, not necessarily to the rigid costs of building practice current in developed countries but to standards that ensure the building is sound and will remain so during its operational life. Gracey (1981) outlines avoidable problems in the construction of new storage facilities and suggests that the rapid development of a modern storage system may overload the limited managerial resources of a developing country. Because of this there is a lack of attention to detail that ultimately results in serious deficiencies in the completed building; causing rapid deterioration and high maintenance requirements. Such defects have been noted by Gilman (1982) in buildings used for long-term storage in the Sahel.

The advantages of building stores sufficiently gas-tight for fumigation of the store and its contents have been appreciated for some time but the additional expenses for purpose-built stores have limited this development. Exceptionally, cocoa stores at Ikeja, Nigeria, with a total capacity of 90,000 tonnes, were built as gas-proof stores and are reputedly very sucessful (Riley and Simmons, 1967). In Kenya, stores of a more conventional design were adapted for fumigation by installing a ceiling, fitting non-operable windows and lining the walls. In practice these stores did not perform well and there has been no attempt to pursue this development. Elsewhere the simplicity of total store fumigation and, in some instances, its lower cost than other methods of disinfestation (Gilman, 1982) have led others to attempt this practise but with limited success and a failure to control insect pests (Champ and Winks, 1982). Taylor (1982) and Tyler *et al* (1982) investigated total store fumigation in Senegal and Bangladesh and in all cases noted that fumigants leaked rapidly through the porous brick and plaster walls of older stores. Modern, well constructed and better sealed stores provided better gas retention but this was less good than treatments applied under sheeted stacks within stores. This inadequate treatment has led directly to insects becoming resistant to phosphine and able to survive treatment.

There are some bulk handling installations in developing countries. These are to be found in some port installations and associated with processing industries. Generally, however, there are far too few of them and the units are too widely scattered to establish a national bulk handling system. Some authorities conclude that these installations are not fully exploited because the necessary infrastructure does not exist (Baehr, 1982). Nevertheless, there have been some successful bulk stores, designed and built primarily for long term airtight storage. Lopez (1973) describes the airtight underground stores in Argentina. He comments that many such silos are no longer airtight but give better storage conditions than above ground silos because of better thermal characteristics. Semi-underground air-tight stores in Cyprus and Kenya (the so-called Cyprus Bins) have not been without problems (De Lima, 1981), but have successfully stored wheat and maize for long periods.

Flexible walled sealed silos ("Butyl Silos") have also been used satisfactorily to store grain for relatively long periods. Insect infestation was well controlled whilst the airtightness of the fabric was maintained. This, however, required exacting standards of site management which was often not available and the silos have been found in practice to have a relatively short life (O'Dowd and Kenneford, 1983).

THE APPLICATION OF CA IN DEVELOPING COUNTRIES

There are compelling reasons why we should continue to monitor current storage practises and pest control problems. There is evidence that significant changes are taking place in farming practices; the extended use of irrigation; new breeds of cereal grains that have different, usually inferior, storage characteristics. More farmers are growing larger crops and storage practices, satisfactory for subsistence farmers' domestic requirements, may not be suitable for a significant marketable surplus. National marketing organisations are finding that its storage operations which were satisfactory for several months storage are inadequate when the storage period is extended into a year or more.

If current generally accepted recommendations on good storage practice and pest control operations were put into practice, there would be a most significant reduction of storage losses. Whilst historical evidence suggests that because of considerable inertia and resistance to change, this can never come about through the application of different operations and procedures, this should not deter a continued and sustained effort to improve present storage practices and encourage their adoption as far as possible.

Meanwhile, pest infestation problems continue to present themselves in different ways. Resistance to insecticides is now widespread involving all major pesticides and most of the important pests of cereal and cereal product storage (Champ, 1978). Control failures in the field have been unequivocally associated with resistance and have forced the use of some insecticides to be abandoned in some areas. With the now proven occurrence of significant resistance to phosphine in the field, Tyler et al (1982) suggest that the effectiveness of phosphine disinfestation procedures require close examination and in particular that phosphine fumigation should not be used as a palliative treatment. The translocation of pests to regions where there are no predators or natural checks raises particular and difficult problems of control (Golob and Hodges, 1982). Equally important, the longer periods of storage raise problems of the preservation of quality without increasing chemical residues to unacceptable levels.

There is no single solution to these problems and CA storage is likely to be one of the alternatives that can be applied as appropriate to a particular situation. The particular advantages of this system have been described at an earlier Symposium. Apart from extending simple airtight storage, CA storage is unlikely to have any application to farmers' storage systems, because the benefits to be gained are insufficient to justify the very considerable effort needed to establish and maintain a system requiring such a level of technology.

A modified form of C.A. storage has been shown to be effective for the storage of milled rice in Indonesia. Stacks of 200 tonnes were covered with transparent plastic sheets and sealed. CO_2 was introduced as a fumigant gas after evacuating the air with a vacuum pump. After eight months storage there were no living insects visible through the plastic sheet and the quality of the rice has been found to be unchanged. No data is available on the levels of CO_2 established in the stack and for how long these were maintained. The trials continue. Costs of the system are not available but it is reported to be considerably more than a single fumigation with phosphine and it is therefore more suited to longer term storage.

The apparent success of these trials warrants further study and similar development trials could be carried out wherever there are locally available supplies of insert gas, and an organisation capable of carrying out scientific work of this nature.

The establishment of a CA storage in purpose built, or modified stores, with continuously maintained levels of atmospheric gases as described by Banks and Annis (1980) has, *prima facie*, little advantage over seasonal bag storage systems in developing countries where in most cases absolute control of insects is not essential. It is more important to ensure that losses during and arising from the storage period, are contained within economic or otherwise acceptable levels. Costs are difficult to apply generally, for example the cost of a store building in Tanzania is approximately twice the cost of its equivalent in Kenya; in Mali it is more than three times and in Nepal more than five times.

Costs analysis are therefore likely to be very location specific. Donor organisations contribute a substantial proportion of the investment in grain storage facilities and, since it is highly probable any CA storage development in a developing country will be similarly aided, some form of cost benefit analysis of the system will be required.

A decision to establish a CA system would requir-, in the first instance, a clear identification of an appropriate situation where its particular advantages could be fully exploited. It will also need a firm commitment by both Government and donor to the project as, in the first instance, a development project. The problems of construction, management and supporting organisation which are all too frequently encountered in the development of storage facilities and which have already been referred to, need to be recognised from the outset and a strategy developed to ensure they are avoided or at least mitigated. The project will almost certainly require strong managerial and technical support from Government and donors for significantly longer than is customary with grain storage projects and the timescale should be measured by the attainment of a successfully operating and self sustaining commercial operation.

There are a number of design parameters which should also be considered in addition to general design requirements outlined by de Lima (1980b) and Banks and Annis (1980).

Function

It would be prudent to design stores so that if needed they could be used successfully with more conventional bag storage systems. This would include making provision for ventilators in those areas where they would normally be recommended, providing storekeepers' offices and provision for a fumigable store for bags and a store for chemicals.

Size

Transport to and from storage sites is frequently very difficult to organise properly and rates of grain movement are often below expectation. This has consequences in the time taken to fill and empty stores during which time protection for the grain will be reduced. Individual stores should therefore not exceed 5,000 tonnes capacity.

Acceptable Moisture Contents

A survey on flexible sealed silos ("Butyl Silos") indicated clearly that the principal cause of failure was storing at too high a moisture in conditions which did not permit ventilation or the removal of moisture from the grain by diffusion or along vapour pressure gradients. Some ventilation of CA stores is inevitable through leakage and the balancing of atmospheric pressures, but it should be clearly established for each crop and locality what maximum moisture contents would be tolerable.

The World Food Council and other UN agencies have outlined a number of proposals for the establishment of regional grain reserves in chronically grain deficient areas to avoid the hasty and often expensive arrangements for contingency aid when famine is imminent. There are many practical difficulties in such proposals, including in particular the difficulty of recycling stocks through national markets without disturbing normal marketing operations. The capability to maintain stocks in sound condition in many instances in a hot, and therefore hostile, environment over long periods, is an obvious requirement, and one that could be satisfied by CA storage. Reserve stores would need to be under international management and the problems of ensuring adequate technical and management support should be more easily dealt with. In such a situation the alternatives of bag and bulk storage and, if bulk, what form the structures should take, are legion. It is important that all alternatives are fully explored and that new ideas are not rejected simply because they are novel. The choice will be decided on political and financial as well as technical grounds and these must ensure that the system which is selected should be feasible, economical and meets the needs of people for whose benefit it is intended.

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CURRENT METHODS AND POTENTIAL SYSTEMS FOR PRODUCTION OF CONTROLLED ATMOSPHERES FOR GRAIN STORAGE

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Abstract: In controlled atmosphere storage, the atmosphere around the stored grain is modified to give an environment rich in CO_2 (> 40%) or low in oxygen (< 1%). At present tanker-delivered liquified gases (nitrogen or CO_2) are usually used to create these atmospheres but there are several other processes which appear suitable. These include pressure swing absorption systems, burners fueled with propane, producer gas, biogas or hydrogen, various forms of enhanced hermetic storage systems, fermentation processes and electrolytic or photolytic oxygen removal systems. Many of these techniques utilise materials available from sources close to or at the storage site, a major advantage over tanker delivered gases which may have to be transported over long distances from point of production. The rates of supply required to create and maintain five possible atmospheres are given. The atmospheres considered are low oxygen systems (< 1%) in nitrogen, or nitrogen/CO_2 mixtures, high-CO_2 (> 70%) and intermediate-CO_2 (ca 40%) mixtures, and atmospheres similar to those found in hermetic storages (< 6% O_2, > 14% CO_2). The various potential techniques of gas production are discussed in terms of their mode of operation, fuel and electricity requirements, scale of application and capital cost. It is concluded that on-site processes for gas generation should generally supersede use of tanker delivery and that eventually controlled atmospheres could be generated by a controllable form of the ancient hermetic storage technique.

1. INTRODUCTION

Controlled atmosphere (CA) treatment is now an established technique for the protection of stored grain. It is in limited commercial use in several parts of the world: e.g., Australia (Banks *et al.*, 1980), China (Champ *et al.*, 1982), Italy (Tranchino *et al.*, 1980) and continues to attract development interest in others: e.g., Bulgaria (Stoyanova and Shikrenov, 1979), Nigeria (Adesuyi, *et al.*, 1980), USA (Jay, 1980) and USSR (Anon., 1974). There are three major impediments to its more widespread adoption: the need for well-sealed storage, the long exposure times required for complete effectiveness and the lack of cheap convenient methods for supplying the gases required. Methods for provision of suitability modified storages are currently receiving much attention and with considerable success (e.g. see papers in this conference). The long exposure time appears to be an inherent restriction of the technique. This paper is aimed at illuminating and, perhaps, providing some of the ideas that may help to remove the third impediment, the lack of appropriate methods of gas supply. The discussion is largely limited to the use of CA for insect control in dry grain (< 12% moisture content) although the principles involved in the gas generation systems can usually be extended to CA use with grain at higher moisture content.

Currently, the gases, nitrogen or CO_2, required to create and maintain the atmospheres required for CA storage are supplied either as liquified gases in insulated tankers for large scale treatments or as compressed gas in cylinders or as dry ice for small scale application. We must ask whether these methods of supply are the ones of choice, or are better methods available, but just not used, and what alternatives appear feasible for use in the near future.

Supply of the gases by tanker or in cylinders is convenient. However, it must be difficult to justify economically the transport of gases, often over long distances, from some industrial production centre to the storage. This procedure requires expensive specialised vehicles or heavy cylinders and may contribute a significant part of the overall cost of CA use (e.g. see Banks and Annis, 1977; Connell and Johnston, 1981). However, the storage itself is surrounded both by inexhaustable supplies of nitrogen in the air and large quantities of carbon-containing material, agricultural wastes and even the grain itself, suitable for use as a CO_2 source. Methods of CA used in the future must surely include the generation of the required gases on site.

Research is already under way towards the replacement of the use of tanker gases and the development of on-site processes for gas supply for CA use. There have been successful full-scale field trials using atmospheres generated by burning hydrocarbons in air to provide a low oxygen gas mixture (e.g. Storey, 1973; Navarro *et al.*, 1979). There has been research on ways to improve the ancient and elegant technique of hermetic storage so that it can be used in above-ground structures (Burrell, 1980; Lu, in press). However, it is not widely realised how many different potential methods there are for the generation of the required gases. Some methods of production of nitrogen on site and their economics and scope of application were discussed by Zanon (1980). In this paper, I present a synopsis of the requirements a gas generation system must meet and then a survey of some possible methods†. The systems considered include both those producing pure nitrogen or CO_2 and also mixtures of these two gases. Data is given on the energy or fuel requirements of the processes. However these requirements will vary widely with storage capacity, and location and methods of energy accounting. Thus the values given must be taken as guides only. I hope that this speculative exposition may stimulate consideration of the various methods available and contribute towards the continued development of CA techniques as valuable tools for grain storage and protection.

2. SPECIFICATION FOR GAS SUPPLY FOR CA USE

In order to be able to judge the suitability of a particular gas production process, it is necessary first to have a specification for the basic parameters: the composition of the gas and its rate of supply. Unfortunately, neither of these are well defined even now, despite many decades of interest in CA techniques. The specifications are likely to change with experience and research in the future.

2.1 Composition

Research on the effects of various gas compositions (combinations of nitrogen, CO_2 and oxygen) on insects, microorganisms and the grain, has lagged behind the development of methods of applying these atmospheres in the field. We are now more certain of how to create and maintain a given composition than of the optimum atmosphere itself. There are several important problems yet to be resolved which may have a bearing on what is considered the optimum strategy in the future. In particular:

(i) It may be possible to use atmospheres containing several percent O_2 against insects if the exposure period is prolonged (e.g. several months).

(ii) The influence on insects of the CO_2 concentration in low oxygen atmospheres is not well understood or quantified.

(iii) There is little data on the effect on insects of long exposures at < 35% CO_2 in air.

† Suppliers of equipment are given for example only. The equipment cited has not been tested by the author and there may be other suppliers of similar equipment which may or may not be more suitable for the purpose suggested.

(iv) There is insufficient data on quality effects and mould control to determine which gas mixtures are most suitable for use at > 60% equilibrium relative humidity.

(v) There is no data on whether a particular composition should be created slowly or rapidly for optimal insecticidal effect.

(vi) It has not been determined if CO_2 is detrimental to the structural strength of reinforced concrete storages by promoting the rusting of the reinforcing steel through carbonation of the concrete.

Against this uncertain background, the specifications given here (Table 1) for exposure periods and gas compositions cannot be taken as definite. Indeed two of the atmospheres discussed here are presented because I believe that similar atmospheres will be useful in the future. However, the scientific evidence on which this belief is based is indicative rather than conclusive.

2.2 Rate of supply

There are two separate phases in the use of CA techniques: an initial phase in which the desired atmosphere is created in the storage and a subsequent one in which the composition is maintained within set limits for the exposure period. These are referred to here as the 'purge' and 'maintenance' phase, following Banks and Annis (1977). The purge phase is taken here to include the creation of the desired atmosphere either by the direct addition of gas or by recirculation of the storage atmosphere through some device which modifies the composition appropriately (Fig. 1). The gas requirements for the two phases are summarised in Table 1.

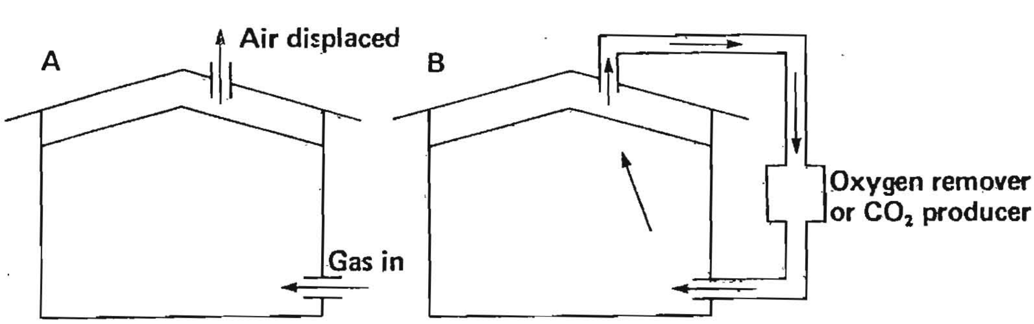

Fig. 1. Purge phase of the CA process. CA creation using a direct purge (A) or by recirculation (B).

TABLE 1.

Requirements for various controlled atmospheres, full storage in well sealed storages (pressure decay test time, > 5 mins, 500-250 Pa).

Type of atmosphere	Target composition	Exposure period	Purge requirements	Maintenance requirements	Status of specification	Background References
Nitrogen-based atmosphere	< 1% O_2	> 24 days (25°C)	1-2m^3t^{-1} added very rapidly (< 1/2 day)	0.01-0.06m^3t^{-1} pure N_2 or remove 0.005-0.02$m^3t^{-1}d^{-1}$ O_2	Largely based on field experience with some laboratory data	Banks and Annis (1977)
Nitrogen-CO_2 mixture	< 1% O_2 with > 10% CO_2	> 20 days (25°C)	1-2m^3t^{-1} added rapidly (< 2 days)	0.02-0.12 m^3 t^{-1} d^{-1} of gas at < 0.3% O_2	Based on nitrogen specification with allowance for increased effect from CO_2	Bailey and Banks (1980)
Hermetic storage atmosphere	< 6% O_2 with > 14% CO_2	> 2 months	0.1-0.3m^3t^{-1} O_2 to be removed over 10 days	0.03-0.01m^3t^{-1} d^{-1} O_2 to be removed	Speculative	Spratt (1979 a,b)
High-CO_2 atmosphere	> 70% CO_2 [a] in air	> 10 days	0.5-1m^3t^{-1} added rapidly (< 2 days)	None	Based largely on field experience	Banks (1979)
Intermediate-CO_2 atmosphere	> 40% CO_2 in air	> 30 days	0.3-0.5m^3t^{-1} added over 10 days	0.02-0.04m^3t^{-1} d^{-1} pure CO_2	Speculative	Stoyanova and Shikrenov (1976) Lindgren and Vincent (1970)

a Initial target composition. May decay to lower values during exposure period (see Banks, 1979).

The rates of gas supply for the purge and maintenance phases differ substantially. The ratio of these rates is typically between 10:1 and 100:1. At present the same gas supply process is used for both phases, but there is no reason why this should necessarily be so. Indeed, it may often be a better strategy to provide a system for purging, capable of high rates of gas output, that can be moved from site to site, and a much smaller system for the maintenance phase, dedicated to one site or storage unit only.

3. TECHNIQUES FOR GENERATION OF GASES FOR CA USE

The techniques for gas generation for CA use can be divided into three categories according to the gas mixture produced:

(i) Nitrogen producers (< 1% O_2).

(ii) Producers of a nitrogen-CO_2 mixture (< 1% O_2), including hermetic storage systems.

(iii) CO_2 producers.

The techniques available for production of these types of gas mixture are summarised in Tables 2, 3, and 4 respectively, together with comment on the scale of applicability, development status and energy or material consumed in course of supply of the gases. The energy and fuel requirements are calculated from a variety of sources, largely descriptive technical leaflets, and from data given by gas supply companies. No allowance is made for the energy cost of making the apparatus giving the gas. Hermetic storage systems are included in the category with producers of nitrogen-CO_2 mixtures, since similar principles of operation are involved. However, hermetic storage systems may not produce as low oxygen tensions as nitrogen-CO_2 producers (up to about 8% O_2 in some cases (see Banks, in press) compared with < 1% O_2).

Many systems are in use or obviously feasible but untried (e.g. pipeline supply). No detailed comment will be given here on these methods. The merits and principles of operation of the other systems are given below.

3.1 Potential systems for nitrogen production

3.1.1 *From exhaust from burning hydrocarbons in air.* Nitrogen can be extracted from the low oxygen exhaust stream produced by burning hydrocarbons in air (Zanon, 1980). CO_2, water and other impurities produced during combustion, can be removed by absorption from the gas stream on molecular sieve. A transportable plant producing 40 m^3

TABLE 2.

Techniques for producing or maintaining a nitrogen atmosphere (< 1% O_2).

Technique	Phase best suited	Capital cost [a]	Scale of application best suited [b]	Development status	Primary energy or fuel requirement	
					Purge phase (per tonne grain) [c]	Maintenance phase (per tonne grain per day) [d]
Liquified gas supplied by tanker and stored on site	Purge or maintenance	Very low	Intermediate to large	In use (Tranchino *et al.*, 1980; (Banks *et al.*, 1980)	4.3 MJ + 0.4 mL diesel km^{-1} [f]	0.08 MJ + 0.0007 mL diesel km^{-1}
Compressed gas supplied in cylinders	Purge or maintenance	Very low	Small	In use (Champ *et al.*, 1982)	3.4 MJ + 0.24 mL diesel km^{-1}	0.06 MJ + 0.009 mL diesel km^{-1}
Air separation on site (cryogenic)	Purge or maintenance	High	Very large	Feasible	4.3 MJ	0.08 MJ
Pipeline supply from cryogenic separation	Purge or maintenance	Low or intermediate	Intermediate to very large	Feasible	3.3 MJ	0.06 MJ
PSA	Purge or maintenance	Intermediate	Intermediate to large	Plant available	1.8 MJ	0.03 MJ
Nitrogen generation from hydrocarbon combustion	Purge	Intermediate	Intermediate to large	Plant available	130 g C_3H_8 + 1.1 MJ	2.5 g C_3H_8 + 0.02 MJ
Oxygen removed by hydrogen combustion [e]	Maintenance	Low	Small to Intermediate	Requires development	0.3 m^3 H_2	0.004 m^3 H_2
Catalytic oxidation of ammonia [e]	Maintenance	Low	Small to Intermediate	Requires Development	120 g NH_3	1.6 g NH_3
Direct electrolytic removal of oxygen [e]	Maintenance	Low	Small to Intermediate	Speculative	4.7 MJ	0.06 MJ
Chemical absorption of oxygen [e]	Maintenance	Very Low	Small	Speculative	Remove 6 g-mol O_2	Remove 0.08 g-mol O_2

a Capital cost assessed as approximate cost to use on the following rating per site of 10,000 t: very low < \$5,000, low = \$5,000 - \$20,000, intermediate = \$20,000 - \$100,000, high > \$100,000.
b Rated according to the following approximate scale: small < 300 t, intermediate, = 300 - 10,000 t, large = 10,000 - 100,000 t, very large < 100,000 t. Allowance made for installation cost in assessment.
c Calculated for a filled 2,000 t capacity bin of total gas volume 1100 m^3, and headspace of 145 m^3 with 90% efficiency (E_3) of purging by direct introduction and 100% efficiency (E_1) by recirculation (E_3 and E_1 as Banks (1979)).
d Calculated for a filled 2,000 t capacity bin as *c* with input rate of 15 L t^{-1} d^{-1} for direct input, 7.5 L t^{-1} d^{-1} for recirculation.
e With recirculation.
f Transportation requirement based on 0.63 L diesel km^{-1} for a 20 t gross load.

TABLE 3.

Techniques for producing or maintaining atmospheres of < 1% O_2 (< 2% for hermetic systems) with 10-20% CO_2, balance nitrogen

Technique	Phase best suited	Capital cost [a]	Scale of application best suited [b]	Development status	Primary energy or fuel requirement: Purge phase (per tonne) [c]	Maintenance phase (per tonne per day) [d]
Combustion of hydrocarbons from off-site sources ('open-flame')	Purge or maintenance	Intermediate	Intermediate	Demonstrated in field trials (Storey, 1973)	66 g C_3H_8 + 0.03 MJ	1.2 g C_3H_8
Combustion of hydrocarbons from off-site sources [e] (catalytic)	Purge or maintenance	Intermediate	Intermediate	Demonstrated in field trials (Navarro *et al.*, 1979)	47 g C_3H_8 + 0.15 MJ	0.6 g C_3H_8
Combustion of hydrocarbons from off-site sources (internal combustion engine)	Purge or maintenance	Low	Intermediate	Plant available but not tested in field (Banks, unpublished)	66 g C_3H_8	1.2 g C_3H_8
Liquified gases supplied by tanker	Purge	Very low	Intermediate to large	Feasible	3.0 MJ + 0.03*f* mL diesel km^{-1}	0.08 MJ + 0.0008 mL diesel km^{-1}
Compressed gases supplied in cylinders	Maintenance	Very low	Small	Feasible	2.4 MJ + 0.17 mL diesel km^{-1}	0.06 MJ + 0.008 mL diesel km^{-1}
Producer gas combustion	Purge or maintenance	Intermediate	Intermediate	Requires development	66 g C	1.6 g C
Biogas combustion	Maintenance	Intermediate	Intermediate	Requires development	74 g $C_6H_{12}O_6$	1.4 g $C_6H_{12}O_6$
Simple hermetic storage	Purge and maintenance	Very low	Small to intermediate	In use (Hyde *et al.*, 1973; Sigaut, 1980)	150 g $C_6H_{12}O_6$	1.8 g $C_6H_{12}O_6$
Assisted hermetic storage	Purge and maintenance	Very low	Small to intermediate	In use (Lu, in press)	150 g $C_6H_{12}O_6$	1.8 g $C_6H_{12}O_6$
Hermetic storage with sacrificed areas	Purge and maintenance	Very low	Small	Demonstrated in field trials (Burrell, 1980)	150 g $C_6H_{12}O_6$	1.8 g $C_6H_{12}O_6$

a Capital cost assessed as approximate cost to use on the following rating per site of 10,000 t: very low < \$5,000, low = \$5,000 - \$20,000, intermediate = \$20,000 - \$100,000, high > \$100,000.

b Rated according to the following approximate scale: small < 300 t, intermediate, = 300 - 10,000 t, large = 10,000 - 100,000 t, very large < 100,000 t. Allowance made for installation cost in assessment.

c Calculated for a filled 2000 t capacity bin of total gas volume 1100 m^3, headspace 145 m^3, with a 90% efficiency of purging (E_1) by direct introduction and 100% efficiency (E_1) by recirculation (E_1 defined as in Banks (1979)).

d Calculated for a filled 2000 t capacity bin as *c* with input rate of 15 L t^{-1} d^{-1} for direct input, 7.5 L t^{-1} d^{-1} for recirculation.

e With recirculation.

f Transportation requirement based on 0.63 L diesel oil km^{-1} for a 20 tonne gross load.

TABLE 4.

Techniques for producing or maintaining high- or intermediate-CO_2 atmospheres.

Technique	Phase best suited	Capital cost [a]	Scale of application best suited [b]	Development status	Primary energy or fuel requirement: To make 70% CO_2 atmosphere (per tonne) [c]	Primary energy or fuel requirement: To maintain 40% CO_2 atmosphere (per tonne per day) [d]
Liquified gas supplied by tanker and stored on site	Purge or maintenance	Very low	Intermediate to large	In use (Jay, 1971; Banks *et al.*, 1980)	0.7 MJ + 0.03 [e] mL diesel km^{-1}	0.03 MJ + 0.0013 mL diesel km^{-1}
Compressed gas supplied in cylinders	Purge or Maintenance	Very low	Small	In use (Champ *et al.*, 1982)	0.7 MJ + 0.07 mL diesel km^{-1}	0.03 MJ + 0.003 mL diesel km^{-1}
Pipeline supply	Purge or maintenance	Low to intermediate	Intermediate to very large	Being installed	0.05 MJ	0.002 MJ
Supplied as dry ice	Maintenance	very low	Small	In use (Sharp and Banks, 1980)	1.8 MJ + 0.03 mL diesel km^{-1}	0.08 MJ + 0.0013 mL diesel km^{-1}
Production on site from hydrocarbon combustion in oxygen	Purge or maintenance	Intermediate	Intermediate	Requires development	300 g C_3H_8 or 610 g $C_6H_{12}O_6$	40 g C_3H_8 or 90 g $C_6H_{12}O_6$
Production on site from carbon combustion in oxygen	Purge or maintenance	Intermediate	Intermediate	Requires development	240 g C	9 g C
Recovery from CO_2 containing gas streams	Maintenance	Intermediate	Intermediate	Plant available	varies with process	varies with process
Anerobic fermentation	Maintenance	Low or intermediate	Small to intermediate	Requires development	610 g $C_6H_{12}O_6$	40 g $C_6H_{12}O_6$

a Capital cost assessed as approximate cost to use on the following rating per site of 10,000 t: very low < \$5, low = \$5,000 - \$20,000, intermediate = \$20,000 - \$100,000, high > \$100,000.

b Rated according to the following approximate scale: small < 300 t, intermediate, = 300 - 10,000 t, large = 10,000 - 100,000 t, very large < 100,000 t. Allowance made for installation cost in assessment.

c Calculated for a filled 2000 t capacity bin of total gas volume 1100 m^3 and headspace 145 m^3, with 80% efficiency of purging (E_1).

d Calculated for a filled 2000 t capacity bin as *c* with a gas interchange rate of 0.05 day^{-1}.

e Transportation requirement based on 0.63 L diesel oil km^{-1} for a 20 tonne gross load.

h^{-1} is available commercially (Kemp, Glen Burnie). The technique suffers from a high degree of mechanical complexity and has probably been superseded by the PSA system (see below).

3.1.2 *Pressure swing absorption.* The pressure swing absorption (PSA) system using air as feedstock. The process involves cycles of compression and decompression of the gas mixture in the presence of a carbon that possesses molecular sieve properties and relies on the difference in rate of absorption and desorption of nitrogen and oxygen in the carbon to achieve separation (Zanon, 1980). Nitrogen with < 0.3% residual oxygen can be conveniently produced by the process.

As in the case of nitrogen production from exhaust gases, the PSA systems are mechanically complex. Nevertheless both systems are capable of on-site use. Both produce gas more cheaply than from the usual cryogenic air separation system for nitrogen production (Zanon, 1980) and show promise as ways of obtaining nitrogen for CA use. There are several manufacturers of equipment apparently suitable for grain storage applications (e.g. Gastec, Salinas; Kemp, Glen Burnie; Techsep, London).

3.1.3 *Catalytic oxidation of ammonia.* The aerial oxidation of ammonia gas over a noble metal catalyst can be used to remove oxygen from air to give a low oxygen gas stream containing water according to the approximate equation.

$$\underset{\substack{\text{Ammonia} \\ \text{(1 vol)}}}{NH_3} + \underset{\text{air (3.6 vols)}}{0.75\ O_2 + 2.8\ N_2} \longrightarrow \underset{\substack{\text{nitrogen} \\ \text{(3.2 vols)}}}{3.2\ N_2} + 1.5\ H_2O$$

The process has been proposed for producing atmospheres for CA storage of fruit (Anon., 1971). Figures for minimum oxygen levels produced are not available but could be very low. For use with grain, the process would need development to ensure that no nitrogen oxides or residual ammonia were added to the storage and the water was removed.

This process appears worthy of close consideration as a method of maintenance for nitrogen atmospheres. Anhydrous ammonia for use as a fertilizer is available in many grain growing regions. It is easily stored with low toxicity and flammability risks. However, the production of ammonia requires substantial energy input. Transport costs may also be important.

3.1.4 *Hydrogen combustion* Combustion of hydrogen in air removes oxygen with the formulation of water according to the approximate equation:

$$\underset{\substack{\text{Hydrogen} \\ \text{(1 vol)}}}{H_2} + \underset{\substack{\text{air} \\ \text{(2.4 vols)}}}{0.5\ O_2 + 1.9\ N_2} \dashrightarrow \underset{\substack{\text{nitrogen} \\ \text{(1.9 vols)}}}{1.9\ N_2} + H_2O$$

The process can be carried out in a direct combustion system or, more conveniently, over a catalyst. Industrially-produced hydrogen supplied in cylinders could be used for removing oxygen from an atmosphere by recirculation through a combustion system. However transport costs for the heavy and bulky cylinders containing the hydrogen would probably rule this out. However, hydrogen does appear potentially useful as a fuel for removing oxygen during the maintenance phase, where much lesser quantities are required. Combustion of hydrogen and oxygen can be achieved at normal ambient temperature over some catalysts, a substantial advantage over catalysts for propane combustion which require heating to more than 300°C before they operate efficiently. The hydrogen could also be produced by electrolysis of water on site or perhaps by photolysis of water with sunlight, a system currently under active research (Wrighton, 1983; Anon., 1982). It would be necessary to remove the water produced during combustion.

Catalytic oxidation of hydrogen (e.g. with a Deoxo unit (Engelhard Industries, Newark)) could also be used to further lower the oxygen content of a low oxygen stream (e.g. 0.5%) produced by another process. This option is already available for PSA systems (e.g., with Techsep systems (British Oxygen Co., London.))

3.1.5 *Direct electrolytic or photolytic removal of oxygen.* Various inorganic ions can be reversibly reduced and reoxidised, with liberation of oxygen during the electrolytic reduction and subsequent direct reoxidation of the species by molecular oxygen. For instance:

$$Cr^{+++} + \epsilon \underset{-O_2}{\overset{+O_2}{\rightleftarrows}} Cr^{++}.$$

It may be possible to find materials (e.g. cobalt-organic complexes similar to those discussed in McLendon and Martell (1976)) which can reversibly bind oxygen in the dark and liberate it in light. These reversible reactions could be used to remove oxygen from a gas stream:

O_2 - poor gas	oxidised form	light or electrical energy
O_2 - rich gas	reduced form	O_2 liberated

The direct current electrical energy required could be supplied from a photovoltaic device (solar cell), where the light could be sunlight itself.

This scheme is still only speculative but there is much research interest in similar fields for the photolytic generation of hydrogen (Wrighton, 1983; Anon., 1982). Because of its lack of moving parts or dependence on external material or energy supplies, the scheme appears to merit investigation.

3.1.6 *Removal of oxygen by chemical reaction.* It may be possible to develop oxygen-removal processes based on materials which react reversibly with oxygen, perhaps chemically similar to the biological oxygen carriers (e.g. systems described by Basolo, 1978). In small scale CA applications, a reactive material could be provided in a recirculation system with sufficient capacity to absorb all the oxygen leaking into the system during the exposure period.

3.2 <u>Potential systems for nitrogen-CO_2 mixtures</u>

3.2.1 *Combustion of hydrocarbons from off-site sources.* There are machines available which produce gases for CA storage of fruit by burning hydrocarbons in air to remove oxygen. Because the fruit, such as apples, generally cannot tolerate < 2% O_2 and more than a little CO_2, these machines are not directly suitable for use for CA storage of grain. In general they could be adapted, with suitable tuning, to give a lower oxygen level and remove the water formed.

The combustion of propane in air follows the equation

$$\begin{array}{llcl} C_3H_8 \; + & 5.0\ O_2 + 18.8\ N_2 & \text{---->} & 3.0\ CO_2 + 18.8\ N_2 + 4\ H_2O \\ \text{propane} & \text{air} & & 14\%\ CO_2,\ 86\%\ N_2 \\ \text{(1 vol)} & \text{(23.8 vols)} & & \text{(21.8 vols)} \end{array}$$

There are three forms of burner:

(i) 'Open flame' burners (e.g., those from Capco, Melbourne and Gas Atmospheres, Port Washington (Anon., 1981)).

(ii) Catalytic burners (e.g. Arcat and Tectrol systems).

(iii) Internal combustion systems.

In open flame burners, air and propane are burnt continuously in a combustion chamber. They are capable of producing high rates of gas at low oxygen tension. Catalytic systems can be used in a recirculation system. They do not give as high rates of gas output and only reduce the oxygen concentration by a fixed fraction (e.g. to one third) during passage through the catalyst. Catalytic systems use less propane per tonne grain stored than open flame to create a low oxygen environment. However, they consume more electrical power, particularly in the later stages of the oxygen removal where heat is required for the catalyst to maintain the catalyst at a high temperature (see Table 3). Presumably open flame systems can be modified with suitable mixture control to allow combustion of the propane in a mixture of fresh air with some recirculated gases, thus reducing the oxygen concentration in the combustion mixture and thereby using less fuel.

The third alternative, a modified internal combustion engine, is currently under development at CSIRO Division of Entomology (Wiseman and Banks, 1979). Its main advantage over the other systems is that it can be easily made independent of cooling water and electricity requirements that can often pose a problem in arid or remote, unserviced areas. Also the output flow can be regulated over a wide range, giving a system which may be suitable for both purging and subsequently maintaining a storage under a low oxygen atmosphere.

3.2.2 *Producer gas combustion.* A variety of carbonaceous materials, including wood, straw, rice hulls and other agricultural wastes, can be converted by combustion in a limited supply of air to producer gas; a gas mixture of variable composition depending on the fuel, but typically about 30% CO_2 with the balance largely inert gases. Producer gas could be burnt with air to give a low oxygen exhaust mixture in systems such as given in Section 3.2.1. A typical combustion equation using producer gas from charcoal (Breag and Chittenden, 1979) would be:

$$\underbrace{0.3\ CO + 0.04\ H_2 + 0.04\ CO_2 + 0.62\ N_2}_{\text{producer gas (1 vol)}} + \underbrace{0.17\ O_2 + 0.64\ N_2}_{\text{air (0.8 vol)}} \longrightarrow$$

$$0.34\ CO_2 + 1.26\ N_2 + 0.02\ H_2O$$

$$21\ \%\ CO_2,\ 79\%\ N_2\ (1.6\ \text{vols})$$

Producer gas systems, using coke, have been used for generating CA gases for grain storage in the past (Winterbottom, 1922), though the purity of the gas used then would not meet today's standards (e.g. CO often > 5%, U.S. maximum permitted level 4.5% (Anon., 1980)).

A producer-gas-based system has two important advantages over propane-based ones: it can use locally produced and easily stored fuels and, with charcoal as fuel, very little water is produced during combustion. Water removal from the combustion gases in propane-based burner systems makes the apparatus required much more complex. Note, however, that propane-fuelled systems give about 1.5x as much gas per mole of carbon burnt as given by producer gas systems.

3.2.3 *Combustion of methane from fermentation.* Many agricultural wastes (e.g. straw) can be fermented anaerobically to produce a methane-rich gas called biogas, typically 50% CH_4, 50% CO_2 when produced from carbohydrates (Lane, 1980, 1981). Combustion of biogas in a burner as described in Section 3.2.1. would give a low oxygen gas suitable for CA use. Biogas combustion in air typically follows the equation:

$$\underbrace{0.5\ CO_2 + 0.5\ CH_4}_{\text{Biogas (1 vol)}} + \underbrace{1.5\ O_2 + 5.6\ N_2}_{\text{air (7.1 vols)}} \longrightarrow \underbrace{1.0\ CO_2 + 5.6\ N_2}_{\substack{15\%\ CO_2,\ 85\%\ N_2 \\ (6.6\ \text{vols})}} + 2.0\ H_2O$$

Biogas must be generated in a specially constructed digester. Gas storage facilities may be required to cope with changes in output and usage rates. Both items may result in significant capital cost on site. Waste heat from biogas combustion could be used to maintain the fermentation at optimum temperature. Biogas systems produce slightly less gas, about 0.9x per mole of carbon burnt, than propane-fuelled systems. Biogas sytems appear very suitable for CA generation in less developed regions because locally produced fuels are used, methods of constructing digesters have been developed relying only on local materials (Lane, 1981; Meynell, 1982) and there is no need for extensive mechanical servicing and parts required by more complex systems.

3.2.4 *Hermetic storage with sacrificial areas.* Storages must be very well sealed and permit very low gas interchange rates with the external atmosphere, if they are to be suitable for true hermetic storage of dry grain (< 12% m.c.) (Oxley *et al.*, 1960). Even with grain of 12-16% m.c., the rate of air ingress must be very low to ensure that mould and insect growth is restricted. In order to create low oxygen tensions in more leaky structures, it is necessary to increase the rate of respiration within the structure so that initial insect infestation cannot persist (e.g. as observed by Oxley and Wickenden, 1963). This may be accomplished either by seeding a small region of the stored commodity with a large number of insects (S.W. Bailey, unpublished data) or by intentionally wetting a small region of the bulk (Burrell, 1980) thus stimulating mould growth and respiration. In either case it is useful to provide some form of gas permeable containment of the sacrificial region so that it can be removed and discarded when the commodity is removed.

3.2.5 *Assisted hermetic storage.* The category 'assisted hermetic storage' is used here to define a hermetic storage process, with the reduction of oxygen level, occurring normally by respiration activity in simple hermetic storage, augmented by some other controlled process, usually external to the storage system. It is distinguished from 'sacrificial' storage by the higher degree of control of the oxygen removal process. In sacrificial storage there is no regulation of the rate of oxygen removal after the initiation of the process. An elegant example of assisted hermetic storage is now in use in China (Lu, in press). In this case to remove oxygen the storage gases are circulated through an enclosure containing racks of moist grain and bran seeded with a particular mould culture.

3.3 Production of gas for high- and intermediate-CO_2 atmospheres

3.3.1 *On-site production by burning carbon-containing materials in air.* CO_2 can be collected from a gas stream by reversible sorption processes (e.g. into ethanolamine, with the Vetrocoke process or by PSA systems). Such a process could be used to remove CO_2 from the exhaust from the burning of hydrocarbons in air, to give a CO_2-rich gas at an adequate concentration (> 70% CO_2) for creation or maintenance of a high- or intermediate-CO_2 atmosphere. Small-scale industrial plant operating on this principle and utilizing fuel oil as a CO_2-source is available. Presumably the process could be used to recover CO_2 from gas streams from fermentation processes or from burning producer gas or biogas.

3.3.2 *On-site production from burning coal or charcoal in oxygen.* Burning carbon directly in oxygen could produce pure CO_2 directly. The easily available carbon sources, dried powered coal, coke and charcoal, all contain sufficient residual hydrogen compounds to produce some water during combustion and would produce a gas requiring purification to remove undesirable contaminants such as sulphur-containing material or unburnt hydrocarbons. Nevertheless this system appears to be a possible route to producing a CO_2-rich gas

(> 70%) without the need for extensive drying or enrichment of the combustion products.

Oxygen of adequate purity could be supplied on site by a PSA system or on a smaller scale by direct photolysis or electrolysis of water or from plant photosynthesis.

3.3.3 *CO_2 from fermentation.* CO_2 is produced industrially from the anerobic fermentation of carbohydrates by yeasts and other organisms. Presumably CO_2 could be produced close to a grain storage, providing a source of gas for CA use and fermentation products (ethanol, industrial solvents, etc.) at the same time. By carrying out fermentation near grain production regions, the energy cost of the transport of grain to processing centres is avoided and products valuable to the food grain storage system are made locally. Low quality or high moisture grain could be used as a feedstock. Other carbohydrate-containing material, notably straw, can also be used.

4. DISCUSSION

With such a wide range of possible processes for the creation of suitable atmospheres for CA storage it is difficult to make a choice as to which may be dominant in the future. The situation is further complicated by the inadequacy of our knowledge on what is the optimum gas composition for particular situations. Nevertheless there are some generalisations and predictions which can be made reasonably:

(i) CA systems will be generated by means other than from tanker-delivered gases. This is particularly relevant to sites distant from industrial centres.

(ii) The greater fuel consumption of on-site CO_2 generation processes place these systems at a disadvantage relative to the processes giving nitrogen/ CO_2 mixtures.

(iii) Unless CO_2 is found to be detrimental (e.g to quality or structures), processes giving mixtures of nitrogen and CO_2 with low oxygen contents are the most likely to be used for CA grain storage at sites distant from industrial centres in the future since their energy requirements are low.

(iv) Systems of CA generation will incorporate some recirculation to permit the refining of some of the internal gas so that its composition remains within specification.

(v) Simple, passive systems of CA generation will be developed which may find widespread application, displacing those systems relying on much machinery (e.g. burners, PSA systems).

(vi) CA generation systems will be developed which utilize materials available cheaplv. close to the site of use. These will include agricultural wastes and air.

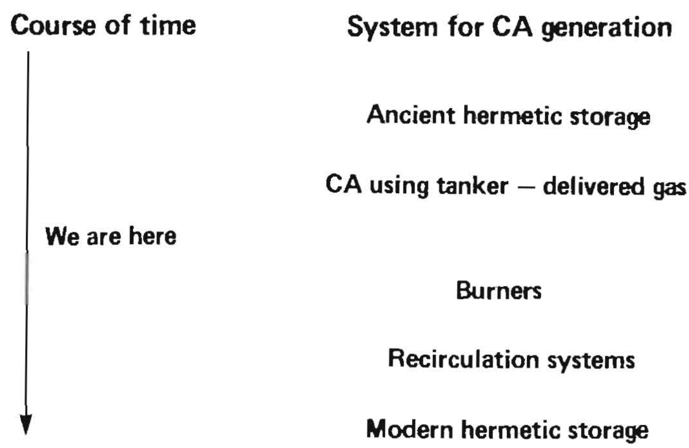

Fig. 2. Time chart for the progress of methodology for CA generation for grain storage.

In the spirit of these comments, I predict a progression in the processes used for CA generation as given in Fig. 2. The ancient hermetic storage system has been in use, almost unchanged, for several thousand years. Tanker gas-based systems and their forerunners using dry ice or cylinders have been available for a few decades. Burner systems have been used but are still largely in the research phase of development. Recirculation systems for CA use have been demonstrated but are not yet in routine use. Lastly, we reach the system I term 'modern hermetic storage', where the elegant process of hermetic storage is refined and controlled so that it meets the current constraints of grain handling and marketing, utilizing a small, and perhaps substandard, portion of a commodity stock, as a source of gas, to preserve the remainder. The current interest and speed of development of systems in CA grain technology suggest that we could reach Time Level 5 by 2000 AD.

There is no reason why any of these processes should become the sole system for CA generation. Indeed it is likely that all will exist together in time, with the use of particular systems dictated by local conditions. In some places, grain storage may develop so that some of the products of the system used for CA generation (e.g. fermentation products) may become industrially useful, contributing to the economics of the storage process. In other places, simple solutions may be more appropriate.

It can be seen that much remains to be done before CA technology can be regarded as fully developed. Nevertheless enough is now known at least for the problems to be defined. Let us hope that they are now solved quickly so that CA systems will continue to play a prominant and well founded role in grain storage.

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SESSION 8.

RAPPORTEURS REPORTS OF FIRST WEEKS SESSIONS

Reports by:

Dr J.L. Multon	-	France
Dr M. Bengston	-	Australia
Dr C.H. Bell	-	United Kingdom
Dr E.J. Bond	-	Canada
Mr D.J. Calverley	-	United Kingdom
Dr S. Navarro	-	Israel
Mr G.G. Corbett	-	Italy
Mr B.E. Ripp (Moderator)	-	Australia

Rapporteur's Report

Session 1

CURRENT USE OF CONTROLLED ATMOSPHERE STORAGE

Dr. J. L. Multon

During all this first week of the Symposium I have been very impressed by the new, recent, and wide development, and application of this old known method already used in antiquity by Egyptians, Romans and some other people and consisting basically of sealed storage. But if the method is old, the technology used has been incredibly improved. It is one of the major topics of this Symposium to inform the delegates of the ultimate improvement of the Controlled Atmosphere and Fumigation System.

The Session 1, for which I am the Rapporteur was, more or less, an introduction to this Symposium, and has offered three papers concerning the "Current Use of Controlled Atmosphere". Of course it was not at all intended to overview such a wide programme in four papers! But as far as it was necessary to make a selection of the presented topics in order to give a view of the recent developments in the current use of Controlled Storage Atmosphere, the choice was very good and interesting, because it was very representative of some of the main tendancies now in progress in the world.

The first paper presented by Dr. E. G. Jay, U.S.A., has focussed our attention on the problems due to insects in rich industrial countries which export large proportions of their production far away by ship. By some aspect the case of my country is similar. And I have been very interested to see that large industrial companies in U.S.A. have a growing interest in Controlled Atmosphere as a technique for eliminating insects. It is no less interesting to learn that U.S.A. is mainly thinking towards CO_2 atmosphere which is not dangerous, not too expensive, and non-chemical residue producing, this last point having been covered by Mr. Sticka. In this paper Dr. Jay describes some sophisticated equipment of sealed storage used in terminal and exporting elevators and it was by fact a very good introduction both to the following lectures that we heard during the week and to the extremely interesting technical demonstrations that we saw yesterday. He had also described some farm equipment, farm storage being often the first stage of the storage chain.

The second paper presented by Professor Lu-Qian Yu, Peoples Republic of China, has shown on the opposite how it is possible to realise a good sealed storage with limited financial and technical resources. I have been myself in the Peoples Republic of China three years ago, and I have seen a sealed silo in a peoples village near Canton, and I remember my admiration for the precision, the quality, the efficiency, with which the plastic sheet was disposed in an old warehouse and the care with which the wax was applied for sealing. I have also been very impressed and interested by the original technique used for reducing O_2 concentration with mould culture in boxes connected to the silo. Using a fashionable word, I would say this is an interesting biotechnology, alternative to the use of chemical treatment. Anyway I think this paper is an excellent example of what could be done by countries having limited financial resources but having a good know-how and developing a great quality and great care in the utilisation of an old or very simple warehouse. I think that such a paper should represent a reason of hope for developing countries. From this point of view, it is a pity, that the paper by Sowunmi and collaborators have not been presented, because according to the abstract distributed it would show a good example of the adaption of metal sealed silos with a Controlled Atmosphere in a hot humid climate of Africa.

The third paper presented by Dr. Akiyama, Japan, is representative of the problems of an industrial country which is obliged to import very large quantities of grain (about 10 million tonnes). By fact such a country is perfectly right when it demands to the seller a good quality of grain, guaranteed insect-free. Also, I have been personally very interested in the quarantine process and fumigation technics developed by Japan in order to check if cereals arriving are insect free or not. A very impressive and efficient service of quarantine and fumigation has been initiated in that particular country from as early as 1914. But it has been continuously improved since that time until its present state. It is also important to note the great attention given by Japan to the problem of the residue due to chemical treatments and we may thank Dr. Akiyama for his excellent studies of the residues remaining after fumigation.

In these three papers it has been emphasised that if it is absolutely necessary to kill the insects, it is not less necessary to avoid, as much as possible chemical residues in the grain. This is why a well done fumigation under Controlled Atmosphere (with CO_2 or nitrogen) seems to be the most attractive solutions and alternatives to the use of insecticides associated with ventilated storages. Phosphine and Methyl Bromide fumigation are the more efficient, the quicker and the cheaper methods. But they are dangerous to workmen and could leave some residue. The CO_2 or Nitrogen perhaps are more expensive and requires of course, strictly sealed silos, but they are certainly not residue producing. All the following papers and technical visits we have made gave ideal opportunities for delegates to make their choices between the methods to which the first session was, as I said in the beginning, a good introduction. Also I think we may thank very much the speakers - Dr. Jay, Dr. Lu-Qian Yu, and Dr. Akiyama for their excellent and most interesting presentations.

RAPPORTEUR'S REPORT

SESSION 2

ENTOMOLOGY OF CONTROLLED ATMOSPHERE STORAGE

Dr M. Bengston

The session comprised papers discussing the response of insects to fumigants and atmospheres.

Dr. F. Attia's work established that for some important species in Australia, phosphine resistance is unrelated to pesticide resistance. Detailed data are valuable to geneticists and mathematical modellers endeavouring to compare strategies aimed at delaying resistance. Questions and discussions indicate confusion and conflict arising from the use of the term resistance. Laboratory tests are designed to give very accurate comparisons among strains and on this basis one strain may be significantly more resistant than another, and this may or may not be related to control failures in the field. In the current instance the laboratory resistant strains collected in Australia are still controlled by normal commercial phosphine fumigation. Speakers in other sessions have reported failures of field fumigations in Bangladesh. Clearly resistance is a central problem and wherever practical we need to manage the use of fumigants so as to delay its development.

Dr. P. Williams reported field work with combination of methyl bromide and carbon dioxide which enabled the dose and hence residues of methyl bromide to be reduced to one third of their current values. The technique is clearly more complex than current methyl bromide fumigation but it is worth reminding ourselves that in future buyers and consumers could well set more stringent standards for residues arising from the use of methyl bromide.

Dr. J. M. Desmarchelier's paper established the interesting possibility of accellerating phosphine fumigations through the addition of carbon dioxide for what he described as the non-*Sitophilus* situation.

There were anomalies in regard to results with *Sitophilus* pupae. Williams reported potentiation of methyl bromide with carbon dioxide but Desmarchelier reported that for phosphine the effect was significant at the LC_{50} level but not the LC_{99} level. This would be a key issue in regard to implementation of the technique and needs to be further clarified.

Dr. C. Bell described the effects of oxygen on the toxicity of carbon dioxide in a paper which brought out the complexities of the situation and underlined deficiencies in our understanding regarding the action of carbon dioxide. His data confirmed that carbon dioxide is more effective at high temperatures. The central point arising is that biologists need to define more accurately the lethal effect of the various atmospheres.

QUESTION

Dr. J. M. Desmarchelier, CSIRO, Canberra pointed out that the engineering and operational requirements of using controlled atmospheres or fumigants in large storages would result in delays between inloading and subsequent application of control procedures. Insect populations would increase during this time which would be two weeks or longer. He enquired whether the build up in insect numbers before fumigation would have a long term effect in regard to the development of insect resistance.

Dr. Bengston replied that it would be desirable to keep the number of insects exposed to fumigation to a minimum. He added that the worst situation in regard to the development of resistance would be the repeated use of partial fumigations in semi-sealed storages. This would result in an isolated population being subject to repeated selection with the resulting possibility of rapid build up of resistance genes.

Rapporteur's Report

Session 3

PRESERVATION OF QUALITY IN CONTROLLED ATMOSPHERE STORAGE

Dr. C. H. Bell
UNITED KINGDOM

The session comprised three papers, one dealing with the quality of maize for flour manufacture and other processing, one dealing with quality as associated with the ability of malting barley to germinate and the third dealing primarily with quality in terms of taste in tests on coffee beans.

Grain quality is linked very closely with microbiological activity or 'spoilage' which in turn is linked with moisture content and equilibrium relative humidity temperature and availability of oxygen. It has long been known that grain stored at moderate to low moisture content in low oxygen and at low temperature keeps in good condition for many years. Currently, wheat kept at Slough at 5°C and low oxygen concentration has retained good baking qualities and powers of germination for well over 20 years. At high temperature, however, germination gradually falls although baking qualities are retained, while at high moisture contents spoilage occurs.

The first paper described the storage of wet maize after partial drying in 1 m^3 capacity structures specially constructed to simulate larger scale storage conditions. At present maize is either allowed to ferment as silage to produce cattle feed or for other uses is dried in a 'crib' store by the ambient atmosphere, a process depending on good drying conditions to prevent spoilage. Unlike other cereals it is difficult to dry maize to a moisture content below 20% by hot air methods without adverse effects on quality. The idea of storing maize dried to 21-22% moisture content in airtight conditions was therefore explored. Although there was some fermentation with oxygen levels in the bins falling below 1% within 3 days, good quality of the stored maize was retained for 6 months. After 11 months, although fat acidity and the numbers of yeasts and to a lesser extent other micro-organisms were increasing, the starch producing quality of the maize was unimpaired. A similar test with a larger structure ($10m^3$) showed good retention of quality after 5 months. During the discussion of the paper the problems of water production by respiration and of water migration were discussed. It was felt that the size of storages might be severely limited in an outdoor situation due to moisture migration, but that further experiments would be worthwhile to define such limits.

In the second paper the results of storing malting barley in a nitrogen (N_2) atmosphere were presented. Tests were being planned in a range of controlled atmospheres of cheaper operating cost than N_2, such as exothermically or biologically generated atmospheres, to see if a cheaper method could be found for preserving germination than the present technique of refrigerated aeration. In the initial tests with N_2 interpretation of results was hindered by the presence of a layer of poorly germinating barley near the surface of the treated bins, but there did not appear to be any advantage over the existing refrigerated aeration technique.

It is perhaps worth adding that although the saving of germination capacity under inert atmospheres may not be any better than the current refrigeration methods, their operation may prove cheaper. Also carbon dioxide (CO_2) is known to prevent loss of grain viability, even in the presence of oxygen, and it was suggested that CO_2 could be considered for this particular purpose.

The third paper described the effect of sealing bagged green coffee beans on pallets in a PVC envelope chemically bonded to a ground sheet, and then purging with a 75% CO_2 mixture in air. Coffee is a crop that deteriorates within two weeks in hot, moist atmospheric conditions, but under CO_2 the coffee remained fresh for 6 months, except when the moisture content under the sheet was raised by wet pallets. In the discussion a somewhat similar sealing method for storage of coffee beans in India was described. The method of testing for gas tightness before commencing the CO_2 application was discussed together with arrangements for stopping the purge at the appropriate time.

At the close of the session it was apparent that on all the issues presented confirmatory tests were required over longer periods before the full potential of controlled atmospheres in the preservation of quality of foodstuffs could begin to be ascertained.

In the final discussion the need to avoid internal supports within grain bins from the entomological point of view was stressed and it was agreed that any attempt to reduce the moisture content of a commodity before sealing in a storage was to be encouraged.

Rapporteur's Report

Session 4

STORAGE SEALING TECHNIQUES 1

Dr E. J. Bond

This session included papers on storage design, sealing techniques and sealants for a variety of grain storages. A great deal of the information required by both managers who make decisions on storage systems and contractors and technicians who seal storages to prescribed criteria, was given in this session. Three of the papers dealt with the sealants themselves, two described the properties required in sealants and the techniques used for effective application and three discussed storage design and problems relating to sealing to gas-tight standards.

The first paper on 'Sealing techniques for grain storages' outlined the characteristics required in sealing materials to make effective and long lasting seals and it emphasized the importance of the surface for good adhesion. The seal can only be as good as the surface to which the sealant is applied; if the surface is not clean the sealing compound will not stick properly nor provide an effective or durable seal. Depending on the type of surface, e.g. galvanized, aluminium, painted, etc. and whether new or weathered, will determine the requirements for primers or other treatments. Different products used in silo sealing have different properties and capabilities and should be selected according to manufacturers specifications to achieve best results. Silos may be sealed by application of the sealant internally, externally or by a combination of both internal and external treatments. Numerous slides were shown in the presentation to illustrate the problems and to show the areas that required special attention in the sealing operation.

The paper entitled 'Polyurethane foam for sealing grain storages' gave essential information on the major problem of filling openings and bridging gaps in storage structures. The great potential of the foam for many uses was emphasized, particularly since it can provide suitable surfaces for application of other materials including continuous sealing membranes. After outlining the chemical and physical properties of polyurethane foam, some details of the techniques used for applications were given, i.e. preparation of various substrates, environmental conditions most suitable for applications, spray techniques, installation time and cost and precautions to be taken. Some discussion was devoted to the critical areas requiring most attention for filling with the foam, including ridge capping, side and end walls as well as sites in the penthouse.

A number of the sealants used for the various applications were described in the next two papers and it was pointed out that external sealants may be white to give maximum reflection of heat and they may include rust inhibitors. Internal sealants may consist of polymer concrete for floors, acrylic mortar for connections and bituminous primer plus polychloroprene coating for walls. The importance of proper surface preparation before application of the sealant was emphasized. The use of a fleece to provide a substrate for sealants in bridging gaps, joints or connections to overcome problems of movement at such points was outlined.

These papers also included useful information on the chemistry of the materials along with physical properties. Basic information such as this is invaluable to contractors and other personnel who are testing and adapting new products for new and untested situations.

In the paper on 'The properties of various sealing membranes and coatings' the physical factors and changes that may occur in metal storages along with the charateristics of sealants required to deal with these changes, i.e. tensile strength, tensile strength vs. temperature and adhesive strength, were outlined. After this presentation the question of how to determine the effectiveness of these materials in practice was posed. Technical data on physical and chemical properties plus knowledge of the physical stresses to which the material will be subjected are obviously of great value, however short term and long term experience is perhaps the only way to get definitive answers.

Following the presentations on sealants, storage design, construction and operational problems were discussed. A low cost 'total concept' in the construction of large capacity horizontal storages that included the capability for controlled atmosphere treatments with coincident capabilities to conduct all other functions in the storage, without breaking the seal, was described. Such a storage has been developed at Moura in the state of Queensland but some problems with inloading, outloading and aeration in the sealed, gas-charged storage have yet to be solved.

The next paper described the modifications needed in design of standard structures to make them suitable for sealing and for controlled atmosphere treatments. Such changes as ventilator design, conveyor entry ports, doors, etc. are necessary along with elimination of such items as translucent sheeting and replacement of bird netting with sealing plates, were cited. This paper stressed the important point of liason between management, operation and control personnel. Communication is an area that is frequently overlooked but it is crucial to the solution of many problems - good liason is essential to success in developing efficient designs for storage.

The final paper entitled 'Design of fumigable stores in the tropics' took us from a highly developed technology and highly organized systems to areas where storage techniques were very dependent on local conditions and local resources. Design of such storages is dependent on the purpose for which they are intended (often for bagged goods), the climate and the need to ventilate, as well as the materials that are available locally. Storages may be made gas-tight for fumigation using temporary sealing materials; permanent, long term sealing may not be feasible or even desirable in many of the storages used in tropical countries. Storages made available to developing countries through aid programs should be designed so that they can be made gas-tight for fumigation. These buildings, which are often susceptible to damage and difficult to maintain, should be assembled by skilled personnel to ensure specified standards.

All of the papers given in this session provided a great amount of information that would be needed by planners and contractors to make storages gas-tight for controlled atmospheres and fumigation. Throughout the session discussion on the practical aspects of sealing was well balanced with the basic physical, chemical and engineering information needed to give some understanding of the problems and solutions to the problems. Other points raised in the discussion period included sealing of vertical storages, sealants for low temperature climates, gas-tightness standards, leak detection and pressure testing.

From the session it was obvious to the outside observer that a great amount of information on many aspects of sealing for various types of storage situations has been developed in recent years and that the field of sealants, sealing techniques and sealing standards has reached an advanced state of development. The question of how to obtain all of the information needed by interested parties to plan and carry out sealing operations becomes a real one. At the present time many pieces of information are available from many sources and there is a great need for this to be assembled under one cover in a well balanced, comprehensive and objective form. In the discussion period following the session a suggestion was made that the Australian Grain Institute should develop training courses on the subject, both for Australia and for other countries. A publication on the subject (that might evolve from a training course) which would include all of the relevant information needed by both planners and applicators involved with sealing of storages would be invaluable.

RAPPORTEUR'S REPORT

SESSION 5

STORAGE SEALING TECHNIQUES (2)

Mr D J Calverley

Ladies and Gentlemen I have to report on Session 5 - Storage Sealing Techniques 2. And I think you can't divorce this session from the previous session. You'll find similarities in the workings and that we discussed the practical applications of the materials and technique procedures which have been discussed in a number of papers. The session also elaborated on the establishment of the rate of deterioration in sealed storages for fumigation or for controlled atmosphere.

The first paper by Sheryl Buchanan describes the techniques and materials used in sealing welded steel bins, with a conical roof, flat bottom, with a total volume of 2,400 cubic metres. It was stressed that particular care was needed at the wall to floor joint because of the differential movement between the steel and concrete bin, that the doors and the walls are sealed with a sealant generally described in the previous session. The pressure relief valve was traditionally operated, with the internal pressure over ambient of 750 pascals which is not quite clear from the data or from the ensuing discussion. The silo was painted silver except the roof which was painted white to reduce solar heat generally. In spite of this, condensation was apparent on the underside of the roof of the silo and the moisture migrated with warm upward air in the grain mass. This statement I must say rather surprised me, as in my experience the temperature differential will generally move moisture through the cooler area of the silo. Silver paint does not always have such good solar reflection properties as white paint. The effect of painting white the roof of a large horizontal silo was dramatically illustrated in a paper by Mr C Barry and Mr Ripp. There appears to have been no attempt to investigate the effect of painting the whole cylinder of the silo - the complete silo. There was also, I think some uncertainty of the effect on the headspace and I was also not clear on the result from the discussion. The paper by A Wilson considered that the head space of only 1 metre depth should be left. Other authorities during discussions in Rome were remembered as recommending a complete fill, even to the extent of topping up to the highest level of grain. Here we have again the question of moisture content, which is a crucial factor and was suggested that it should be at least below 12%. The standard pressure test was carried out according to plan and appeared to be successful. High levels of CO_2 were maintained after two weeks since the purging with CO_2 was carried out, but no data was offered on the insect stages of the grain at the lowest, which to me is the most important particular aspect of the whole exercise.

Mr Barry and Mr Ripp in their two papers described a number of stores and the sealing operations that were carried out. These papers that they gave were made quite alive during the last 1½ days of our tour here. There were quite irregular capacities in their descriptions from 20,000 tonnes to something like 2 to 300,000 tonnes, but in all cases the sealing can be called similar. Polyurethane foam filled void followed by a flexible polyvinyl chloride membrane overlapping the joints.

Mr Barry gave considerable details of the pressure tests on the sealed structures using the Banks and Annis criteria. Mr Ripp gave a general description of the methods of leak detecting but he didn't give us any data of what happened on the pressure tests on the large silo. He did however, give a description which was there for all to see, of methods of leak detection, including a thermographic survey. But he concludes, and I think you saw that demonstrated, that the application of water and detergent to the surface of a sealed storage provides a reliable and a very simple detection system.

It is obviously not easy to show how the valves vaculated. Yesterday we saw that it is clearly not easy to carry out satisfactory pressure tests unless the storage reaches thermal equilibrium with it's ambient atmosphere. Two of Mr Barry's tests were carried out as the building was cooling. It clearly over estimated the degree of leakage. The data by Banks and Annis considered quantitative effects of the various factors or agencies which cause gas loss from the grain store. The level of heating with the influx of wind and the chimney effect - albeit at a low rate - compared to the level of sealing which reduced the loss caused by barometric pressure and temperature changes. The criteria for minimum loss rates were taken at the rate tolerable in insect control processes using phosphine fumigation and sealing-injecting CO_2 and also for long term exposure for nitrogen. This paper suggested that there may well be ambiguity in setting a very simple decay half life as a method specifying work patterns. It suggested, for example, that in the case of silo bins using carbon dioxide the currently accepted standard, a pressure decay time of five minutes, that is a pressure drop from 120 pascals to 60 pascals in five minutes, may need to be increased to ensure gas loss is not excessive even under very adverse environmental conditions.

Unlike Arnold Wilsons paper, a very deep dependence of the effectiveness of this fumigation at each of the silos described using phosphine which was the object of the exercise. In one storage with a white roof, concentrations of phosphine were maintained at all times and points within the store, 30 parts per million from Day 3 to Day 19 and in another store at 50 parts per million from Day 1 to Day 12. Further results were given for other stores. But it seemed to me looking at the data that it was quite noticeable the deterioration rate was generally slower during fumigations carried out at a cooler temperature than those carried out in the summer in December and early January. And I think that this generally may well be compatible with the theory which Jonathan Banks and Peter Annis put forward in their paper.

The session clearly demonstrated in all papers, particularly the practical ones, the technique of sealing to fumigate with carbon dioxide and with phosphine. Mr Ripp in his paper, I think, spoke with confidence rather than with fact, on the integrity of sealing in the very large store, when it was expressed quite clearly that he had no doubt about its ability to respond quite as well as the stores in Barry's paper.

Two very important aspects were brought up concerning safety, and these were very properly covered in Wilson's paper and by Mr Ripp. The first concerns the safety of personnel going into underground installations and the recovery conveyor in which personnel may be involved. In common with many other factors, Barry pointed out it blocks off an unnoticed facility, once you stop natural ventilation in the building and he noted the very large increase in dust concentration in some of the stores when there was inadequate ventilation to remove it. This really makes me comment on the paper by O'Neil, which is in somewhat contrast with this. O'Neil did not use fans, you will recall, for his ventilation of a large store, but he was quite confident that by opening the two gable end ventilators this would be sufficient to remove the air borne dust from the inside of the storage. I find this a very interesting comment. The gable end ventilation, I would suspect unless it is fanned ventilation, would not necessarily be terribly efficient - and great airflow is needed to achieve adequate ventilation. There is a little bit of conflict on this particular point.

Sheryl Buchanan gave costs for sealing and for carbon dioxide usage that seemed to be keeping with other authorities and with papers given later in the session bearing in mind the sort of inflation that would have taken place since her costs had been prepared. Mr Ripp in his paper also covered costing but he, I think, was rather optimistic in the kind of costing which he showed for this store. And it would be interesting to understand why he was able to show that the costs of fumigants in the Kwinana store were much, much lower than they were in the other stores. He neither gave a reason for the discrepancy nor did he adequately describe what the other stores might be.

Another point I would also make on costing, I think is very important and I made the point earlier this morning. The depreciation period of the sealing has been taken as twenty years. We've already had a comment from the floor that we're really not quite sure what might happen in due course of time to the seal. Are we right, are the costings justified in looking at the life of a sealant of only seven years and have you regarded also there can be a technical change - is twenty years an over long period on which to write off expenses of this sort and not allowing yourself sufficient anticipation for any further development that might take place?

Nevertheless the papers do clearly demonstrate that the capability of providing a physical sealing to a building would prove to adequately and easily allow for efficient controlled atmosphere and fumigation.

COMMENTS

Moderator: (Mr Ripp) Thank you David Calverley.

Any comments or queries to be directed to Mr Calverley or the paper authors of that session please?

Dr J Banks: I don't believe Charles O'Neil is here - is he - and with that knowledge I can comment more freely!

The Mooree shed that was constructed and done over as a sealed shed has on it two gable-end ventilators and a number of raised ventilators just above the pitch. The design of this is such that you can in fact seal all these ventilators should you wish to. And I think this is important, because it is an approach to the general concept of fully controllable ventilation.

Mr Calverley has, perhaps, doubted that the gable end ventilators are sufficient. In fact normally you, I believe, have to open one or the other ridge ventilators - no, not only ridge ventilators - eaves as well in order to get the full ventilation effect. But I am assured that this was adequate and it may be that Rex Sticka will be able to comment on whether this was indeed so. But that is an example, I believe, of fully naturally ventilated sheds, which have controllable ventilation and I would believe that often, particularly in more humid areas, we are going to have to build such general facilities for the system.

Moderator: (Mr Ripp) Thank you Jonathan. I am not sure whether Mr O'Neil was referring to that one, or the Queensland store that he has under construction at the moment.

C O'Neil: Force ventilated at Queensland.

Moderator: (Mr Ripp) Force ventilated at Queensland - Thank you.

Perhaps you might like to comment on David's other comments on Sheryl's paper where the cells were not painted exterior even though there was a stretch coat and coupled with that, of course, is that we apparently didn't make clear the effect of the headspace or excessive headspace on the respiration of the storage.

Dr J Banks: To some extent you have the advantage of me, Mr Chairman, because I came into the session rather late and therefore I don't know of your second problem.

The first one the data does show that there is a very substantial variation of the difference in headspace temperature, whether you paint it silver or paint it white. What was the particular problem with it, though?

Mr D J Calverley: The point I did make was that it was surprising that although the headspace was painted white and yet although moisture migration was recorded within the silo, and, I believe, on an earlier occasion as in the note given in the paper, the barrel of the silo remains silver, which is substantially inferior to white as a reflector and had it been painted white this would have helped the moisture migration problem. Furthermore, there was uncertainty as to what amount of headspace there should be in order to prevent condensation and high moisture content condition, high humidity conditions occuring within the headspace. And during the discussion on this paper there was some divergence of opinion. But nobody was able to be authorative about it.

Dr J Banks: I fear that is the state of the art at the moment. The reason why only the roof was painted white was because it was strictly an experimental treatment, where we wished to show that there was a difference and indeed we did show it and normally the whole structure would be painted white.

Moderator: (Mr Ripp) Thank you Jonathan.

Mr Z Gollop, Israel: Mr Chairman - In your interesting paper on Controlled Atmosphere in a large storage you gave cost comparison between various treatments in sealed and unsealed stores. Amongst those there is the price of 20 cents for methyl bromide both in sealed and in the unsealed storages. I would like to make a few remarks:-

1. From a number of experiments, especially those which were carried out in Holland, it was proved that in an airtight storage the dosage can be reduced by at least 40%.

2. The current price of 2 dollars per kilo of methyl bromide in Australia and a half pound per 1,000 square feet, which is 35 grams per tonne or 50 grams per cubic metre, would cost in a sealed storage 4.6 cents and in an unsealed storage 7 cents and what is the 20 cents that you have mentioned? I assume that probably the price that you got from the accountants was old and things have maybe changed.

Mr D J Calverley: Acting as the Rapporteur and forced to act as Secretary during the election of Chairman I think this question should be addressed directly to the man who wrote the paper.

Moderator: (Mr Ripp) Certainly. Yes, you are quite right I did take old figures because we haven't used methyl bromide for some time and even the methyl bromide we did use in the trial was in fact old stock. So I did just take the last figures and I normally wouldn't take old figures but upgrade them for inflation and so on, I didn't do that. Apparently I should have gone the other way.

The reference to the difference is certainly correct - you will use less methyl bromide or anything else in a sealed storage so that is quite correct, so that would be my error in transferring the figures across to the other side. But thank you for bringing that up. Your figures were, if I remember rightly, about 4 cents.

Mr Z Gollop: 4.6 cents sealed and 7 cents unsealed.

Moderator: (Mr Ripp) Have you ever sold us any methyl bromide - I think we will buy it from you next time! Thank you for your comment.

Mr B Elder, CSIRO: I'd like to buy in on this moisture migration question, which certainly is a vexed question. Certainly the state of the art is poor. But one thing that we perhaps ought to remember is that the roof painted white, or whatever it is painted, underneath the roof is going to respond very rapidly to changes in atmospheric temperature, whereas the wall which is in contact with the grain, a silver painted wall, during the day will presumably heat up quite substantially and may not cool down to as low a level as a white painted roof which has got no contact with anything.

Howard Greening, NSW Dept. of Agriculture: We've heard about the bird damage that was done to insulation coatings on the outside of the storages now that the insulation and sealants are being put on - when I say insulation, I mean polyurethene, I would assume used as a filler. Now these materials are being put on the inside of storages, I am just wondering about the possibility, if there is any lapse in control measures, of insects attacking those sealants and damaging the seal for future use?

Moderator: (Mr Ripp) Would anybody like to comment on that question?

Mr D J Calverley: If I could extend the question a little bit. We did discuss yesterday morning the question of rodents - the buildings are not particularly rodent proof and they should be fairly tolerable, but during the period when the storage was empty, with the doors left open I would have thought that there would be good harbourage in some of that soft material for rodents. Clearly once you put a gas in there you are going to 'do' any rodents, but they may already have 'done in' the polyurethene foam.

Mr Ripp: Yes, thats true - we do have rodent bait stations all over our facilities and we hope they have an effect. But it is very difficult to attract rodents to a bait station in the presence of 20/30,000 tonnes of their natural food anyway, but we do make our rodent baits very attractive using chocolate, and peanut paste and make a very nice sandwich. But those same baits in an empty storage, of course, have much more attraction because they are certainly better than the concrete or the sealing materials. But I know that rodents, of course, like to play and its not just a matter of eating, so they certainly will get into it. We are very conscious of the fact that the 'urethene foam' would be attractive to rodents, rats and mice etc. They will play on it. We did mention a few days ago the parrots are getting into it on the outside, so there's no doubt we get bird problems on the exterior, rodent problems exterior and interior, but generally at low level. Insects that were mentioned will certainly get into any cracks and crevices be it polyurethene foam or sealing materials, concrete cracks or whatever. Actually I get the feeling it reduces the potential of the insect development, but they can get into the materials, expecially if there was polyurethene foam placed interior, either full sealing or just using it to cover up the cracks. Another little problem we have come across, of course, is termites. So if the sealing material is near the ground the termites love to come up and play with the sealing material. But these are all just little obstacles to overcome and I'm sure there is an answer to them. Sometimes we are not allowed to carry out extermination procedures on some of the wildlife, because of their being protected, and I might tell you the story about possums. Possums are protected in Western Australia. If we catch any possums in our storages we are not allowed to cause them any harm, but we have to take them out to a wildlife reserve where-ever that may be. And one of our Superivors put in a written report on one occasion that he took this possum twenty miles out to a reserve and had to do sixty miles an hour to beat it back to the storage! And the other one was that he had this possum ready to take out to a reserve, it bit him on the finger and a twenty-four inch crescent fell on its head!

R Jones, CBH: The two points I'd like to make. The first one is the statement that you made, Mr Calverley, that it's not necessary to obtain a complete seal, or a total seal, that perhaps a part seal, or a short term seal would be satisfactory in some cases. The way I see it is that the sealing serves two purposes. One, it keeps the fumigation in and second it stops reinfestation, insects coming in from outside. Would you like to comment on that please?

Dr D J Calverley: I said so much that I can't quite know the context in which I made the statement. If I think right, what I said was, that it may not be possible to achieve the standards of sealing that you are achieving here.

R Jones: The second point was, the fact that they didn't have any regular inspection system to know whether in fact the grain had gone off. Now I feel that possibly in the design of sealed storages an inspection manifold or inspection hatch maybe has to be included in the actual design, and I do feel it is a dangerous practice to fumigate a storage and then possibly not inspect it for some months later. Not particularly, in the climatic elements in Western Australia where we do enjoy fairly mould free storage conditions, but certainly in France, for instance, where I can see big problems if grain in sealed storage conditions are not inspected.

Thank you.

Moderator: (Mr Ripp) Any other point?

So there are no points left in your mind that needs expression on Session 5.

Don Chantler: I'd like to ask a question of Dr Banks regarding the standards or pressure tests on sealing silos. The smaller the silo generally, the much more difficult it becomes to establish any sort of recognised test. I wondered if any work could be done on possibly a sliding scale relationship? Volume to surface area achieving a satisfactory seal seems to be a big thing in getting a standard.

Dr J Banks: Mr Chairman, Delegates may recall that my presentation was a trifle rushed on this point and I could have given more detail to this. The general feeling, there is either a need for increasing the standard for large vertical cells or alternatively slightly decreasing the standard required for very small cells. I think we are in a very difficult situation here, where we have to make a matter of judgement as to what is the right standard and really I have to say to you how many fumigation failures can you tolerate? If somebody can come up with a nice answer about that then we can begin to define standards more clearly, but at the moment I think the decision is, we should not get fumigation failures. And if we say we should not get fumigation failures, undoubtedly we have to go for very stringent standards of gas tightness. If you make the theoretical considerations which I find in my favour, which I hope will be included in the proceedings, you will find that there is not a very great variation of pressure test decay with volume. That is, it is almost independent of volume. If you have a 5 minute decay time for a farm bin you can expect a fairly similar interchange in a large structure. That is, there is very little variation in the gas tightness required to retain gases at below a certain interchange rate. But there is a little variation and if, for instance, we decide that 5 minutes is a very good standard for vertical storage, its very likely that 5 minutes is also a very good pressure decay standard for a large horizontal and perhaps three minutes will be sufficient for a very small farm bin. However, if the decision goes towards tightening the standard, and there is some evidence from Sheryl Buchanan's paper that a slight tightening of the standard for large bins would be appropriate, then it could well be that 5 minutes, which is the current standard I believe, for farm bins is quite an appropriate one. There is some degree of judgement that has to be exercised on this and must be a balance between the possibility of getting that standard, the effort of getting that standard, the situation in which the risk is situated and some idea of how you can tolerate fumigation failure. I hope that sort of explains it. It is a very difficult thing to appreciate, because its very much a matter of judgement.

And may I just make one point about pressure decays. In a pressure decay standard you have to specify the time at which the pressure falls from one value to another and we are looking at the moment at a 5 minute decay time for a 500 pascals to 250 pascals for most structures and I think this is a standard that is beginning to be accepted across the country. It is dependant on things like the quantity of fill, but nevertheless it seems to be an attainable standard - is being attained and it does appear to give you a very good fumigation with an acceptable loss rate.

Don Chantler: The farm bins which have been sealed to the level of 250 to 125 pascals in 5 minutes. The question is related to pressure testing. Has any calculation been made to the size valve required for any particular exchange of air? There are a lot of queries as to the amount of air exchange necessary for normal or extreme natural conditions. Is there any relationship volume to the size of the outlet valve?

Dr J Banks: The equations that are given in my paper are of the type that can be used to calculate the air flow rate which you could expect from a given climatic change. If you could set the design parameters - supposing we said the headspace could drop by 10 degrees centigrade per minute - we can get a very reasonable idea of the air flow rate that the valve should handle and I have done this particular calculation with some other valves. Its not impossible from the equations that will be available.

Mr Ripp: Actually I would point out that Jonathan's description of the words "attain the degree of seal" is the thing that we work on. Because we can show that it is relatively simple to attain the fifteen minute standard, why accept less, although two minutes, three minutes will give a satisfactory fumigation and even if we got zero, if we weren't able to pump it up, the fumigation in that store would be much, much more efficient than in a similar storage where there is no attempt to seal.

S Buchanan: I would like to make a small comment pertaining to the aspect of the extension purposes. My answer was with respect to the recirculation period - there was no inspection of the grain. After that it is actually inspected fairly regularly. The other point is with the expertise we now have with carbon dioxide usage in welded sealed bins. I feel that really an inspection hatch is only going to give you peace of mind, just to be able to look in and see on the surface that everything is going alright. The main problem we had with that moulding on the surface was due to over filling the bin and the recirculation pipe was in fact blocked. So in a normal application I don't see any real problem at all.

RAPPORTEUR'S REPORT

SESSION 6

ALTERATION OF STORAGE ATMOSPHERES

Dr. Shlomo Navarro

The papers presented in this session may be dealt with under the following subjects:

a. Generation of controlled atmospheres and purging grain storages to modify their atmospheres.
b. The application of fumigants.
c. Safety measures in the application of controlled atmospheres.
d. Fumigants monitoring techniques.

The method of generating nitrogen (N_2) in-situ using a generator is a relatively new approach for the controlled atmosphere (CA) storage technology. In the paper of Dr. Ward and Mr. Mackenzie such equipment to generate N_2 through air separation by an absorption process was described. For outlet purity of 98% N_2, and outlet N_2 flow rate of 120 m^3/hr can be achieved using the described equipment. The potential use of this equipment for C.A. storage was mentioned. However, in view of the high price of the equipment as quoted by the author, it seems that further practical work to test the justification of its long-term usage is needed.

In the paper presented by Mr. Guiffre, the application of carbon dioxide (CO_2) from pressurized road tankers was described. The liquid CO_2 can be vaporized on site at a rate of 3 tonne/hr. To obtain a concentration of 70% CO_2 in the silo atmosphere, a consumption of 1 tonne CO_2 per 1,000 tonnes of wheat stored in vertical steel bins with 5% headspace, and 2 tonnes CO_2 per 1,000 tonnes of wheat for horizontal bins with 40-45% headspace, has been found adequate. Cost of application of CO_2 for Australian conditions were given and they consist of three elements: the CO_2 cost ex works, CO_2 delivery cost and on-site purging cost. CO_2 can be purged into the silo either as 100% CO_2 or in a mixture with air (up to 30% air in the CO_2 mixture). The CO_2-air mixtures can be used to purge with a fumigant such as methyl bromide.

Another group of papers deals with the application of fumigants. The "Detia Bag Blanket" fumigation technique presented by Dr. Friemel, was described for the fumigation of large quantities of stationary bulk grain without the necessity of recirculating the fumigant. Each blanket comprises 100 Detia fumigation bags and liberates about 1.13 kg of phosphine gas. The bag blanket is unrolled on the surface of the grain, it being most suitable for phosphine fumigation of ship-holds, steel tanks, large concrete silo bins, warehouses and bunker storages under plastic liners. With a dosage of 1.4 g/m^3 (including commodity) an average concentration of 160 ppm was observed at a depth of 12.8 m in the grain bulk after 5 days. The penetration of phosphine was estimated as 3 m per day. The ratio of silo diameter to height appears to play an important role in the ability of the gas to penetrate the bulk. The most favourable ratio of diameter to height was estimated as being 1:2. For a ratio of 1:4 in a bin 29 m high, the average penetration time was 1 m per day.

In the paper presented by Mr. Cook, a recirculation method adopting a low airflow rate was described. For the conventional fumigant recirculation a flow rate of 1.5 m^3/hr/tonne equivalent to an air change in 20 min. has been recommended, whereas for the "low airflow fumigation method" the time required for air change was estimated as 8 to 12 hours, which is equivalent to an airflow rate of approximately 0.05 m^3/hr/tonne. Accordingly the installation required for this method was a duct of reduced diameter for recirculation and gas penetration, and smaller fan power requirement compared with the conventional recirculated fumigation. With this method phosphine was applied on the surface of the grain bulk and the air-gas mixture was drawn from the headspace of the silo, through the return pipe into the fan, and from the fan the gas was distributed into the grain bulk through the aeration system. This method is not recommended for non-gastight structures.

Two phosphine distribution methods were described by Mr. Boland. In the first a low airflow distribution was experimented on. The airflow rate was 0.06 m^3/hr/tonne and seems to be very close to the method described by Mr. Cook. The second consists of a black painted galvanized duct designated to create thermal currents, intended to assist in gas distribution. In the latter method the black painted duct described as a thermosiphon, connected the roof with the base of the silo. Although adequate distribution of phosphine was specified, data on gas flow and gas concentration were not presented. It seems that the thermosiphon method deserves further documentation to clarify the relationship between duct size, climatic conditions suitable for the production of thermal currents, and other parameters which may effect the creation of adequate flow into the tube.

Full-scale field trials have revealed that various failures in phosphine distribution may lead to survival of insects and inefficient use of fumigant. An indicator of distribution based on the ratio of minimum to maximum concentration of phosphine, was presented by Dr. Banks and Dr. Annis. Using this criterion, the risk of selection of phosphine resistant strains of insects, may be avoided.

Distribution of CO_2/methyl bromide (MB) mixtures in vertical silos of 1,000 and 5,000 m^3 were described by Mr. Viljoen. The gas mixture was applied from the headspace of the bins. High MB concentrations were observed at 29 m depth, upon completion of the application of the gases. Although, lethal MB concentrations were detected through the major part of the grain bulk, in some experiments pockets of low MB concentration were observed. The distribution patterns observed with this method of fumigation may be correlated with the physical composition of the grain bulk and the behavior of the gas in the system.

The use of Ethyl Formate as a fumigant was presented by Dr. Muthu. An effective dosage of 400 g/m^3 for an exposure period of 72 hr was recommended. The simplicity in application of the fumigant, which is a liquid at room temperature, holds certain advantages. However, in large-scale fumigations, its low toxicity and penetration ability should be considered.

The importance of grain dust control and the generation of lethal atmospheres in sealed storages were described by Mr. Ripp. Different safety measures to be taken were emphasized on the basis of know-how gained during the application of modified atmospheres in gastight structures. The flammability limit of pure phosphine at atmospheric pressure was assessed in a study presented by Mr. Green. Adequate dispersion of the gas and avoidance of application of concentrated amounts of phosphine will reduce the flammability risks involved in the use of the gas.

The need for a monitoring instrument specific to MB has long been felt for gas concentration measurements in working areas. The equipment described by Dr. Wiseman should be brought to the attention of instrument manufacturers so that this equipment can be made available. Dr. Bond presented several techniques for determining ultra-low fumigant concentrations. The experience gained during experimental fumigation procedures, seems to have the potential of being adopted by equipment manufactures.

RAPPORTEUR'S REPORT

SESSION 7

FUTUROLOGY

Mr G G Corbett

When Mr Ripp was organising this Symposium I made a particular point that rapporteurs should be given enough time to clearly report on the sessions. As a result of that I am reporting on this morning's session. But, anyway I have the easier job, because that session will be fresh in all your minds and anything I present you would remember.

I would remind you that Dr Navarro opened that session which is entitled "Futurology" by saying that he didn't really know what it meant but would be glad if somebody would define it........ Futurology is the science and/or an art of predicting future events. If you use a computer its science, if you use a crystal ball its art. But that session was not only on futurology, however defined. It had the purpose of identifying gaps in our knowledge and of specifying the research necessary to "do" and this, I think, is not futurology. We apply our analytical minds to what we know at present and to what we don't know and we go ahead and find things out. But the last paper of this session was Futurology in the first sense of the word and we think about Jonathan Banks, and I will come to that in its proper place.

The session began with a paper by Dr Love an economist which is not really on the future in either sense of the word, but is the result of a study carried out in 1979 - 1981 on the comparative costs of controlled atmosphere and grain protection chemicals for protecting grain. Dr Love says that although the figures, the cost calculations, related to that period in general terms, they were still valid, the relativity between the controlled atmosphere and the grain protection still apply. I wonder, in view of the rapid developments in the last few years, actually 4 years since it was started, whether that was true - and you are welcome to comment in the discussions on how far those developments in the last four years in the costs of sealants and of sealing would effect the comparison that was given in the paper. I would, however, like to correct the impressions which arose in the discussion on the length of time over which the capital costs of sealing were written off to arrive at the cost per year of sealed storage. I asked a question and got an answer - twenty years - and I've now had time to read the paper and find that it was assumed that the reflector coat would be repeated every eight years. And as we also see in the paper the capital cost was 75% with the sealing operation and 25% with the equipment, written off over twenty years. Obviously the costs of sealing are pretty well loaded if it is assumeed it would be done every eight years.

Now for the operating costs. We are given the costs based on CO_2 and nitrogen by liquid tanker and phosphine by the methods we know of and have seen. The conclusions drawn were that the total cost of both capital and time and operating costs of the controlled atmosphere store were comparable to those using grain protectants as used in the Eastern States but that the cost of using CO_2 in a controlled atmosphere store are roughly twice fenitrothion cost. Now people may like to discuss how much the cost of protection may rise as protectants have to be discarded and new ones used. At the moment we have to use phosphine, pyrethroids and fenitrothion but who knows what would be used in one year, five years, ten years and I think thats a point for discussion.

Then we had Dr Jay's paper which, not being an entomologist I find rather difficult to summarise, but I will report as well as I can.

I think the diagram, which showed all the variables that can affect the results of controlled atmosphere operation, really sums up what he was trying to say. All the variables are around the outside and as I understood it, he showed that in practically every one of those variables our knowledge is incomplete. Whether it is on the effects of percentages of concentration of CO_2, the effects of temperature on certain insects respiration, the insect species, the insect life stage, the insect age and condition. I'm not sure whats meant by condition, whether its when insects get old like I would and lose their hair .... but anyway the insects age and condition is a factor and the relative humidity is important. I felt that the biggest gap existing in our knowledge applies to effective concentrations, where figures on research work showed that the 60% concentration seemed to be more lethal to internal feeders and 100% concentrations to external feeders, feeding insects of the kinds on which the research was done. And in the discussion I note that this was confirmed by Dr Bell from Slough. But when someone asked Dr Jay to use his crystal ball and lay down the minimum rate concentration and to be certain to get a complete kill we began to get lots of complications. The concentration of 60% CO_2 I noted at 5 to 7 days or held for 5 to 7 days at 27 degrees centrigrade, would do the trick. But this period would rise to 30 days as temperature fell to 16 degrees centigrade and if the temperature fell below 16 degrees centigrade you wouldn't do it at all. I hope Dr Jay will correct me if I'm wrong. But all that applied to internal feeders only. Higher concentrations were necessary for external feeders and something much longer and higher still for trogoderma. As the concentration is reduced say, to 35% CO_2, the time factor is longer and Dr Jay agreed that the concept of CTs, concentration time product, would be useful in working out a complete model of resistance. Controlled atmosphere storage systems with all the variables we had so well described, but, there were many, many gaps which had to be filled before such a model could be accurate. Those gaps, some of them he mentioned, some he didn't, will appear in the paper, but they relate to every variable that appears on the diagram and it simply shows that much more research needs to be done to fill in the picture.

Then there was the paper by Mr Calverley, which I will leave until the end because it is rather different in nature to the other three.

I take Doctor Jonathan Banks' paper next. I enjoyed this paper considerably It followed the title of the Session very well. Of course it was a look into the future. It defined what we were trying to do, the constraints, the impediments he called them, to a C A operation. First the sealing, second a long enough exposure period, and third the lack of assured controlled atmosphere gas supply. And his look into the future was focused on the last of those three, the gas supply. I note the limitation which applied to everything he said, which was that his talks were focused on controlled atmosphere operation for insect control on dried grain as is being done in Australia. He dealt with both nitrogen and CO_2, delivered by tanker, as a liquid, but doubts whether this practice can, or will continue, that "on Site" generation of the CO_2 and nitrogen will take over. He gave very good summaries, I thought, of the different kinds of gas available and the different concentrations and periods for which they should be used. On nitrogen, again there are different ways in which the supply is divided, the likelihood of those different ways being used, or not used and coming into use, and their relative appropriateness for the purge period.

Then he describes for us the different ways of using what is known as burnergas, leading up to the possible use of organic materials, local materials, where-ever you may produce this method that could be obtained from agricultural waste, rice husk, producer gas or methane produced from biomass. All these hold out hope of the method being applicable in developing countries and other countries at places far distant from industrial gas production facilities for gas requirements. You may forgive me for looking at these again. I thought they represented the best summary of what Jonathan said. The gas requirements were given for different forms of gas, both for purging and for maintenance. And these summaries will provide for many of us a very good reminder as we are looking at all the methods of controlled atmosphere of what is required. Then just as for nitrogen we had a run through the different ways of producing CO_2, which I won't read out, its only two or three hours since you've heard them. And what I thought was quite exciting the electrolytic oxygen pump, which I didn't quite understand, but will no doubt when I read the paper, whereby we pump air from one container to another increasing the oxygen that flows back to atmosphere.

But, it was this last overhead which I think summed up the value of Dr Banks' look into the future. It is the one, you remember, in which he starts with the ancient hermetic, the Inca stage of C A operations and then with the line saying "You are here" - I love these archaeological time scales which start 5 or 10 million years back then you come almost up to the present day and you can hardly see it and he says "You are here" - there's been an awful lot gone before. In this case the next stage that Jonathan Banks predicts is the use of a burner, after that recirculation, using something like electrolytic method of oxygen reduction and finally, we don't know how far in the future, a modern hermetic method which will truly return, Mr Chairman, to the natural method of controlled atmosphere, reduced oxygen. Either the natural method, where natural respiration, insect respiration reducing oxygen, the assisted method of a sacrificial area, a bag of wet barley, or perhaps that comes in the artificial system. The bag of wet barley alongside the fermentation box is used in China to reduce the oxygen in the air that is going into and coming out of the controlled atmosphere enclosure. But in any case these methods are all what we call natural methods and they will, I agree with Jonathan, eventually be the ideal at which we should aim. A natural airtight reduction of oxygen to give us CO_2 or nitrogen, and not phosphine, as we use at the moment.

Right, now let me turn to the last paper which was so different in nature from the others. Mr Calverley's. I'm so familiar with my friend David's ideas and we have discussed them back and forward so many times that I find it difficult to make an objective summary. But I will try.

He very neatly summed up at the beginning of his talk the ingredients of a perfect controlled atmosphere storage as demonstrated to us during this last week. The four points of good management, good enlightened management which includes the commitment by management to put the necessary resources at the disposal of the technicians. Secondly, a very good link between research and management and technicians. Thirdly, a good link between industry, manufacturers of sealants or agents and the management. To finally the availability of materials to suit the purpose from anywhere. And then he went on to discuss the position with the developing countries, we are now talking about tropical and developing countries, showing I think in that description how very rarely these four desiderata which have been available here are available there.

He discussed on farm storage the available systems and the fact that the work in the last five to ten years has shown that as long as a traditional storage system is not disturbed losses are likely to be low. But disturbance is going on in developing countries, thats what we mean by development - disturbance. Its going on all the time and new variations in cropping patterns, higher production by each individual results in the system being disturbed. The question is - how far do we try to modify this exisitng system, which is working well now, to cope with the new problems? The answer, I think, is that we go very cautiously in trying to apply any of this controlled atmosphere technology in that situation.

In central storage, the bigger types of storage, which in round figures comes to some 200 million tons in the developing countries, roughly half local, half of it is imported. Again management, application by management of existing methods of insect/pest control are not as good as they might be. Of course, Mr Calverley rightly mentioned the tremendous variation in conditions between developing countries and that includes benevolent management from one end of the scale to the other in the developing countries.

I think its appropriate to mention the point that was brought up in the discussion, that in certain countries controlled atmosphere storage is already being talked about and the fact is that Malaysia is a pro atmosphere country and has a milled rice storage nearly finished. That as far as I know is the only example of a large scale store, using this storage method, although Mr Calverley referred to fumigable stores which have been built in Africa, using phosphine fumigations. Again for simple storage it is his conclusion that the future of the controlled atmosphere storage, in the near future at least, would be moderate. Caution should prevail. Because of the many constraints, people who come to developing countries and people who work there know very well. This is not to say that development in certain places for selected purposes should not be tried. It should, and as Mr Calverley pointed out that these developments are usually donated. It is therefore very important that those developers - and I hope Australia is one - should utilise to the full the experience and the expertise that is available within our own countries or elsewhere when they finance any kind of controlled atmosphere development in developing countries. Therefore if there is anyone in the hall this afternoon who can have an influence on a donor, please take that message back, use the expertise, particularly the expertise from Australia which is now available for controlled atmosphere, before investing in development in developing countries.

In the discussion on Mr Calverley's paper a subject was raised which we have mentioned many times during the week and that is the question of the developing resistance to phosphine and the imperative needs to improve the standard of phosphine fumigations. The phrase that Dr Evans used which I thought summed it up is to "avoid repeated unsuccessful phosphine fumigations" I don't think there is any need for me to stress this as it has been mentioned several times already and it is an even greater reason why the excellent means that we have seen here of sealing should be made available widely and as soon as possible so that everyone who is concerned with fumigation can apply methods that are appropriate to improve the standard of sealing for fumigation.

Questions and Answers

Moderator: Mr B E Ripp

Prof. Dr K Zanon, Italy: Talking about the future and futurology I think we have to keep in mind that there is a definite direction towards avoiding the use of toxic materials in most countries in the world. Now in this respect I think that low oxygen is a good approach to the problem. I have to remind you that there are big political movements for environmental improvements, there are many reports regarding toxicity brought forward with the integrated pest control methods which are not well known in many, many countries. So I think we should keep in mind also these problems if we are storing our grains or our agriculture produce. There are methods, different methods of producing, of course, these low oxygen atmospheres. We had some examples this morning and I want, since I have a special experience on generators, to point out a few basic facts about those generators. They are in common with carbon dixoide that they are not toxic at all, they take in no residues and they don't create any resistance existing against anoxia, we know that. Besides if you go to generate this especially there are some special reasons for informing them, since you can produce inert atmosphere locally on the spot, mostly with fuels which are available and not too difficult to adopt. They don't give the transportation problems which others do and so that's again a point in advantage. Besides the generated atmosphere, inert atmosphere is a combination, as we know, of very low oxygen in CO_2 of 0.5% and 9% to 11% or even more carbon dioxide and we know that is a definite action against insects of such combinations. We know that from publications and scientific work done all over the world. Finally, I must say that this system is applicable very well to a lot of special applications, special products like Alfalfa in France, it's applicable for oil seeds, its applicable to tobacco and so on. So there are a lot of factors we can keep in mind, which give us a proper certainty, a security that such matters can be applied in the future in special and certain cases. There will be a large program arising if this can be economic, and I can tell you from experience that it can be done economically, since there are machines for every size going up to 500 cubic metres of inert gas per hour. Such a machine will cost around, for everything \$25,000/30,000 - thats much less than the figures we got the other day. I'm very optimistic on cereal grain storage and storage of produce and I'm sure that different methods will be applicable in the future and the method of generation creates a clean atmosphere. Thank you very much.

Mr B E Ripp: Thank you Professor for your comments.

Dr S Navarro: May I make further comments about futurology. Firstly I thank Mr Corbett for this definition and secondly, I wouldn't like to ignore this terminology. The truth is that we, as entomologists or biologists working in this field, have quite a lot of unsolved problems. For instance when we think about this resistance problem we are talking all the time about phosphine resistance, or to some it's so called tolerance for others it's resistance. I wish to mention that even for carbon dioxide there is a certain tolerance evidence in some species and we don't know yet what are all the problems we are going to face. We do know that one of the problems we should look at is the tolerance problems even to oxygen atmospheres. It has not yet been proved that all the insects we have can not tolerate the very low level oxygen concentrations. But, this is only one side of the problem. We know the problems arising during these treatments: we have to solve them and we know what is to be done for the next, at least 5 to 10 years time and thirdly, if my comment stimulated some discussion among the delegates I am glad. Thank you.

Mr G Love, Bureau of Agricultural Economics

It was very useful to, in fact, present the papers after we had seen a practical demonstration, because that gave you some practical idea of the kind of costs involved in sealing for modified storage. What we did is that we got the stream of sealing and modification costs spread over a twenty year period. Now within that stream you have the costing of the reflector coat every eight years. When these costs were first derived - and none of us were very sure how long the seal would last - we thought we dare not be too optimistic. In regards to the other comment on the paper, the relativity between the fumigation and modified atmosphere techniques probably hasn't changed much to chemical protectants. That's pretty obvious now that the relativity has produced costing of fumigation modified atmosphere and the costs don't appear to have risen much over the last three years, whereas the costs of protectants have.

Mr B E Ripp:

Thank you Graham.

Dr Jay, USA:

Dr Navarro commented on the apparent tolerance to CO_2. The only time tolerance has shown to be present is at 100% relative humidity. If you are going to store your grain at 100% relative humidity you are going to get tolerant insects.

Dr S Navarro:

Of course I should state, that Dr. Bond's work on this tolerance in insects to Carbon Dioxide and our work on the same lines have mostly been to look at the future, what will happen, and I won't say that this would show up any emergency situation. so it has more scientific value than the practical.

Thank you.

Mr P Williams, Dept. of Ag. Vic.

I'd just like to extend slightly Graham Love's point about costs, point out to the particular people overseas that in the Eastern States of Australia we have insect resistant problems with resistant Rhyzopertha and a result of that, frequently an insecticide cocktail mixture involving fenitrothion and bioresmethrin is used and this considerably increases the costs of the insecticide treatments. Graham quoted 70¢ to 55¢ or so for such treatments per tonne which is much greater than the cost of fenitrothion alone and this is what we can look forward to in the future in other areas.

Pro. Dr K Zanon:

I hate normally to talk twice, but this time I am obliged because maybe there is a misunderstanding between me and Dr Navarro. I said there is no resistance that I know to anoxia for insects and I firmly agree that I don't have any information of the opposite.

Dr Desmarchelier: C.S.I.R.O.

We've got to a position where Dr Banks, Dr Jay and lots of people today have pointed out we don't have the basic biological data on most of these insects. There is a lot of discrepancy in the literature. You do a study of resistance. The first thing you have to do a whole lot of cross checks. There is a myth that it has been done. If we must be thoroughly relevant - because, look at the history of pesticides - resistance very rarely came out of the laboratory studies, it came in the field. Do you really think we can says things, like we haven't got resistance, we can't get resistance, and all the rest of it?

Mr H Greening:

I'd like to make a comment. Because the Conference is due to reach a conclusion very shortly before all the papers have actually been presented, (it continues next week) in that it would be easy to make some blanket recommendations that could have far reaching effects. I was thinking particularly about phosphine fumigation on which the farmers of the world actually have depended for quite some time to disinfest their stored grain, but its not a practice that is confined to Australia at all. We have a history of some quarter of a century of use of phosphine on farms in Australia, apparently without any serious problems of resistance developing. In my experience I have found that phosphine applied by probing to grain in small storages can be effective at quite low dosages, down to 2 grams to the tonne, and I feel that enthusiasm for sealing of storages is essential, I imagine, for a method of applying the formulation to the grain surface. You might forget that there are other ways of applying the fumigant and perhaps make some recommendation that excludes that sort of use.

Mr B E Ripp: Is there anything further you would like to discuss? I am surprised that there is no reference to some of the major problems that caused quite a lot of discussion through the week, such things as moisture control, quality control and auto ignition of phosphine. Is there anything that you are sweeping under the carpet, you'd like to get off your chest?
O.K. Thank you all very much, perhaps they may come up during our assessment of objectives.

CONCLUSIONS - ASSESSMENT OF OBJECTIVES

Moderator:
Mr B E Ripp: To recap the purpose of our Symposium I will repeat the objectives set.

1. To discuss developments in the technology of Controlled Atmosphere and Fumigation in Grain Storages since 1980.

2. To demonstrate progress in methods and materials needed to seal all type and sizes of grain storages.

3. To consider future promotion and application of controlled atmosphere and fumigation techniques.

4. To determine the direction of future research necessary within the total ambit of the concept.

I think we have dealt effectively with the first two aims and objectives and would like to look directly at the Third and Fourth which is the promotion of the system and research that is necessary in the future and what may be necessary to bring about that research with the support of the researchers. I know there is alot of work going on right now and no doubt each of us will continue to go about our own research pattern, unless we get some indication from this group as to where research should be heading. We know that manufacturers of materials are developing considerably in looking for increased flexibility, less permeability, easier application of membranes, long lives, low costs etc. We know that we are looking at sealing methods, particularly with automation or some sort of remote application in verticle storages where access is difficult. We need to give some thought to the co-relation between laboratory results and field results. But I think it is the more major research and the direction of this major research, the onward promotional systems, that would do with some discussion. Have you any points to put forward, please?

Dr J Banks: I think one of the things that has come out of this Conference is that there is a moisture problem that somebody is going to have to look at and that we are really going to be in a position where, should there be another Conference, at least part of that conference is going to have to be based on the progress in controlling the moisture problems and also we should be looking at the use of controlled atmospheres at higher humidities, higher moisture contents, than we have really been debating over much of this Symposium.

I think this is something that is very important. And one of the hopes that is available in controlling moisture is recirculation. I think that this is something that some delegates may not have noticed in Charles O'Neill's paper. For instance, he is talking about a recirculation system in which the inert gases are recirculated and in addition to that there is a chiller in the recirculation system which removes excess water. One of the features of the successful Italian system for storing fairly moist barley in Italy has been the use of dry gas into the headspace of storages. In Charles O'Neill's system there is another case where you feed dry gas to the head space of the storage which has got a lot of very damp grain below it. I would hope that this is a thing that in future will give us some cause for hope to get away from the moisture migration problem. We are looking at something here that, while we have not focused on it, I think its an important advance, is something that a lot of people independently and concertively have been looking at, may well come out of this conference. We must, I think, be looking at what happens further.

Mr B E Ripp: I would comment that, I'm sure you are right that this will be the next evolvement. The title of the next symposium could be "Recirculatory Fumigation, Controlled Atmosphere, Drying Conference".

P. Annis: I would just like to make a couple of comments over the full range of papers. They are in a way both a plea for a bit of caution, and for a lot more information. Both with phosphine and CO_2 we have some biological information over some parts of the range of, as Ed said, with CO_2 over the wide matrix of the things which are available. My plea is that we who are involved in the biology of both phosphine and CO_2 should make efforts to fill in the gaps, and that we should not start using the minimum dosages for either of these two gases because the information is very, very hazy and surely one of the ways of encouraging tolerance resistance is to use concentrations which are marginal in structures which may not be fully sealed.

Dr E J Bond, Canada: The instance of resistance to phosphine has been mentioned several times and I think in this very crucial area we should look into it very carefully. If you think of the fumigants we soon come to realise, these are a very unique group of compounds that do a number of things, are very versatile and very effective materials. Of the materials that man has successfully used for fumigation there are very very few compounds, you can almost number them on the fingers of your hands. The number of materials are declining for a number of reasons and at the present time are limited to about two, these are methyl bromide and phosphine and of these phosphine is perhaps the most widely used, perhaps the most useful material at the present time. Now we know from past experience what resistance does, how the insects can respond and become resistant. We have a lot of experience in knowing what can happen and here is a situation where we seem to be doing virtually nothing about it. We know the problem is there, but nobody is taking any active steps to not only do anything about it but to even understand the problems. I suppose you might think - Well who should take the responsibility - should it be Government agencies, or should F.A.O. be involved, or who should be involved? Certainly somebody should be involved unless we are going to do as the ostrich does "bury our head in the sand" - this does seem to be what we are doing and I think it is very serious. I think we should be looking into this problem and trying to do something about it.

Mr B E Ripp: Thank you Ed.

Mr L Booth, S.A.: I think the message to me is very loud and clear. We should be looking into the real essence of our problem. The nature of the beast we are trying to eradicate, we are concentrating a lot on atmosphere and cells we put the grain in but I think I would like to ask Dr Corbett in his exalted position, and I mean it quite seriously. Could he tell me how the world can have an influence into this investigation into the insect that we are trying to control? I feel sometimes that we are trying to hit the thing on the head, rather than make a detailed valid investiigation into the beast itself so that we can more quickly try and cure the problem.

Mr G G Corbett: The question is clear enough, but I'm clearly not the man to reply to it. I think of those present, Dr Evans, in his position, could give an over-view of the insect problem and what people are doing about it.

Dr D Evans: I don't really think I'm in a very exhalted position at all, but I'm certainly in a difficult position in as much that I'm speaking from one organisation, I am speaking as a biologist and entomologist working in an area which is not concerned with fumigants or controlled atmospheres.

What perhaps I can say is a little about the efforts of the Stored Grain Research Laboratory in Canberra. We, in fact, have two people concerned with the biology - or at least we hope to have two people concerned with the biology of insects, their response to fumigants, their response to controlled atompsheres and as I think we have seen during this week - Controlled atmospheres are fairly well known from a technology point of view. The task now is to learn more about the biology of the insects. To this end we see a change of emphasis within our group that Peter Annis has been working, together with Jonathan, on the development side. The technology side will in future be more concerned with biology, the possibility of resistance to controlled atmosphere and as Jim Desmarchelier has rightly pointed out "Before you can talk about tolerance or resistance you've got to know what is susceptible and what are the boundaries of susceptibility" and quite clearly as people much more versed in this area than I am - Ed Jay and Chris Bell have pointed out - "Biology is very scanty".

Following a review of our research programmes in the Stored Grain Research Laboratory we have concluded that we ought to know far more about the physiology of how carbon dioxide works. We have therefore advocated to our Chief of Division that he should appoint a physiologist to look into these things and so far the response to that suggestion has been favourable. As far as fumigants are concerned, in our laboratory we have Dr Bob Winks, who is unfortunately not with us this week, who is very involved in the definition of ground rules for resistance to phosphine, tolerance patterns, that sort of thing, methyl bromide also.

We have a Mr John McKeller who is looking into aspects of how and why phosphine is taken up by the insect. This involves radio-metric techniques trying to find out how the gas penetrates into the pest, where it goes to and how it is metabolized. Because once again you've got a lot of factors involved and why do pests become resistant to phosphine? What are the mechanisms, is it just that the stuff doesn't penetrate, is it that the stuff is metabolized quickly or whatever? I think that's the thumb nail sketch of our Laboratory's activities. Through you, Mr Chairman, I think we really ought to ask Dr Bell and Dr Bond, perhaps, to describe some of the activities in their laboratories.

Mr B E Ripp: Thank you, Dr Evans.

We are actually running a bit short of time now, and I'd prefer if we could refer to specific points of research that people think should be done at this stage.

O.K. - we are running right on time, and I think thats ideal after such a full week.

I would like you to join with me, if you would please. To all participants thank you for participating so well, to our Chairmen - those learned and distinguished gentlemen who steered us through the various sessions and to our rapporteurs. Thank you most sincerely for your efforts.

SESSION 9. (SECOND WEEK)

THE BUNKER SYSTEM

Papers by:

C.J. Yates and R. Sticka	-	Australia
S. Navarro: E. Donahaye: Y. Kashanchi: V. Pisarev and O. Bulbul	-	Israel

DEVELOPMENT AND FUTURE TRENDS IN BUNKER STORAGE

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ABSTRACT

The Grain Handling Authority of N.S.W. has developed a system of bunker-type storage that was originally envisaged as an extension of the technique of temporary storage. This has now reached the stage where it is a satisfactory form of permanent storage where only minimal capital requirements are warranted, where capital is simply not available for the construction of storages, or where a storage facility must be provided at very short notice.

The PVC-covered bulk grain storage system is capable of directly responding to two of the current needs of bulk handling organizations: the need to minimise handling and storage costs, and the need to control insect infestation in grain without the use of chemical protectants and, possibly, without the use of fumigants. These storages are now routinely being constructed to 50,000 tonne capacity, and feature movable concrete banks and bitumen surface. A range of portable grain handling equipment has been designed to complete the new storage and handling system. The operational problems encountered in bunker storages include grain contamination with earth, damage to the covers and difficulties with grain receival under some conditions. All these problems have been overcome or reduced to a low level.

A major advantage of the PVC bunker-type storage is that it can be fumigated satisfactorily with phosphine. This treatment is a very cheap form of fumigation and experience to date indicates that the storages are sufficiently gastight to render it completely effective.

Grain storage in bunker systems is now substantially cheaper than in permanent structures (e.g. steel bins $20 per tonne capacity, bunkers ca. $8 per tonne capacity per annum, including handling and capital charges).

INTRODUCTION

With wheat farming pushing further and further into hitherto marginal country, some bulk grain handling organizations in Australia will find it necessary to provide extensive storage capacity in areas where harvests fluctuate widely from season to season and where in some seasons there may be no crops at all. Other organizations may face the need to store an increased crop, but have access to only limited capital funds. In these circumstances there can be no justification for a high level of capital investment as, with the exception of the labour requirement, all the basic requirements of storage can now be met by the use of low cost bunker-type storage, as developed in New South Wales in recent years.

BACKGROUND - GRAIN HANDLING AUTHORITY OF N.S.W

Role of the N.S.W. Grain Handling Authority

In Australia, the primary role of State bulk handling authorities is to act as the agents of the Australian Wheat Board for the receival, storage, preservation and handling of wheat. The operations of each authority include:

- provision and maintenance of a country elevator system and terminal facilities for the storage and handling of grain;
- provision of labour to operate the elevator system;
- arrangements for the segregation of different grades of wheat and other grains in the elevator system;
- arrangements to control pest infestation of wheat in storage;
- receival of wheat, by quantity and grade as specified by the Australian Wheat Board, and arrangements for the transportation of wheat consignments to destinations nominated by the Australian Wheat Board.

Development of the N.S.W. Grain Storage System

Wheat was first received at country silos in the 1920/21 season, when total receivals amounted to 52,844 tonnes from a crop of 1,513,865 tonnes. Record New South Wales wheat crops since the 1920's are given in Table 1.

Table 1
Record wheat tonnages in N.S.W.

Season	Total crop (tonnes)
1932/33	2.1m
1947/48	2.6m
1962/63	3.0m
1963/64	3.3m
1964/65	4.1m
1966/67	5.5m
1968/69	5.8m
1978/79	6.5m

The development of our bulk storage system is summarised in Table 2.

Table 2
Storage Capacity in the N.S.W. bulk handling authority system

Period (approx.)	Total capacity available at end of period (tonnes)
1918-1920	332,030
1921-1936	796,681
1937-1942	892,071
1943-1950	897,514
1951-1960	1,981,204
1961-1965	2,616,823
1966	2,775,762
1967	3,161,951
1968	3,614,001
1969	4,412,505
1970	5,293,744
1971	5,705,651
1972-1973	5,764,981
1973-1974	5,783,100
1974-1976	5,829,000
1976-1977	5,855,400
1977-1978	5,854,400
1978-1979	5,966,750

Table 2 clearly reflects the accelerated rate at which the storage system expanded over the period from 1951 to 1970. It is interesting to note that when silo construction was interrupted by World War II the total capacity, including terminal elevators, stood at approximately 847,029 tonnes. In fairness to the authorities of the time, however, it must be stated that the need for bulk handling facilities was much less pressing than it became in the post-war years. Many farmers, in fact, preferred to deliver their wheat to bag stacks. Pre-war, moreover, the production of wheat in New South Wales had never reached 2.15 million tonnes.

During the 1960s, as record crop succeeded record crop (Table 1), the demand for bulk storage facilities accelerated. Considerable strain was thrown upon the resources of the system. Despite the enormous amount of storage built from 1961 onwards, the increase in wheat production continued to out-strip that of the capacity of the elevator system, except in the drought years 1965/66 and 1967/68. The storage lag was not finally overcome until 1969/70. The total space available for the 1969/70 season was slightly greater than the record delivery of the preceding season.

One can appreciate the physical pressures exerted at the point of receival, reflected in a constant flood of telegrams and telephone calls to Head Office asking for, or demanding, rail trucks to make space for futher receivals. Nowadays such telegrams are quite rare. Very long queues were once a familiar sight at country plants, often lasting for weeks on end. Even when space was available the queues at some plants were so lengthy that farmers managed to deliver only one load per day, or sometimes one load in two or three days. Wealthier or more enterprising farmers countered by hiring several carriers so as to have a number of loads in the queue at one time.

Another practice was to listen to early morning broadcasts concerning truck placements or watch to see where rail trucks were being placed for loading. In the days before some form of zoning was introduced, farmers' deliveries were unrestricted. They could thus move their vehicle from one queue to another in pursuit of space, and it was not uncommon for wheat to be carted distances of up to one hundred and sixty kilometres.

With the exception of a very small number of temporary bulkheads, all storage built up to 1950 was in the form of orthodox vertical silos. From then until very recently, storage in horizontal sheds predominated; so much so that of the total capacity of almost six million tonnes, only 25% is vertical storage. In recent years, permanent storage expansion has been in the form of vertical cells and other storage in the form of large PVC-covered bunker storages. We now have about 480 country elevators (including sub-terminals) and a growing number of bunkers, ranging in capacity from 800 tonnes to 60,000 tonnes. A large bunker storage is shown in Figure 1. These storages are located at approximately 270 centres. Handling rates vary from 50 tonnes per hour to 400 tonnes per hour.

THE BUNKER-TYPE STORAGE

Application

The Grain Handling Authority of N.S.W. has developed a system of bunker-type storage. The system was originally envisaged purely as temporary storage but has now been developed to a stage where it is a satisfactory form of permanent storage. It is particularly appropriate where only minimal capital requirements are warranted, where capital is simply not available for the construction of storages and where a storage facility must be provided at very short notice. In the 1978/79 harvest, 200,000 tonnes of grain was stored in bunker-type storages, and in the 1979/80 harvest approximately 1.5 million tonnes of grain was stored in this fashion.

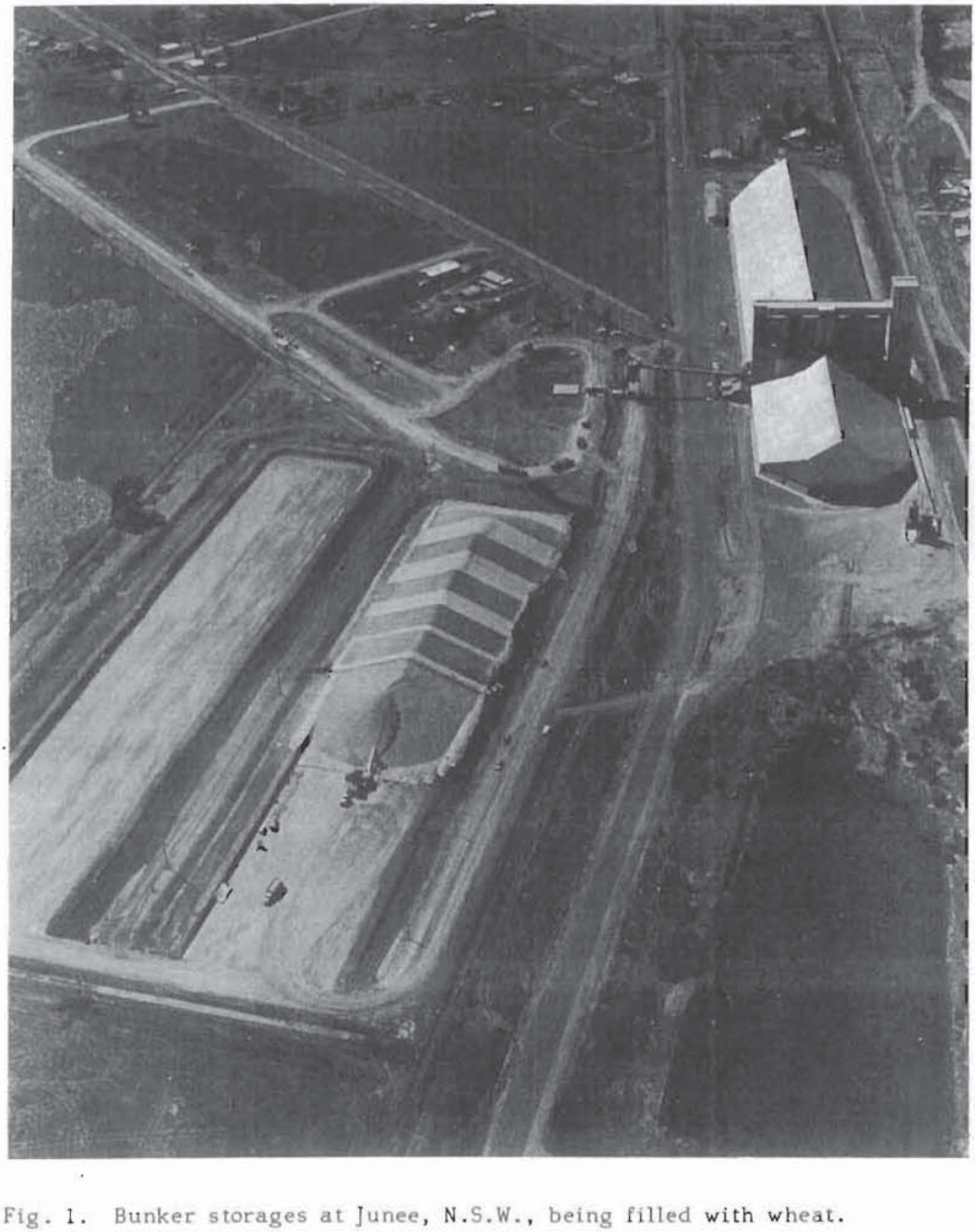

Fig. 1. Bunker storages at Junee, N.S.W., being filled with wheat.

Development

This new bunker-type storage evolved from an earlier concept of underground storage, now greatly modified with assistance from commercial suppliers of materials and equipment to make it more efficient operationally and more economic.

Some years ago, the Commonwealth Scientific and Industrial Research Organisation (CSIRO), in collaboration with the Authority, put down an experimental underground storage in the northwest of New South Wales. The trial was designed to provide medium to long-term storage for grain at a relatively low cost, and was undertaken in anticipation that the storage provided would obviate any problems of insect infestation. The store was entirely underground. A pit was excavated and filled with wheat and then covered with a thin PVC-cover and then with soil. Undoubtedly, in certain soil types and selected positions, this type of storage does provide a solution to the problem of long-term storage where permanent storage facilities are not available, but it was found to be highly labour-intensive and to involve working conditions which are very severe. The earth covering presented particular problems. A further trial in conjunction with the CSIRO involved the provision of an above-ground storage covered by sand and earth. This used an earth-walled bunker with a PVC cover on which approximately a metre of covering soil was placed. This type of storage has been used quite extensively in Victoria, but the use of the earth cover is now discontinued.

While it was an improvement on the underground storage, the earth covering in particular presented appreciable operational problems. Having regard to the availability of high-quality PVC materials, the Grain Handling Authority of N.S.W. took the view that an earth covering was not necessary. It was considered that storages could be made more cheaply and in an operationally superior manner by covering the grain with a heavy-duty, high-quality PVC material.

The Authority carried out a trial of this system in 1977 which was relatively satisfactory. Further trials were planned, but a heavy demand for additional temporary storage in the 1978/79 season necessitated immediate and relatively large-scale use of this type of temporary storage. The need increased still further in the 1979/80 season, when 1.5 million tonnes of wheat was placed in this type of storage.

Operational Problems Encountered

While a number of problems were experienced in the earlier stages, all of the serious problems which can be resolved have been resolved although a few minor ones remain.

Problems which have been experienced included:

a. Admixture of some earth with the grain.

b. Admixture of stones with the grain.

c. Damage to PVC material from various causes, including birds and hail.

d. Failure of adhesive, allowing PVC sheets to come apart.

e. Inability to receive into storages in high wind.

f. Vandalism of storages which were not protected by fences.

g. Rain during inloading and outloading.

With respect to (a), admixture of earth with grain, the problem still exists where earth banks are used, although it has been, in all but a few instances, quite minor. Provision of concrete banks rather than earth banks and the sealing of the storage floor surface with asphalt completely eliminates the problem and also (b), the admixture of stones. A solid asphalt base was also found suited to direct tipping from growers' vehicles, greatly assisting the rate of receival into the new type of storage.

The problem of hail damage, (c), still remains. What might be termed normal hail does little or no damage. It is only when large, jagged hail falls that serious damage occurs. The authority has had bunker storages at 96 different sites (with several bunkers at some sites) over the past few years and only one bunker has been seriously damaged by hail. In this case, no significant damage occurred to the grain but there was considerable work involved in repairing the PVC cover. However, the incidence of such damage over a number of sites is very small and, while it cannot be ignored, it can be easily coped with, providing adequate inspection of storages is maintained.

The problem of the failure of the adhesive used on the PVC has now been overcome by sewing the sheets together rather than using a sealant. This also obviates the need to clean the used PVC sheets as thoroughly as was necessary when an adhesive was relied on. A bag-sewing machine, preferably one that provides a lock-stitch so that there is virtually no possibility of the stitch coming undone and allowing the sheets to part, is used to sew the sheets. It is only fairly recently that stitching has been adopted but it appears to have been completely satisfactory. A sealant is provided over the stitching to ensure gas-tightness.

Inability to receive into storages in high wind remains a problem, although the new grain throwing machines referred to below (Section 4) can be used in moderate wind, depending, to some extent at least, on the direction of the wind. The ideal loading machine would be one which is not influenced by wind and there is no reason to believe that such a machine cannot be designed in the future.

In respect to (f), vandalism should be almost completely eliminated if good, solid "man-proof" fences are erected around each storage. Such fences are also a necessity if storages are to be fumigated.

Fumigation

In the 1978/79 harvest, grain loaded into bunker storages was treated with fenitrothion (12 mg/kg^{-1}) and bioresmethrin (1 mg/kg^{-1}) for protection against stored products pests. However, in the 1979/80 harvest, approximately 250,000 tonnes of grain loaded into bunker storages was not treated with any chemicals at all, whereas the remaining quantity was treated with fenitrothion (12 mg/kg^{-1}) only. This decision was made because of the lack of sufficient bioresmethrin from commercial sources and the successful fumigation of a 10,000 tonne bunker storage at Grenfell in September, 1979 with phosphine-generating sachets manufactured by 'Detia' of West Berlin (Banks and Sticka 1981).

In the trial at Grenfell, aluminium phosphide sachets in the form of a blanket (30 cm wide x 4.6m long, containing 100 x 34g sachets, each liberating 11g of phosphine) were placed on top of the grain mass below the plastic covering. This trial demonstrated that phosphine fumigation of PVC-covered bunker storage was feasible commercially, and gave data on the dosage of phosphine required.

Further trials (Banks and Sticka 1981) were carried out in the PVC bunker storages at Grenfell in March 1980 using phosphine-generating 'plates' of both aluminium and magnesium phosphide as supplied by 'Degesch' of West Germany. Representatives of 'Degesch' were present and assisted in the initiation of these trials.

Commercial fumigation of the 1.5 million tonnes of grain placed in bunker storages during the 1979 harvest intake throughout New South Wales was commenced on 7th January, 1980 and has continued since the completion of the harvest intake.

The Grenfell trial, which was intensively monitored, and subsequent extensive commercial use of the phosphine-generating blanket have demonstrated that this fumigation technique can be applied easily to PVC-covered bunker storages as constructed in New South Wales. It has been shown that these storages are sufficiently gas-tight to retain phosphine for long periods, thus allowing natural gas movements to distribute the gas throughout the bulk. Futhermore, the length of time that phosphine is retained is sufficiently long for even a low concentration of phosphine to be effective. The levels of phosphine gas obtained are generally sufficient to give a product substantially greater than that necessary for complete insect kill. It is only necessary to carry out minimal gas monitoring during fumigation, and the treatments conducted to date have all given consistent and satisfactory results.

Whilst the blanket technique is simple to use, careful attention must be given to ensure that enclosures are well sealed. Because of slow and continuing evolution of phosphine gas from blankets, fumigated stacks should not be moved for about 30 days after fumigation. In general, this delay should not create any operational problems and, from a pest control point of view, the evolution of phosphine gas is advantageous in maintaining the overall phosphine concentration for sufficient time to ensure adequate distribution of the gas within the bulk, despite the small, but significant, loss rate.

GRAIN HANDLING SYSTEMS FOR BUNKER STORAGES

To place the grain into the storage requires a mobile throwing machine which throws the grain to the height required and can be retracted as the length of the storage grows. When the large storages were conceived, it was realised that, with existing equipment and procedures, it would take 100 days to inload and another 100 days to outload a 60,000 tonne storage. A new thrower was developed which could place 250 tonnes in an hour, or with 2 machines, place 5,000 tonnes in a day. Two 50,000 tonne storages can then be filled in 20 days. The throwing height was increased from 7.5 metres to 11 metres with this new machine. The same machine outloads at 200 tonnes in an hour or 2,000 tonnes in a day. This machine, which we call a 'Lobstar' (Fig. 2), represents a major step forward in handling large bulks of grain in open positions. The Authority now has about 40 Lobstars, half of which are electric-powered and half diesel-powered.

While labour costs for this type of storage had at first appeared to be high, the capital investment was very low. Provided a fast receival system could be coupled together with a good outloading system, the total scheme could be very cost effective. Development effort thus began to centre on high-capacity portable equipment to suit the new bunker storages.

New modifications to the highly successful Lobstar were developed, greatly improving the versatility of this machine such that the unit could be positioned to load grain directly into rail wagons (Fig. 3). Rail outloading capacity could now be flexible, and could be positioned with most effect in co-ordination with the State Rail Authority's train program. Large front-end loaders with specially designed wheat buckets were obtained to work in with the Lobstars. Special tractor attachments minimise labour needs on the new storage sites.

Dealing with the road truck traffic at bulk sites, new methods of marshalling traffic were developed using traffic lights, modern streamlined sampling stands that handle four vehicles at a time, and multiple weighbridges.

The design of these bunkers was greatly improved. Large 50,000 tonne bunkers are routinely being constructed with movable concrete walls, and a solid asphalt base to permit direct tipping of growers' vehicles. New methods of PVC tarp handling using cables and capstan have minimised the labour requirements to place these large covers.

With the successful development and introduction of these "building blocks", the Authority has now laid down the basis for a new and highly cost-effective grain handling system.

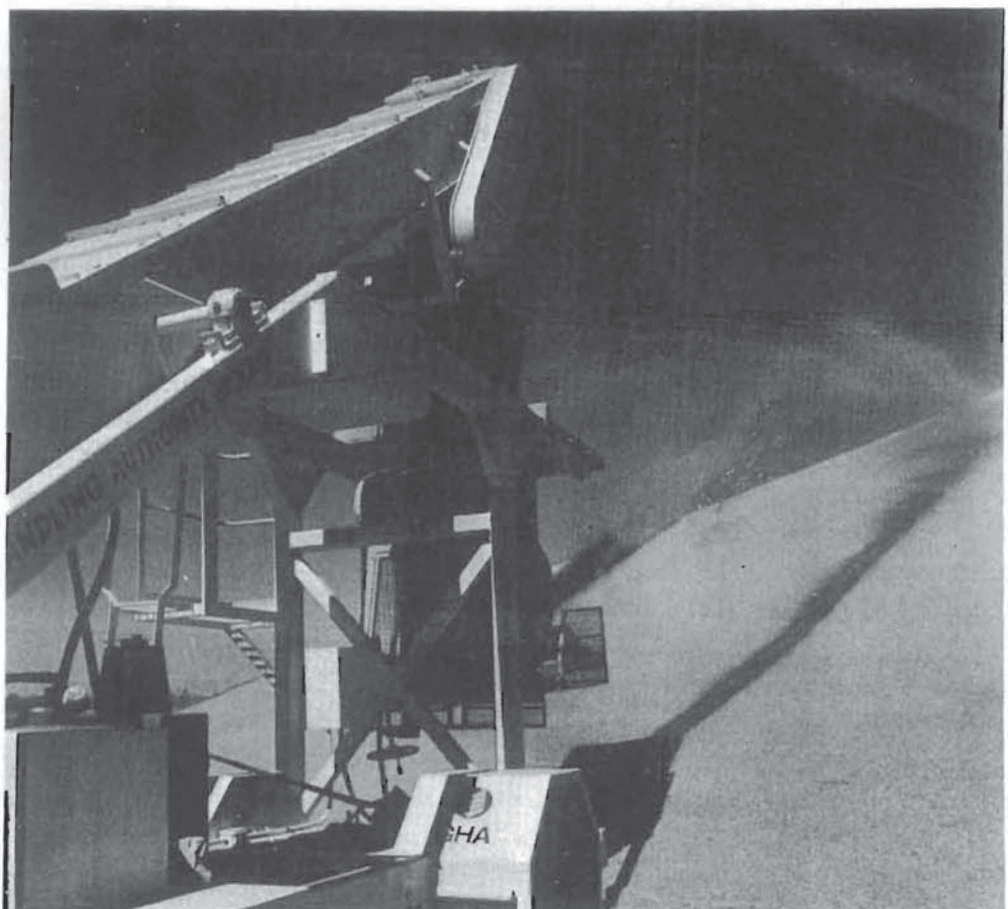

Fig. 2. A G.H.A. designed 'Lobstar' grain thrower loading wheat into a temporary storage.

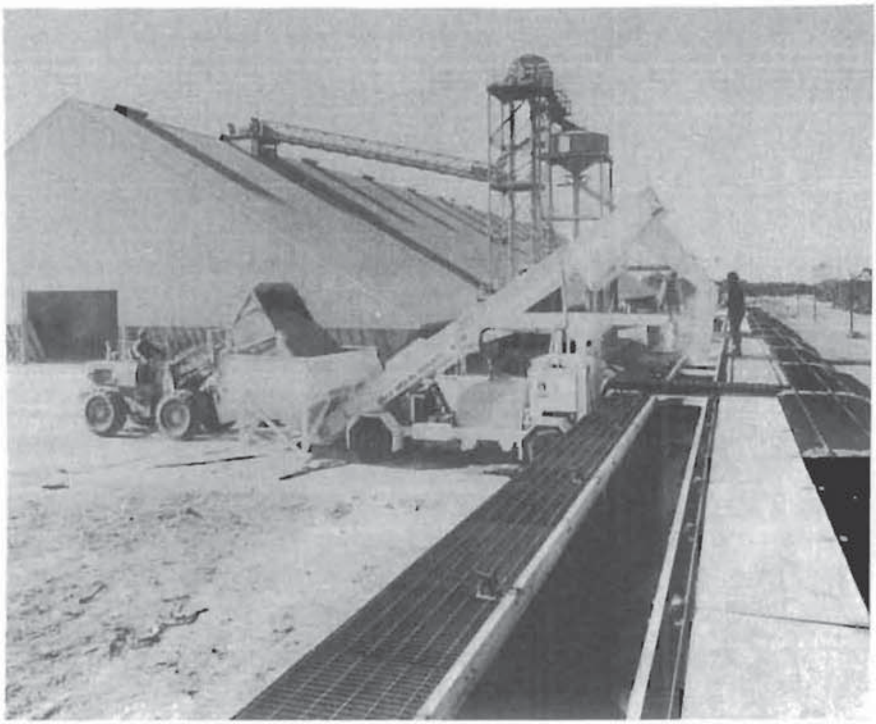

Fig. 3. Loading direct to rail with a modified 'Lobstar' at Wyalong, N.S.W.

COSTS OF BUNKER STORAGE

While further work needs to be done in respect to the costing of different types of storage and its utilisation, there is increasing evidence that the bunker-type storage now in use may be more economical to use, taking account of all costs--capital and operating, than traditional storage designs.

For example, a PVC bunker-type storage of approximately 30,000 tonnes capacity would cost A$7.82 per tonne to utilise, including all costs of inloading and outloading, if the storage is used annually. However, the equivalent costs of storage in steel bins erected at present costs would amount to at least A$20 per tonne capacity including capital charges and amortisation.

CONCLUSION

The storage, handling and fumigation techniques which have been outlined, using PVC-covered bunker storages, have provided the basis for a completely new grain handling system in New South Wales. The system is not only very economic, but offers a degree of flexibility in grain handling which is well matched to the fluctuating demands placed upon a bulk handling authority.

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AIRTIGHT STORAGE OF WHEAT IN A P.V.C. COVERED BUNKER

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ABSTRACT

Wheat of 11.4% moisture content was stored in an airtight structure formed by a polyethylene liner at its base and with a UV-resistant P.V.C. sheet over the surface. The wheat bulk of 15,566.5 t was supported by 2 m high earth banks. The oxygen concentration fell to 6% and the carbon dioxide concentration increased to 9% within 3 months. Insects were present only in the part of the upper layer where the wheat moisture content and high germination and baking quality was preserved. Wheat damage attributed to insects was estimated at 0.15%, and that to moulds at 0.06%. The P.V.C. liner remained in good condition and retained its elasticity, and no rodent damage was detected.

INTRODUCTION

Emergency or temporary storage facilities are frequently needed when very large crops are harvested following unusually favourable growing seasons, or in areas where buffer stocks are required yet permanent storage structures are lacking. This need has arisen after several recent large harvests in Israel so a trial was conducted to examine the suitability of bunkers for emergency storage.

The above ground bunker is an Australian adaptation of the previous underground pit type of bunker (Woolcock and Amos, 1976: Holley, 1979) which used the principle of hermetic storage to preserve the grain. It was lined with plastic sheeting, and wheat was loaded on to this and was then covered first by a top liner and then by a layer of soil. This final layer provided mechanical protection and also served as insulation. However, it added to the labour costs and considerable skill was required to avoid damage to the top sheet. The storage conditions maintained during the Australian trials and the final grain quality justified futher trials in Israel with appropriate modifications to suit local conditions.

This trial was conducted in southern Israel, in an area bordering on the Negev desert, which has long hot summers, mild winters and a low winter rainfall of 200 to 300 mm. The aims were to evaluate: (a) the airtightness achieved with the plastic liners, (b) the climatic and biotic conditions in the bunker and, (c) the grain quality and insect damage at the end of storage.

MATERIALS AND METHODS

The bunker site measured 50 x 150 m and was bordered on three sides by ramps of earth taken from both inside and outside the site with the fourth end left free for loading wheat. The ramps were 2 m high and 8 m wide at their base, with angles of slope permitting drainage of rainwater away from the outer sides. Before loading, the floor and ramps were lined using 250-μ polyethelyene sheeting with an overlap and fold-over at the edges to obtain a continuous underliner.

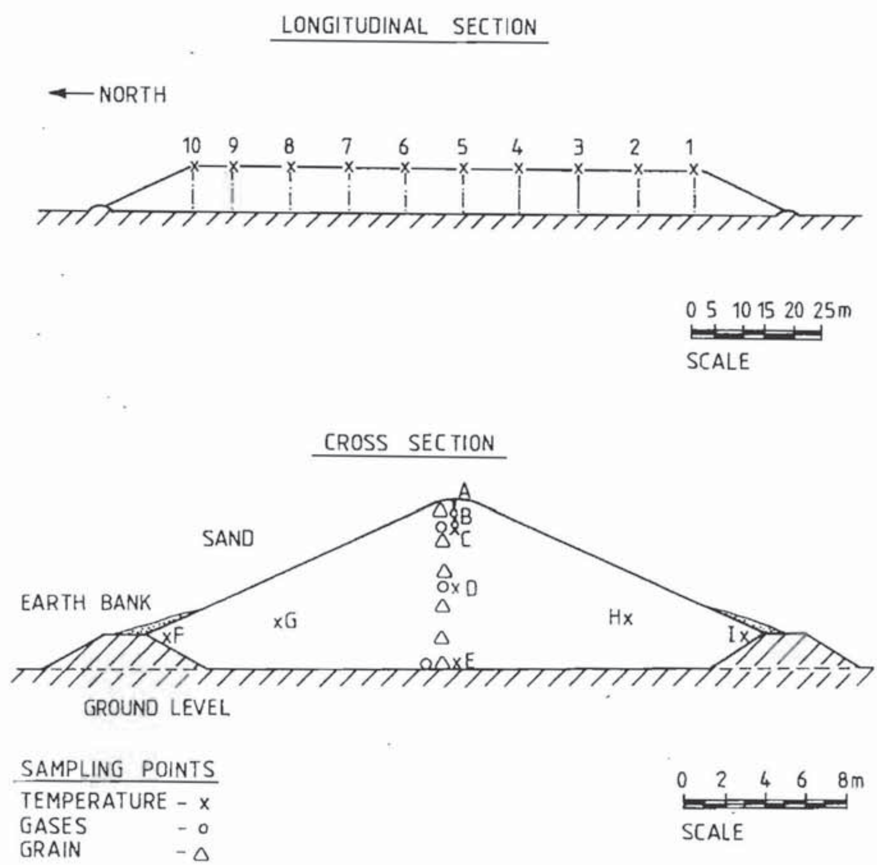

FIGURE 1

POSITION OF TEMPERATURE AND GAS MEASUREMENT, AND GRAIN SAMPLING POINTS IN THE EXPERIMENTAL SILO CONTAINING 15,566.5 TONNES OF WHEAT.

The overliner was 830-μ white polyvinyl chloride (P.V.C.) with an UV inhibitor (Haogenplast 5.196). The two liners came into contact on the outer bank of the ramps where they were folded over and covered with earth to provide a hermetic seal.

The bunker was loaded with wheat (var. Miriam) from the 1980 harvest and was transported to the site directly from the fields or after being cleaned on the farm. A total of 15,566.45 tonnes was loaded into the bunker in 28 days, and during this period 209 wheat samples were taken. The grain bulk was approximately 8.5 m high at the apex, and was contained between the earth ramps (Fig. 1).

Thermocouple cables for temperature measurements (90 points) and tubing for gas measurements (30 points) were placed in the bunker during loading at the points shown in Fig. 1. The cables and tubing were bundled together and directed out of the bunker at the junction between the over and underliner, so that ten measurement stations were set up along one of the ramps. Temperature and gas measurements were made approximately every 2 weeks.

To determine airtightness, a series of lowered pressures were applied by sucking air from the bunker at different flow rates and recording the pressure differential from ambient (Sharp et al., 1976; Navarro et al., 1978).

Sixty grain samples (1-kg each) were withdrawn from the bunker using a suction-sampler from ten equidistant sampling areas along the axis at 6 depths as noted in Fig. 1 on 10 occasions during the storage period. Moisture content of the grain was determined using a capacitance moisture meter, (Motomco Model 919). Insects were removed from samples by sieving the grain through a 10-mesh sieve with 2x2 mm holes. Germination was determined by the ISTA method (Annon., 1969). Baking tests were performed by a standard method that resembles closely the commercial method of bread making in Israel (Calderon et al., 1970).

Unloading was carried out after 15 months of storage. A 1-kg sample was taken from each truck (total, 585 samples) during the 3-week unloading period. Each sample was examined for insect infestation and moisture content. From every ten consecutive samples, two 1-kg composite samples were formed, using a Boerner divider. One of the composite samples was used to evaluate weight loss by the count and weigh method (Adams and Schulten, 1978) and for germination studies. These samples were also incubated in the laboratory at 26°C and re-examined after one month for insect development. The other composite samples were used to make up 13 samples destined for baking quality analysis.

RESULTS

Ambient conditions

Daily temperature fluctuation were especially large during the summer when the average maximum in both years of storage reached 37°C. Ambient temperatures fell progressively from October until January, when the average maximum temperature was 17°C. Relative humidities were high at night exceeding 90%, and between 30% and 40% during the day. Short periods of very low minimum relative humidity of around 20% were recorded in spring and early summer.

Grain temperature

The average temperatures at different depths in the grain bulk over the whole storage period from all 10 sampling stations are given in Fig. 2. Initially grain temperatures were uniform at ca 30°C. At the surface of the bulk the temperatures were obviously influenced by ambient conditions and a considerable drop was observed from ca 25°C in mid October down to 16°C in January. Deeper in the bulk, from 1 m to the base of the bunker there was a progressive lag in temperature when compared to ambient. Temperatures were also more uniform in the middle of the bulk over the 15 months of storage. During the final 3 months of storage the lowest temperature was recorded at the base of the bunker. The steady fall of temperature at the base even in the second summer reflected the insulating influence of the grain mass as well as the low ground temperature.

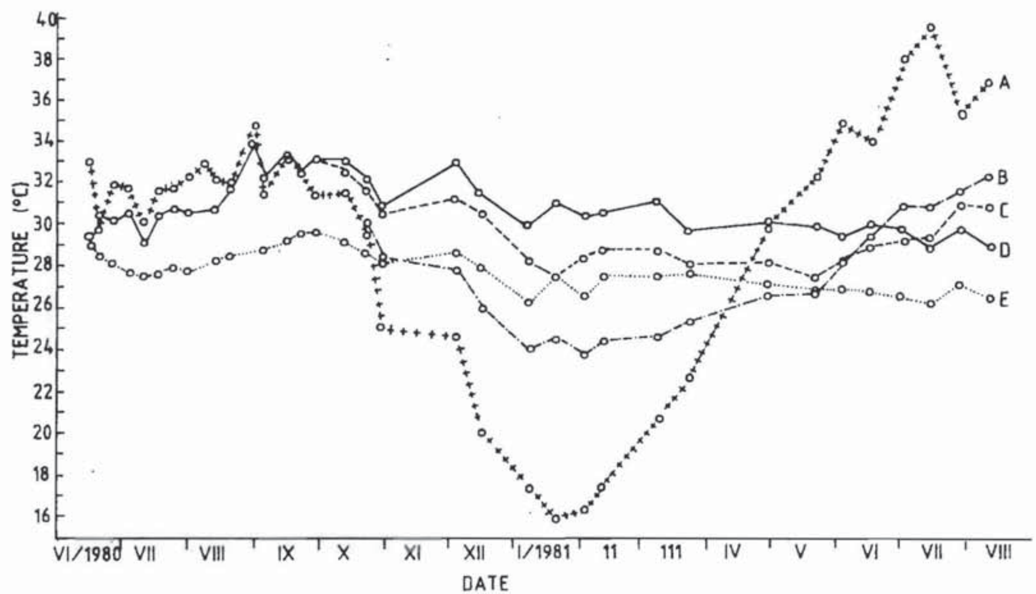

FIGURE 2

TEMPERATURES AT 5 DEPTHS IN THE WHEAT BULK PRESENTED AS THE AVERAGES FOR EACH DEPTH FROM TEN STATIONS ON EACH OCCASION OVER THE WHOLE STORAGE PERIOD. REFER TO FIGURE 1 FOR DEPTHS OF SAMPLES.

Wheat moisture content

The average m.c. of 209 wheat samples taken during inloading of the bunker was 11.4% $\pm$ 1.0% S.D. (range 8.7 to 14.3%). The highest m.c. averaging 12.6 $\pm$ 0.9 S.D. was found during the last 2 days of loading. This wheat was placed in the upper layers of the northern part of the bulk at stations 9 and 10 (Fig 1).

A low m.c. was maintained in the deeper layers of the bulk, but there was significant and progressive increase in parts of the surface, at stations 5, 6, 9 and 10 along the apex ridge of the stored wheat. The average m.c. of samples taken during unloading of the bunker was 11.4 $\pm$ 0.6% S.D.

Changes in atmospheric gas composition

The concentrations of oxygen (O_2) and carbon dioxide (CO_2) in the bunker expressed as averages of the 30 values taken on each occasion are shown in Fig. 3. The changes in gas composition reflect the total of all respiratory process in the grain bulk during storage. The grain was dry and no insecticidal treatment was applied before loading although insects were present. The rapid fall of O_2 concentration to 6% in 3 months of storage can be attributed to the insects, as can the rise of CO_2 concentration to 9%. A minimum O_2 concentration of 5% was reached in December, and this was followed by a gradual increase to 8% by the end of March when the CO_2 concentration had fallen to 8%. During the balance of the storage period the O_2 concentration decreased and the CO_2 concentration increased.

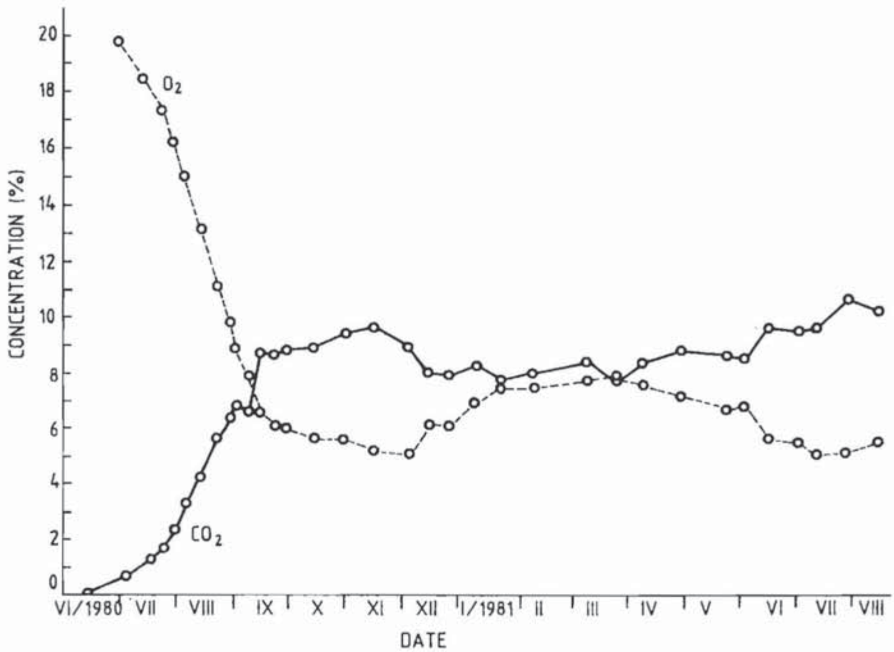

FIGURE 3

OXYGEN AND CARBON DIOXIDE CONCENTRATIONS IN THE WHEAT BULK PRESENTED AS THE AVERAGES OF ALL SAMPLING STATIONS ON EACH OCCASION OVER THE WHOLE STORAGE PERIOD.

Insect presence

Insects were not sieved from any of the 209 wheat samples collected during inloading. However, after incubation for 4 weeks at 26°C, *Oryzaephilus surinamensis* (L.) and *Sitophilus oryzae* (L.) were found in 7 of the 209 wheat samples examined.

Insects were found in the bulk during storage only in samples taken from the upper layers at stations 8, 9 and 10. The most abundant species were *Tribolium castaneum* (Herbst), *Rhyzopertha dominica* (F.) and *Oryzaephilus surinamensis*.

The infestation which accumulated at the surface layer was controlled with a spot fumigation using 600 tablets of phosphine each containing 1 g PH_3. This fumigation was carried out in April, 1981 and was judged to be efficient since no live insects were found in samples taken at the end of storage.

Germination tests

Analysis of the wheat samples taken during loading indicated an average of 97% germination. The only samples in which a loss of germination was detected during the storage period were those from the upper layers of stations 9 and 10. In tests carried out on 65 composite samples taken during unloading the average germination was 91%.

Loaf volume

Measurements of the loaf volume of bread made from flour prepared from wheat samples taken during the storage period were based on pooled samples which represented groups of stations and different regions of the bulk. The loaves baked from wheat from the upper layers of stations 9 and 10 were adversely affected. This effect on baking quality was found to a depth of 3.2 m at these stations, where as at the other locations in the bulk the baking quality was preserved at an acceptable level for the local Miriam variety of wheat. The average loaf volume from composite samples taken during unloading was 2252 ml $\pm$ 18 SE as compared with 2263 ml $\pm$ 58 SE for the same variety of 1980 harvested wheat stored in conventional silos.

DISCUSSION

Insect presence

The storage method examined in this trial utilised the principle of airtight or hermetic storage (Hyde *et al.*, 1973) which was provided by the P.V.C. and polyethylene liners. Under these conditions the depletion of the O_2 and increase of the CO_2 in the container atmosphere proceeded to a level that controlled insects before they caused economically significant damage to the grain.

Calculations made on the data given in Fig. 3 show that over the period from the start of the observations until mid-September, the daily O_2 reduction rate was 0.185%. From the analysis performed to determine the degree of airtightness (Navarro *et al.*, 1978), it was calculated that the storage structure allowed the entry of O_2 at a rate of 0.494%/day. Taking these together, the estimated average O_2 consumption was 0.679%/day. This is equivalent to a total consumption of 51.6 m^3 O_2/day. Assuming the average daily O_2 consumption by insects to be about 103 μl O_2/ insect (Birch, 1947, Chaudhry and Kapoor, 1967), 51.6 m^3 O_2/day represents the consumption of 500×10^6 insects. The estimated population calculated from samples taken during September 1980 was about 352×10^6 insects. The discrepancy between these two figures is sufficient to require an additional source such as respiration of slightly damp grain for additional O_2 consumption. However, most of the insect infestation was found at the surface of the bulk. The average number of live and dead insects found in the deeper layers of the bulk was 4.6/kg wheat, and the average number of live insects was 1.3/kg wheat. On the basis of the number of insects found in the bulk, the dry matter consumed by insect activity was calculated to be 0.0133%/month. Live insects were found until the O_2 fell to its lowest level, after which more dead insects were found in samples taken below the surface layer. Therefore, for the period ending in late September 1980, or the first 4 months of storage, the estimated dry matter consumed by insects was 0.0532%. Fig. 3 shows that during the remaining storage period, equilbrium was reached between O_2 entry and the O_2 consumed by the insects. Then, for this period, the dry matter consumed by the insects would be ca 0.00967% per month or, for the 11 month period, ca 0.1064%. Dry matter loss that could be attributed to insect activity was estimated to be ca 0.1596% for the entire storage period.

Weight loss caused by insect activity was estimated to be 0.1472% from wheat samples taken during unloading (Table 1). This value may be compared with the calculated figure of 0.1596% based on dry matter loss derived from metabolic activity of the insects in the bulk.

Insect mortality recorded during the observation period was in accordance with the O_2 depletion recorded in Fig. 3. In work with *T. castaneum* and 30°C Calderon and Navarro (1979) found that a combination of 3% O_2 and 10% CO_2 caused high mortality of adult beetles at 57% relative humidity after a 5 day exposure. The lowest O_2 concentration obtained in our tests was 5.1% (Fig. 3), but, the effect of the adverse atmospheric composition obtained is dependent on length of exposure. It was evident from the dead insects found in the samples that the atmospheric composition obtained resulted in adequate control of insects in the deeper layers of the bulk. However, at the surface layer of the bulk where the wheat moisture content was considerably higher some insects survived. The effect of interdependence of atmospheric gas compositions and humidity on insect mortality has been demonstrated by Navarro, (1978). The composition of gas samples taken from the surface layer did not differ from that of samples taken at depth within the bulk. Therefore, insect survival at the surface layer could be attributed to the influence of high humidity in this region.

Wheat moisture content

Wheat at 11.4% m.c. (average of the bulk) is in equilibrium with 60% relative humidity (Pixton and Warburton, 1971). At station No. 10 the wheat m.c. was high from the start of the storage period (12.6%) and by September, 1980 had increased to 14%, which would be in equilibrium with 74% relative humidity. A drop in temperature to 28°C would be sufficient to cause this increase in relative humidity, (Navarro and Calderon, 1982), while a temperature drop to 23°C was necessary to cause water condensation on the surface layer of station No. 10. Temperatures lower than 23°C were recorded in December, and the most marked m.c. increase coincides with these low temperature measurements of this same period. From this discussion and the analysis of the possible contribution of insects to increasing grain moisture, it seems very probable that the major cause of moisture deposit on the surface layer of the bulk was steep temperature gradient in the surface layer. This was responsible for moisture migration to the cooler surface layer of the bulk.

Analysis of the samples taken during the unloading process revealed that the final average m.c. of the grain was 11.4% (Table 1). The fact that the average grain m.c. remained unchanged so well may serve as evidence that all the above mentioned moisture increase at the surface took place endogenously in the airtight structure.

Table 1 - Assessment of damage caused to wheat during the 15-month storage period.

		Initial	Final
Weight of wheat (tonnes)		15,566.450	15,537.155
Average moisture content (%)		11.41	11.41
Measured total loss	tonnes	0	29.295
	%	0	0.1882
Estimate of mould-damaged grain unfit for human consumption*	tonnes	0	9.0
	%	0	0.0578
Calculated average weight loss due to insect activity**	tonnes	0	22.914
	%	0	0.1472
Calculated total damage	tonnes	0	31.914
	%	0	0.205

* Weighing the damaged grain was not possible; 300 bags, each containing about 30 kg mould-damaged grain, were removed.

** Based on count and weigh method.

Preservation of wheat quality

Germination, baking quality as determined by loaf volume, and insect-damaged grains were used as criteria for quality determination. High germination and baking quality were preserved for most of the bulk. The damaged portions of the bulk were found at the surface layer, at stations 9 and 10, and down into the bulk to a depth of 3.2 m at stations 10. The major cause of reduced quality in this portion of the bulk was the high m.c. of the wheat. The estimated amount of wheat with reduced quality was approximately 30 tonnes. However, from tests carried out during unloading the amount of grain unfit for human consumption was determined. The amount of grain discarded was ca 9 t (Table 1). Samples taken from this portion of grain were analyzed for mycotoxin production. Analysis for the presence of aflatoxins and ochratoxin using thin layer chromatography method (AOAC, 1980) gave negative results.

Resistance of the P.V.C. liner

Analysis of the P.V.C. liner taken from the top of the bunker silo after 15 months of exposure to weather conditions showed that its original elasticity and resistance to tear were preserved. Although rodent activity around the bunker was recorded, and some poisoned field rodents were found around the bunker, neither gnawing of the liner nor penetration into the bunker was observed. The inability of rodents to damage the liner during 15 months of storage may be attributed to the smooth texture of the liner surface. In previous experiments, rodent damage was recorded mainly at the folds of the liner (Navarro and Donahaye, 1976). A possible explanation for this phenomenon could be the fact that rodents need to have a projecting end of the plastic material (such as the edge of the fold) to start gnawing However, the absence of rodent damage requires further investigation.

Assessment of total damage

A summary of different measurements and calculations is given (Table 1) to assess the total damage caused to the wheat stored for a period of 15 months. Although all the trucks during the loading and unloading process were weighed on the same scale near the storage site, the accuracy of the scale was not determined.

The average m.c. remained unchanged (Table 1), and therefore the measured total loss was attributed to biological and physical damage. Thus, the measured total loss was 0.1882%. The mould-damaged grain unfit for human consumption was estimated at ca 9 t. This, together with the calculated weight loss due to insect activity, was considered as the calculated total loss, which amounted to 0.205% of the initial weight of the bulk. There was a difference of 0.0168% (ca 2.6 t) between the measured (0.1882%) and the calculated (0.205%) loss. During the unloading process great care was taken to avoid mixing sand with grain at the edges of the uncovered base of the bunker. However, this procedure was not always successful, and some earth or sand was mixed with the grain. The difference between the measured and calculated amount of grain could be attributed in part to grain mixed with sand, which increased the actual weight of the unloaded grain.

CONCLUSIONS

At a degree of airtightness obtained with the above described bunker storage method, insect activity caused a reduction in O_2 to 5.1%, and an increase in CO_2 of 9.8%. The atmospheric composition obtained as a result of the airtightness of the structure provided an adequate control of insects and prevented damage due to insects.

An increase in moisture content, caused mainly by temperature gradients, was observed in the surface layer of the bulk. Wheat in the surface layer that entered storage with an initial moisture content of 11.4% was preserved adequately, while wheat with $\geq$ 13.5% moisture was considerably damaged. Visible mould was noted on high-moisture wheat and damage was accompanied by a reduction in germination power and in baking quality of the wheat.

Loss derived from insect activity was estimated at 0.1472%, where as mould-damaged grain unfit for human consumption was estimated at 0.0578%. Thus, the calculated total damage for the 15 month storage period was 0.205%.

The P.V.C. liner remained well preserved throughout the storage period and rodent damage was not detected.

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SESSION 10.

PHYSICAL METHODS

Papers by:

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THE DEVELOPMENT OF A CONTINUOUS-FLOW FLUIDIZED-BED HIGH-TEMPERATURE GRAIN DISINFESTATION PROCESS

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ABSTRACT

Laboratory-scale experiments on a continuous flow high temperature disinfestation plant demonstrated the technical viability of the process, and indicated its economic soundness. In order to determine problems of scale-up, to ascertain realistic cost data and to illustrate how such novel equipment may be easily integrated into the grain handling system, a 50 tonne per hour plant was installed at Dunolly, Victoria. The plant was extensively instrumented to determine its entomological and thermodynamic performance. Early results show that heating grain to 65°C or more results in the total kill of all developmental stages of insects. Furthermore, the process causes no detectable change in either grain quality or moisture content. Total energy costs have been measured to be 62 cents per tonne of grain treated using LPG as the heating source; if natural gas were to be used the cost would be 46 cents per tonne. The plant is easily integrated into the existing grain handling system. Husk and straw entrainment in an air recirculation duct proved to be an unexpected problem, but a simple modification to the plant will overcome this.

INTRODUCTION

Fluidized bed heating offers a rapid and residue-free method of disinfesting grain. In continuous flow form, the process relies on heating grain to temperatures that are lethal to all developmental stages of insects within the grain and then cooling it rapidly to safe storage temperatures. Heating in fluidized beds was chosen because the good mixing of solids allows high inlet air temperatures to be used, yet individual grains are not overheated. Large scale fluidized beds are commonly found in the process industries and scaling up disinfestors to handle the high grain throughputs at export and large subterminals should not present problems. Furthermore, fluidized beds are simple and rugged and their operational stability renders them ideal for integration into the grain handling system.

LABORATORY EXPERIMENTS

Initial experiments were aimed at demonstrating the entomological efficacy of the process, and establishing heating regimes that result in no detectable grain damage yet are lethal to insects. It was shown that 10kg batches of wheat were disinfested of immature stages of *Rhyzopertha dominica*

(F.), *Sitophilus oryzae* (L.) and *Sitotroga cerealella* (Oliv.) when exposed to inlet air temperatures of either 80°C, 70°C or 60°C for periods of 4, 6 and 12 minutes respectively. Such exposures gave respective maximal grain temperatures of 65°C, 62°C and 59°C and did not impair baking quality or germination, and the grain moisture content was not changed appreciably (Dermott and Evans 1978).

Further experiments with a batch heater were carried out in which the influence of varying bed depth and inlet air temperature on the mortality of *R. dominica*, the most heat tolerant species, was examined. The results clearly indicated that insect mortality was related to a heat dosage factor incorporating both temperature and time components (Evans & Dermott 1981).

The importance of the heat dosage factor was highlighted in an experiment in which wheat was heated to 56-58°/C and then allowed to stand unfluidized for up to 15 minutes before cooling. The soaking period increased the insect mortality from about 50% to greater then 99.9%.

Experiments were also made with a continuous-flow disinfestor. The heating chamber had an area of 200 x 400 mm and grain flowed through it in a zig-zag horizontal path. Baffles were fitted to reduce the spread about mean residence time and hence approximate the plug-flow conditions of the batch systems. The cooling chamber had an area 100 x 360 mm and was fitted with a water spray for evaporative cooling. Repeated trials showed that grain infested with *R. dominica* flowing at 360-500 kg h^{-1} at depths of 100-250 mm could be disinfested by exposure to air inlet temperatures of 80-90°C for periods of 2-5 min. that provided maximal grain temperatures up to about 65°C (Evans, Thorpe and Dermott (in press)).

50 TONNE PER HOUR PILOT-PLANT

The laboratory-scale experiments on a continuous-flow, fluidized-bed, high-temperature, disinfestation plant demonstrated the technical viability of the process, and indicated that it was likely to be competitive in cost with pesticide treatments commonly used. However, it was clearly important to build a plant capable of handling commercial quantites of grain to determine possible problems of scale-up, to ascertain realistic cost data and, together with the grain handling industry, determine how such novel equipment may be integrated into the existing grain handling system.

Good progress in achieving these objectives has been made with a nominally 50 tonne per hour plant installed by the Grain Elevators Board of Victoria at Dunolly, near Bendigo.

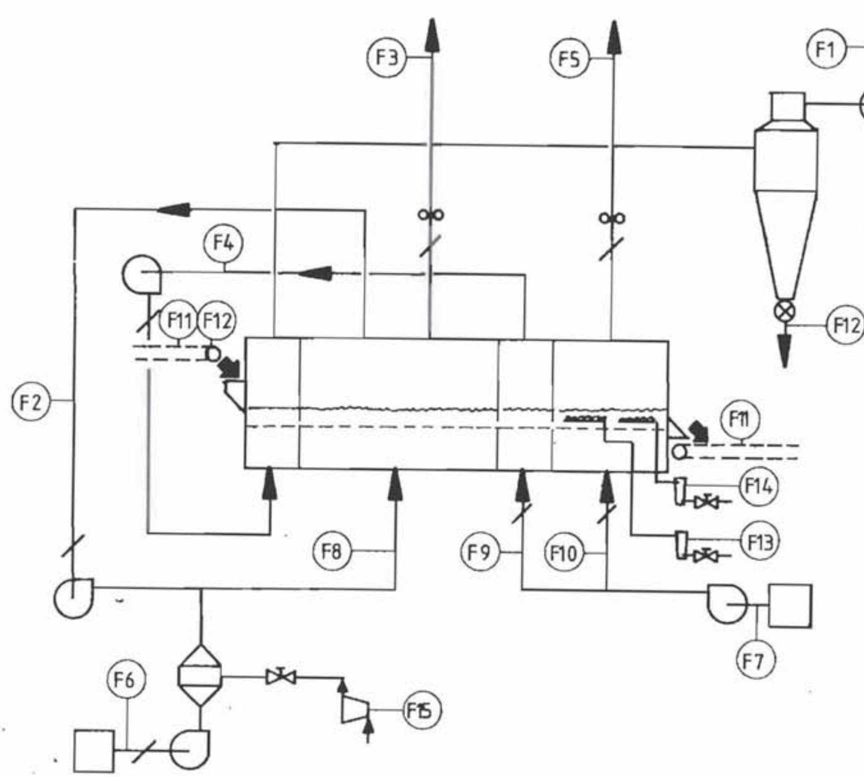

Figure 1. Process flow sheet of 50 tph high temperature disinfestation plant showing process flow streams referred to in text.

The plant consists of four chambers, 1 a preheating and de-dusting section, 2 a heating chamber, 3 a cooling section supplied by atmospheric air and 4 an evaporative cooling stage. The process flow sheet is shown in Figure 1. The grain stream, F11, and any chaff and dust associated with it, F12, enters the pre-heating chamber; this chamber also cleans the grain and the dust is subsequently separated from the fluidizing air by means of a cyclone. From the pre-heating stage the grain flows into the main heating bed in which it is heated to disinfestation temperatures before it is partially cooled in the heat recovery stage and then finally cooled in an evaporative cooling section. Cooling air for the process, F7, is divided into two streams: F10 is fed to the evaporative cooling chamber before being expelled to atmosphere by means of a vent stack; the other component of the cooling air, F9, is fed to the heat recovery section where it is heated by the hot grain and then, as stream F4, enters the pre-heating and de-dusting section. Hot air from the main heating chamber is re-circulated as a heat economy measure whilst the exhaust gases are vented as stream F3, and the make-up combustion air, F6, enters the burner. The fuel for the process, liquefied petroleum gas, enters the burner, stream F15, through a turbine gas meter. F13 and F14 are the cooling water streams.

In order to kill any free living insect stages that may be removed from the grain in the de-dusting stage, the dust leaving the cyclone is passed through an entoleter that kills the insects by impact.

The plant was designed to be very simple and safe to operate. The plant instrumentation housed in a control room conforms to the high standard traditional in the chemical and process industries and the key process variables such as plenum and free board pressures, air temperatures leaving the burner and entering the heating bed and the heated and cooled grain temperatures are clearly displayed. Either the heated grain temperature or the heating air temperature may be automatically controlled, and investigations are being carried out to determine which of these is the more suitable.

If the heated grain temperature should vary from its set point by more than $\pm 3^{\circ}C$, audible and visual alarms are activated. However, if the grain temperature should deviate by more than $10^{\circ}C$ indicating either over-heating or poor insect control the plant is automatically shut down. An explosion hatch on the de-dusting section is provided for rapid pressure release in the event of explosion. Should a rise in pressure above 2kPa occur in the de-dusting section a pressure sensor activates a fire extinguishing system and the fluidizing chambers are sprayed with water at a rate of 350 litres per minute. Temperature sensors are located in the de-ducting section exhaust and recirculation ducts and should the temperature at either of these sites exceed $100^{\circ}C$ an audio and visual alarm is activated; should either temperature exceed $150^{\circ}C$ the fire extinguishing system is also activated.

The CSIRO has augmented the standard plant instrumentation so that comprehensive measurements may be taken that allow the entomological efficacy of the plant to be determined and to enable us to accurately determine the thermodynamic states and flow rates of the process streams. Details of the CSIRO instrumenation system are given by Thorpe et al (1982).

PRELIMINARY RESULTS

Results obtained from the plant to date are encouraging and they vindicate the laboratory experiments which showed that all developmental stages of insects are killed if the grain is heated to temperatures of $65^{\circ}C$ or more. In the first trial 50 tonnes per hour of wheat were heated to $60^{\circ}C$, $65^{\circ}C$ and $70^{\circ}C$ with a total residence time in the unit of about 4 minutes. Complete kills of *R. dominica* were obtained except when the wheat was heated at $60^{\circ}C$. It must be stressed that our results are preliminary but measurements show that there is no detectable change in the moisture content of the grain as it passes through the unit. Measurements also indicate that there is no change in grain quality as determined by germination; this result is in accordance with laboratory studies (Ghaly and Taylor, 1982).

The fluidizing air velocity in the unit was typically greater than 2 m/s and in order to heat 50 tonnes per hour of wheat to 65°C the heating air temperature in the plenum was found to be 100°C, exactly the design figure. Initial energy consumption figures for the plant operating with a grain throughput of 50 tonnes per hour are respectively 2 kWh and 20 kWh of electrical and gas energy per tonne of grain treated. On the basis of 7 cents per kWh for electricity and LPG priced at 34 cents per kg the total energy cost to treat one tonne of wheat is 62 cents. If natural gas were to be used, as is likely at seaboard terminals, then the energy cost would be 46 cents per tonne of grain treated assuming a gas tariff of 0.45 cents per MJ. The total capital cost of the plant, including the provision of services was $400,000.

Presently the plant is operating in a sub-optimal mode in so far as the air flow rates are severely throttled by dampers, hence there is scope to reduce the electricity consumption by slowing down the fans. The electrical energy cost per tonne would also be reduced if the throughput of the plant were to be increased. To date the highest grain throughput achieved at Dunolly is 70 tonnes per hour, which is the maximum capacity of the grain handling system presently servicing the unit. There is no apparent reason why the grain throughput cannot be increased further.

The plant performance is very stable as evidence by the fact that the heated grain temperature remains steady for prolonged periods without any operator intervention, even when the system is under manual control. This leads to the conclusion that once such a plant is properly integrated into the grain handling system it will operate with minimal attention, and would not require a full-time operator assigned exclusively to the plant.

A serious, but tracable problem that has emerged during the trials is the blocking of the distributor plates with husk and straw. This is most serious in the heating chamber where husks and straw are carried over in the air re-circulation duct. For the purposes of the entomological trials the problem was overcome by pre-cleaning the grain. This would not be an economically feasible system for commercial use. A more elegant method of eliminating the unwanted husk and straw would be to insert a cyclone in the re-circulation duct.

FUTURE RESEARCH

In the immediate future, work will be aimed at eliminating the entrainment of husk and straw in the air re-circulation duct, and investigating the range of operating conditions which yield complete insect mortality. These latter experiments will allow the plant operation to be optimized. it is also planned to carry out further trials to confirm that wheat is in no way damaged by the disinfestation process.

Work is also aimed at evaluating alternative hardware for heating grain such as spouted beds, which are a special form of fluidized bed, and pneumatic conveyors which simultaneously convey and heat the grain, and minimizes dust extraction problems. Fundamental studies of heat, mass and momentum transfer in high temperature disinfestation processes will be made.

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Niro Atomizer Pty. Ltd. were the project engineers for the disinfestor.

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GRAIN REFRIGERATION TRIALS IN AUSTRALIA

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ABSTRACT

A summary of the results of trials commencing in 1967 using mainly wheat in bulk grain storages ranging in capacity from 500 to 15 000 tonnes shows the feasibility of various refrigeration strategies and their costs and effectiveness against grain infesting insects. Air flow through the bulk in an upwards direction brought insects to the surface where they could be treated readily with an insecticide if and when necessary. Partial filling of horizontal storages is seen to have an important influence on duct layout design. Insulation with rigid polyurethan foam, spray-applied to the outside of storages and protected with an appropriate coating, has to date been satisfactory, apart from easily repairable minor damage by birds and rodents. The refrigeration plant developed on a unit module concept is shown to have other applications in the industry. These will assist in the gradual introduction of the technology.

INTRODUCTION

The motivation for conducting trials using refrigerated air to cool bulk stored grain in Australia arose form the industry's recognition that total reliance on chemical control of insect infestation is vulnerable to insect resistance, objections by markets and industrial bans, and the belief that cooling with untreated atmospheric air would not provide an adequate degree of insect control. Overviews of the role of various complementary physical methods of insect control in Australia have been given by Elder (1978) and Evans (1981). As part of an overall strategy, a research programme on refrigeration of grain commenced in the CSIRO Division of Mechanical Engineering in 1967 with fundamental studies of heat and mass transfer in porous hygroscopic materials (Sutherland *et al.*, 1971), computer simulation of cooling system performance and a field trial in Queensland involving non-insulated silos and non-recirculation of cooling air. Subsequently a series of trials was conducted in insulated silos with recirculation, the objective being to develop a commercial prototype system for shed type storages which account for over 60% of Australia's storage capacity. It became evident that there could be a number of different applications for refrigeration in the industry, and that it would be possible to design the plant with a high degree of standardization to meet the various specifications and criteria.

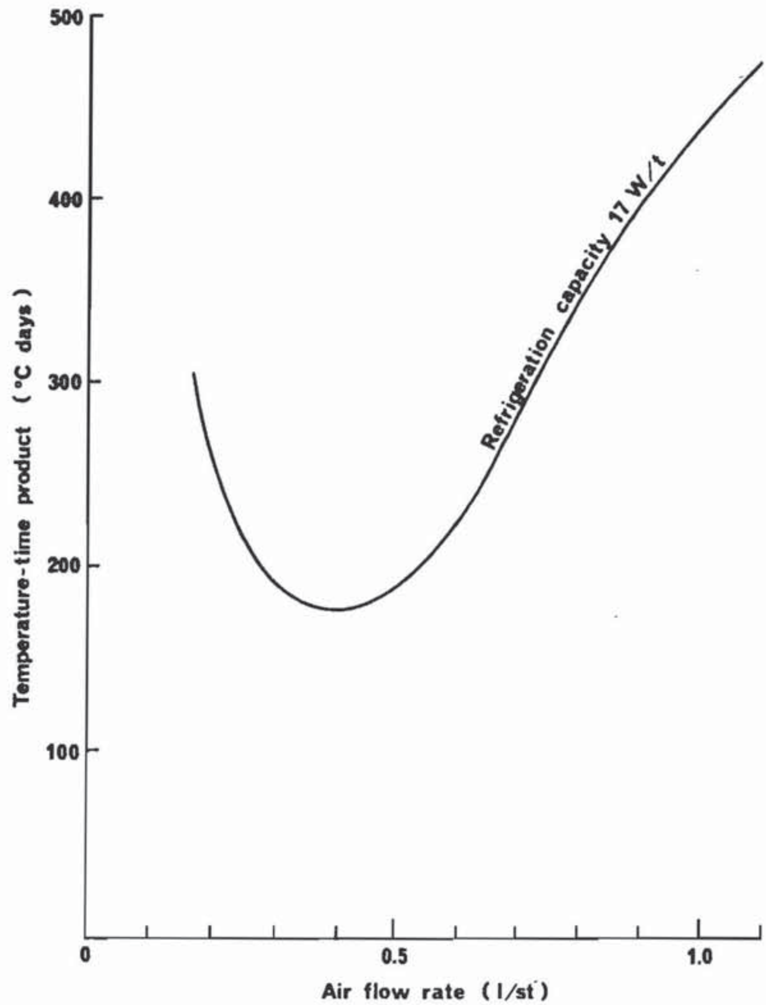

Fig. 1 Variation of insect population growth potental (^{o}C days above threshold) with refrigerated air flow rate showing existence of an optimum air flow for a given cooling capacity in a sub-tropical climate (Brisbane).

REFRIGERATED AERATION WITHOUT RECIRCULATION IN NON-INSULATED SILOS

Initial trials involved the placement of a refrigeration coil upstream of the aeration fan blowing atmospheric air into the silo.

Computer simulation of an aerated vertical silo of wheat with a refrigeration coil of fixed capacity revealed there was an optimum air flow per unit mass of grain under given climatic conditions. As a measure of the potential for the growth of grain infesting insect populations, the temperature-time product above a nominal breeding threshold temperature based on the data published by Howe (1965) was used as a criterion for cooling performance. Low air flow rates resulted in high temperature-time products in the grain last to be traversed by the cooling wave (or front). Higher air flow rates increased the speed of the cooling wave but at the expense of higher grain temperatures behind the wave some times above the breeding threshold. Excessively high air flow rates therefore also led to high temperature-time products as shown in Fig. 1. The optimum air flow rate was taken as that which minimized the temperature-time product. This value was used as a basis for design.

The plant used for the first trial at Dalby, Queensland in collaboration with the State Wheat Board was described by Sutherland *et al.*, (1970). It incorporated an automatically adjusting damper to vary the air flow to keep the temperature of the air entering the grain just low enough to arrest population growth behind the cooling wave.

The results of three trials at Dalby are summarized in Table 1. The effect of the air flow being too low is clearly shown by the much larger infestation level at the end of Trial 2. The low air flow and consequent inadequate cooling at the walls of the silo was concluded to have supported insect population growth after the initial cooling. Although insect control performance was good in the other two trials, it was concluded that insulation or direct cooling of the walls would be necessary to render all parts of the bulk inhospitable to insects and to achieve the very high level of hygiene demanded by export regulations.

REFRIGERATED AERATION WITH RECIRCULATION IN INSULATED SILOS

Trials at Brookstead, Queensland

A 5400 tonne capacity wheat silo at Brookstead, Queensland was insulated externally with 50 mm thickness of spray-applied rigid polyurethane foam, with the object of maintaining all parts of the bulk of stored grain to be refrigerated below 15°C, the insect population growth threshold.

TABLE 1
SUMMARY OF RESULTS OF THREE REFRIGERATED AERATION TRIALS WITH WHEAT IN 580 TONNE VERTICAL CONCRETE SILOS 6.4 m DIAMETER AND 23 m HIGH AT DALBY, QUEENSLAND

Trial No. Year	1 1967/68	2 1967/68	3 1968/69
Average air flow rate (L/s per tonne of silo capacity)	0.45	0.15	0.6
Compressor rating (shaft W/tonne of silo capacity)	3.8*	1.3*	5.1
Quantity of Wheat (t)	594	594	570
Initial grain moisture (% wet basis)	10.8	10.7	11.1
Final grain moisture (% wet basis)	12.5	10.9	10.9
Storage period (months)	10	10	10
Initial grain temperature (^{o}C)	26.4	33.0	28.3
Initial cooling to 15^{o}C (months)	0.6	2.1	0.6
Final grain temperature (^{o}C)	10.4	12.1	15.5
Total operating time (hours)	5600	5600	1900
Energy used (kWh/tonne of silo capacity)	34	11	18
No. of live insects at inloading	0	0	0
Quantity of grain sieved (kg)	76	72	82
No. of live insects at outloading	0	35	3
Quantity of grain sieved (kg)	214	94	91

*Proportioned in accordance with air flow rate

A series of trials began at Brookstead in 1973. In the first, the existing aeration system was used to blow air throught the grain via conventionally laid out perforated distribution ducting. Return ducts were connected from the headspace to the two aeration fans to recirculate the air. The headspace air was cooled via a separate recirculation system comprising a fan and chilled brine cooling coil connected to a commercial chiller set at 70 kW nominal capcaity. The plant is described in more detail by Elder *et al.*, (1975). The results revealed that the air flow through the grain at the bottom of the concrete wall was insufficient for removing heat penetrating the wall insulation and the silo floor to maintain temperatures below 15^{o}C.

The duct layout was modified for the second trial at Brookstead to concentrate air flow at the base of the silo wall after the initial cooling. The peripheral duct system showed that, over the full height of the wall, grain temperatures could be maintained below 15^{o}C. Other features of the system are described by Elder *et al.*,(1976) and the results given are summarised in Table 2.

TABLE 2
SUMMARY OF RESULTS OF THREE GRAIN REFRIGERATION TRIALS IN AN INSULATED 5400 TONNE CONCRETE WALLED SILO 28 m DIAMETER, 9.6 m WALL HEIGHT AT BROOKSTEAD, QUEENSLAND

Trial No. Year	1 1973/74	2 1974/75	3 1975/76
Air flow rate (L/s per tonne of silo capacity)	0.8	0.8	0.9
Compressor rating (shaft W/tonne of silo capacity)	4.2	4.2	4.2
Quantity of grain (t)	2390	5230	2230
Initial grain moisture (% wet basis)	11.0	11.2	11.3
Final grain moisture (% wet basis)	11.4	11.6	11.7
Storage period (months)	6	9	10
Initial grain temperature (^{o}C)	29	27	26
Initial cooling to 15^{o}C (months)	1.7	1.2	1.4
Final grain temperature (^{o}C)	6	5	8
Total operating time (hours)	Continuous operation		
Energy used (kWh/tonne of silo capacity)	22	30	28
No. of live insects at inloading	17	1	6
Quantity of grain sieved (kg)	384	543	255
No. of live insects at outloading	1	0	0
Quantity of grain sieved (kg)	490	863	429

The third trial at Brookstead was conducted using barley, and the enhanced germination and malt yield resulting from storing in a refrigerated silo has led to continued use of the system.

Trials at Lah, Victoria

Mathematical modelling of refrigerated grain silos had progressed to a stage where a design could be specified for a 1700 t steel silo in northern Victoria. Details of the model and results of optimization studies are given by Thorpe (1976). The significant conclusion of the work was that recirculation systems gave much lower annual costs than systems like that at Dalby in which the air from the silo is exhausted to atmosphere. These studies were based on maintaining wall temperature below 15^{o}C and took account of the drying effect of heat flowing through the wall on grain moisture content and the consequent effects on insect behavior. The eco-system modelling of the complete process is barely within the capabilities of modern computers; but Thorpe *et al.*, (1982) have initiated procedures compatible with both the physical and biological aspects.

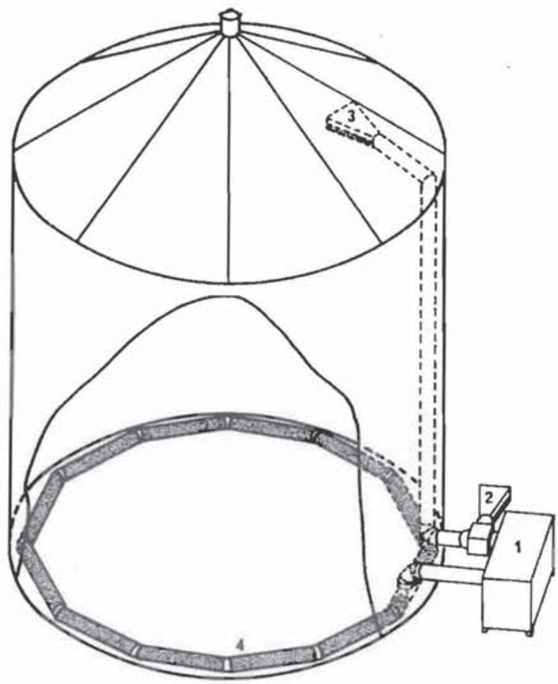

Fig. 2. Layout of ducting in 1700 t capacity insulated silo for grain refrigeration trials at Lah, Victoria.

1. Refrigeration Unit; 2. Atmospheric Air Intake;
3. Return Air Intake: 4. Perforated Air Distribution Duct.

The system at Lah is described by Hunter and Taylor (1980) and comprises a self-contained refrigeration unit with direct-expansion air cooling coil connected to distribution ducting around the periphery of the silo floor and recirculation duct from the headspace as shown in Fig. 2. The insulation thickness on roof and wall, the air flow rate and the refrigeration capacity were all specified in accordance with the optimized model. The results of the two trials at Lah, summarized in Table 3, indicated that adequate insect control temperatures were achievable, that energy cost was comparable with that of new protectant insecticides, and that the peripheral duct satisfactorily cooled the central grain and peak of the bulk in addition to the grain at the silo wall.

TABLE 3
SUMMARY OF RESULTS OF TWO GRAIN REFRIGERATION TRIALS WITH WHEAT IN AN INSULATED 1700 TONNE STEEL SILO 14.5 m DIAMETER AND 13.1 m WALL HEIGHT AT LAH, VICTORIA

Trial No. Year	1 1976/77	2 1977/78
Air flow rate (L/s per tonne of silo capacity)	1.5	1.5
Compressor rating (shaft W/tonne of silo capacity)	4.4	4.4
Quantity of wheat (t)	1650	1750
Initial grain moisture (% wet basis)	8.8	8.3
Final grain moisture (% wet basis)	9.2	8.8
Storage period (months)	9	5
Inital grain temperature (°C)	34	30
Initial cooling to 15°C (months)	1.2	1.4
Final grain temperature (°C)	9	9
Total operating time (hours)	–	–
Energy used (kWh/tonne of silo capacity)	24	22
No. of live insects at inloading	0	0
Quantity of grain sieved (kg)	276	271
No. of live insects at outloading	0	0
Quantity of grain sieved (kg)	204	210

Trials at Gravesend, NSW

The ability of the direct-expansion refrigeration system and continuous air flow through the grain at the silo wall to provide adequate cooling performance led to a concept of modular cooling units of a standard design to suit all types of grain storages (Elder, 1980). Six such units were installed on a 15 000 tonne capacity horizontal shed storage at Gravesend in the northern wheat belt of New South Wales. Each unit recirculated refrigerated air through individual sections of the bulk thus enabling positive cooling of the first loads of grain entering the storage. A description of the system and some preliminary results were given by Thorpe and Elder (1978).

Unlike previous trials where filling of the storage was completed in only a few days, the 15 000 tonne shed took many weeks to fill. In the first trial, cooling was not commenced until the grain at the temperature monitoring position was alsmost at its full height. The objective of this was to observe the development of cooling patterns from the peripheral air distribution ducts towards the central regions of the bulk, and confirm model predictions that central ducting in sheds even as wide as 24 m was not necessary. The patterns developed after 17 and 39 days of plant operation are shown in Fig. 3 and clearly demonstrate that the centre of the bulk is being cooled progressively.

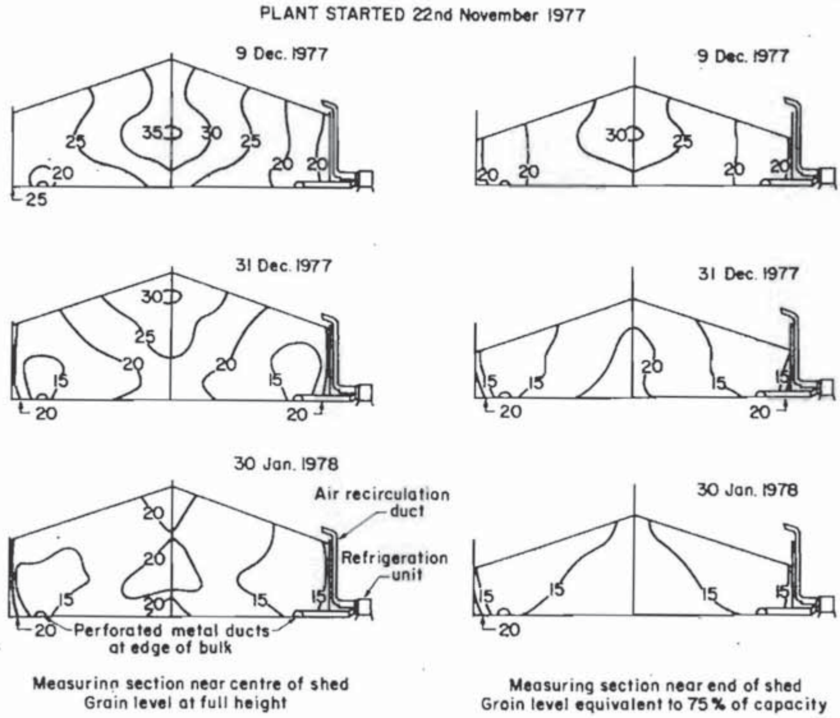

Fig. 3 Grain temperature contours at two measuring sections of 15 000 t capacity insulated horizontal storage at Gravesend, NSW showing progressive cooling from ducts on floor near walls to centre. Note: Air flow of 0.7 L/st only two-thirds of specified rate.

During the trial, the air flow rate was found to be substantially less than the specified value and consequently the cooling performance was not as good as it should have been. However, very good control of infestation was achieved as shown in Table 4. In a subsequent trial when the air flow had been brought up to specification, the cooling of the centre of the bulk was proportionally more rapid as shown in Fig. 4.

The initial cooling performance of the system as shown in Table 4 was never as good as in previous trials, even though the refrigeration capacity was greater. After exhaustive measurements and testing, it was found that outside atmospheric air was leaking into the air recirculation circuit of the refrigeration units downstream of the cooling coil. This leakage amounted to about 12% of the air flow. In the hot summer period following the filling of the storage, this admixture of outside air with the refrigerated air off the cooling coil almost annulled the cooling effect before the air could reach the grain. Consequently, the average grain temperature could not be brought below 15°C until the onset of consistently cooler atmospheric conditions. This is illustrated in Fig. 5 by cooling performance in the third season at Gravesend which is compared with that at Brookstead in the same climatic zone.

TABLE 4

SUMMARY OF RESULTS OF TWO GRAIN REFRIGERATION TRIALS WITH WHEAT IN 15000 TONNE HORIZONTAL STORAGE 96 m LONG, 24 m WIDE AND 6 m WALL HEIGHT AT GRAVESEND, NSW

Trial No. Year	1 1977/78	2+ 1980/81
Air flow rate (L/s per tonne of silo capacity)	0.7	1.0
Compressor rating (shaft W/tonne of silo capacity)	6.5	6.5
Quantity of grain (t)	12160	4335
Initial grain moisture (% wet basis)	9.9	10.9
Final grain moisture (% wet basis)	11.0	11.8
Storage period (months)	10	10
Initial grain temperature (°C)	32	28
Initial cooling to 15°C (months)	2.9	4.5*
Final grain temperature (°C)	9	12*
Total operating time (hours)	-	-
Energy used (kWh/tonne of silo capacity)	26	29
No. of live insects at inloading	0	5
Quantity of grain sieved (kg)	968	404
No. of live insects at outloading	5	19
Quantity of grain sieved (kg)	1016	340

* Weighted mean for three separate heaps of cooled grain

\+ Drought season

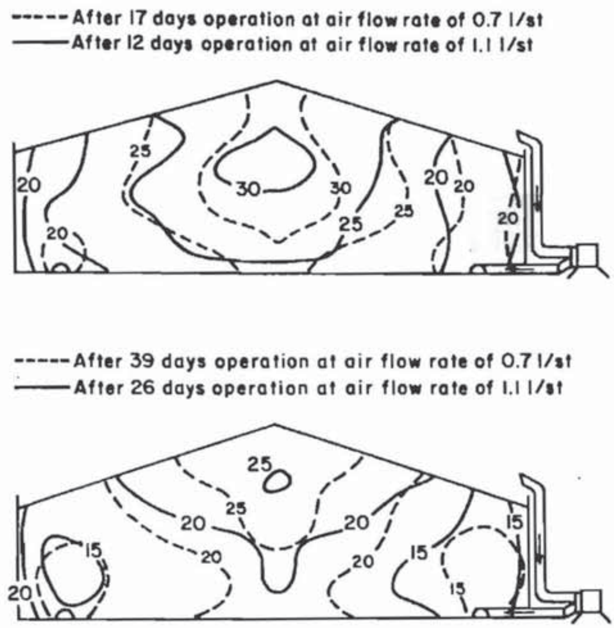

Fig. 4 Grain temperature contours at central measuring section of 15 000 t capacity insulated horizontal storage at Gravesend, NSW showing effect of increased air flow on cooling rate at centre of bulk.

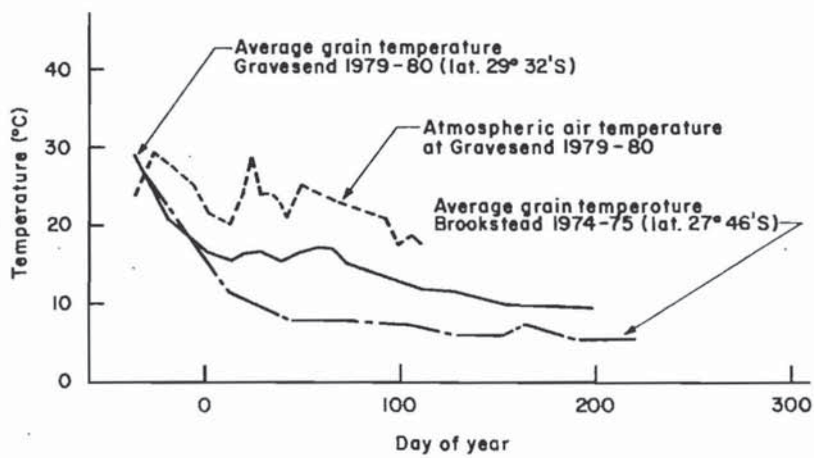

Fig. 5 Comparison of cooling performance between the grain refrigeration systems at Brookstead, Queensland and at Gravesend, NSW showing the dwell in the initial cooling at Gravesend resulting from 12% leakage of atmospheric air into the recirculating air downstream of the cooling coil.

A few live insects were occasionally found at the peaks of the grain throughout most of the storage period. At the conclusion of the first season's storage, grain temperatures were monitored in the railway wagons transporting the refrigerated grain to the port, a distance of some 600 km over four days. The average grain temperature increased from 10 to 12°C during the journey. The grain was not treated with insecticide at any time and passed inspection for export several weeks later.

During the second season, part of the bulk become infested before one of the refrigeration units (being modified for increased air flow) could be re-installed. A surface spray and subsequent cooling arrested the infestation. At the end of the season it was necessary to carryover this grain and it was transferred without the usual insecticide treatment to the emptied end of the shed for continued cooling. The new season's grain was then received and cooled as indicated in Fig. 4. Although it would have been most informative to have sieved the grain from the two seasons at outloading to determine the level of insect activity, the cost of the exercise precluded a rigorous sieving programme similar to that conducted for the first season.

The dynamics of insect activity at the peaks of the bulk throughout the second and third season is shown in Fig. 6. It must be remembered that with cool air flowing up to the surface, insects will migrate as shown by Navarro *et al.*,(1981) to the warmer zones such as the peaks. Therefore the infestation that may be found by inspectors is not representative of the general condition of the grain as in typical storages, but gives a biased result suggesting that the grain needs to be treated to meet export inspection criteria. Although it would be prudent, and in some States of Australia legally mandatory, to treat the surface infestation prior to outloading, the grain may still meet export inspection requirements as was demonstrated in the first season. The concentration of any infestation that may be present at the readily accessible surface, facilitates very effective insecticide treatment and will lead to exceptionally low residual insect populations.

A severe drought in the northern wheat belt of NSW resulted in very little grain being available for the fourth and final trial at Gravesend. The performance of the refrigeration system with separate heaps of grain and no central ducting was observed, and the overall results are summarized in Table 4. The effectiveness of the cooling from the peripheral ducts was related directly to the height of grain. With the grain level at the equivalent of 75% full capacity, cooling was satisfactory and no live insects were found at outloading. However, at the 50% full level, insufficient air was passing up through the central regions of the bulk to cool the peak and live insects could be found easily by sieving. One of the heaps of grain did not flow over any of the perforated ducting near the walls. It did not cool by conduction to the surrounding refrigerated air and, later in the storage period, a hot spot and heavy infestation was detected at the floor near the centre of the heap. To prevent contamination by this infestation of the cooled grain to be sieved during outloading, the uncooled heap was sheeted and fumigated with phosphine. The calculated quantity of gas dispersed in the headspace air would have been well below lethal levels determined by Hole *et al.*, (1976) and would not have affected live insects in the cooled heaps. The insect numbers given in Table 4 are for the live adults found in the cooled grain.

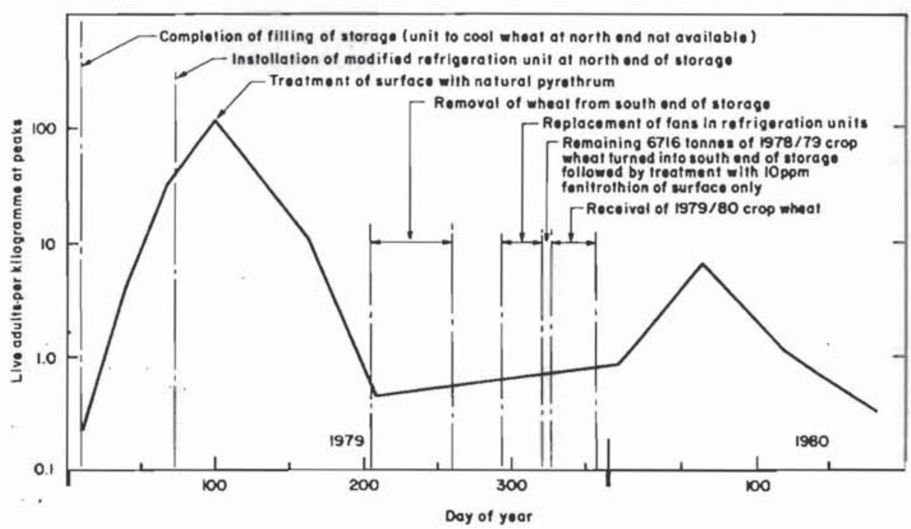

Fig. 6 Overall insect population desities at the peaks of the surface of wheat held over two seasons in 15 000 t insulated horizontal storage at Gravesend, NSW.

Central ducting, although not necessary if the storage is at least 75% full, should be considered to cater for poor seasons or small quantities of grain in separate heaps. The air flow may be changed-over from the central to peripheral ducting at the end of the initial cooling period as at Brookstead, or for simplicity, flow into both duct systems continuously. A central system will also hasten the initial cooling of the peaks of the bulk, but will detract from cooling at the walls for the majority of the storage period.

Performance of insulation

The insulation was applied to the outside of the storages for a number of reasons concerned mainly with hygiene and operational matters. The uptake of moisture by the insulation under the vapour pressure gradient imposed by the Queensland climate was monitored for several years at Brookstead and shown by Elder *et al.*, (1975) to stabilize after an initial increase. The moisture uptake was too small to have any noticeable effect either on the thermal conductivity, or on the roof load.

Various coatings to protect the insulation from ultra violet (u.v.) degradation have been tried. Low cost aluminium paint did not adhere satisfactorily in the long term; but the most disturbing feature of this coating was that it highlighted irregularities in the spray application of the insulation foam, and consequently attracted considerable criticism from the industry. Subsequently, PVA white paint was applied which improved the appearance of the silo at Brookstead considerably. At Lah, a white elastomeric hypalon coating was applied which has proved satisfactory but very expensive. Likewise at Gravesend, a white hypalon mastic coating was applied. The costs of various coatings have been analysed and a panel of a number of these is undergoing long term tests at Gravesend.

Damage to the insulation and coating has resulted from a number of environmental factors. At Brookstead, rodents found the 50 mm thickness of insulation on the roof to be sufficient to form nests. Furthermore, the burrows collected rain water, and this was a major cause of local wetting of the insulation. The subsequent mathematical modelling by Thorpe and Elder (1977) showed the optimum roof insulation thickness to be about 15 mm which would be insufficient to enable burrowing by rodents. At Lah, the insulation was attacked by birds and it appears that this attack is continuing. Pieces of the elastomeric coating could also be peeled off the insulation leaving the exposed yellow surface vulnerable to u.v. degradation as well as, perhaps, making it more attractive to birds. Being an isolated site remote from housing, some vandalism of the insulation has occurred but this was not as serious nor as persistent as bird damage. At Gravesend vandalism was limited to embedding of stones and other missiles in the wall insulation. After the first season, birds appeared to lose interest in attacking the insulation. Rodents have burrowed into the wall insulation at fillets where structural members contact the grain retaining wall. Considering that the total area of insulation is over half a hectare, the proportion of damage is extremely small; however the blemishes in the surface give the impression of a serious shortcoming.

Fears that heavy hail storms will tear the insulation of the storages have to date been proved groundless, although at Gravesend there is evidence that hail has fallen on the storage and caused a crazed cracking of the mastic coating.

Cost

One of the objectives of conducting full scale field trials in collaboration with the industry was to positively indentify the real commercial costs of grain refrigeration. These have been used in various studies by the Bureau of Agricultural Economics and others as well as in our own cost optimization modelling work. Connell and Johnston (1981) have shown that the annual costs are similar to those for controlled atmosphere storage, and of the order of 2% of the value of the grain stored.

Too concentrated a focus on the cost of applying refrigeration can lead to an unbalanced perspective of the role of the treatment in the overall grain storage and handling system. This is perhaps best illustrated by an example. The 5400 tonne silo at Brookstead had a history of uncontrollable insect problems and at the time we chose it for the first trial of full scale refrigeration, it was being used only for short term storage of grain sorghum when absolutely necessary. For less than one tenth the cost of replacing the storage, refrigeration redeemed it as a most valuable facility for long term storage of malting grade barley. The thrust of CSIRO's research programme on grain refrigeration has been towards similar storages such as the shed type. It was thought that shed storages may prove totally unsatisfactory if chemical protectant insecticides could no longer be used to control infestation because of resistance or commercial or industrial factors.

The capital costs for the three refrigeration systems are summarized in Table 5. Using the Consumer Price Index (a measure of inflation) to normalize costs, it will be seen that the cost of insulation per tonne of storage capacity is very closely related to the surface area to capacity ratio of the storage. A generalized graph showing a range of typical grain storages is shown in Fig. 7 with the trial storages plotted. Clearly, the larger the storage capacity, the lower the cost per tonne. For example, the cost of insulating CBH's No. 1 shed at Kwinana, Western Australia, also shown in Fig. 7, is about one third of the cost of insulating the shed at Gravesend.

TABLE 5
CAPITAL COST OF RETRO-FITTED GRAIN REFRIGERATION SYSTEMS IN AUSTRALIA

	Brookstead Queensland	Lah Victoria	Gravsend N.S.W
Year of installation	1973-74	1976-77	1977-78
Consumer Price Index	46.7	70.1	76.7
Capacity of storage (t)	5400	1700	15 000
Total capital cost ($/t)	8.1	18.4	14.2
Insulation cost - ($/t)	3.0	7.6	5.8
- ($/m^2)	10.1	15.6	15.5

Note: Tabulated costs in Australian dollars
A$1.00 in June 1973 = US$1.42
1976 = US$1.24
1977 = US$1.12

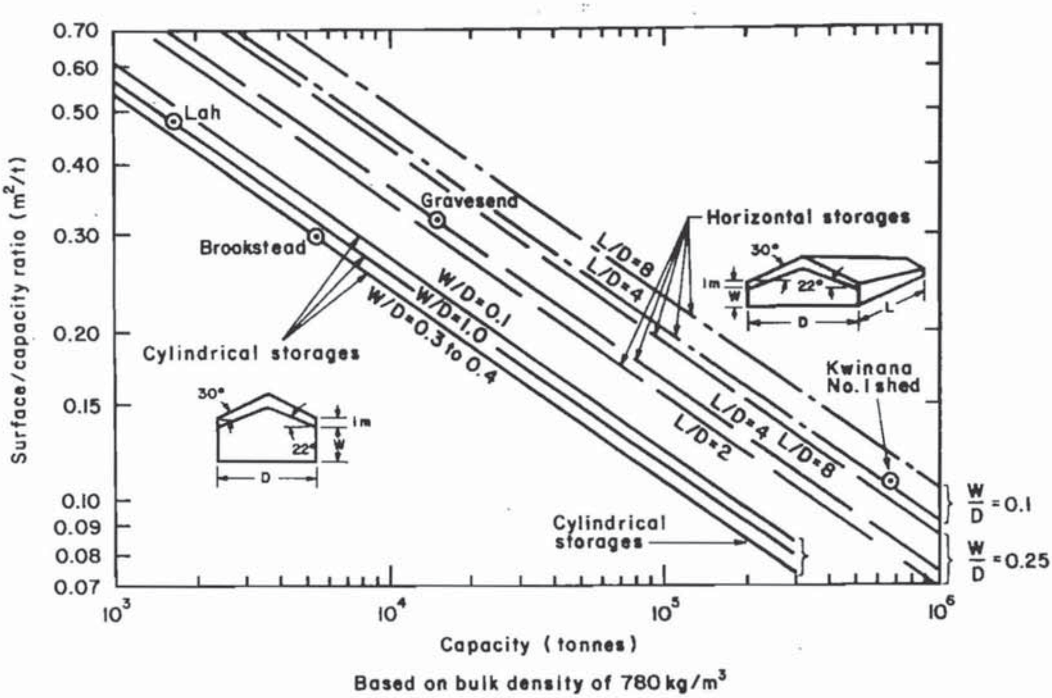

Fig. 7 Chart for surface area to capacity ratio representing capital cost for insulation per tonne for grain storages of various capacities and configurations.

Operating costs are indicated by the electrical energy consumption in Tables 1 to 4. Maintenance and servicing costs vary considerably but do not appear to exceed 4¢/t p.a. (Connell and Johnston, 1981).

Repair costs for insulation are governed by the degree of deterioration tolerated before refurbishing is implemented. No repairs have been carried out at Lah even though a total of approximately 5 m^2 has been attacked by birds. A quotation of $600 was obtained in 1980 for abour 2 m^2 repair. The cost per unit will obviously be higher than that quoted in Table 5 because of relatively high establishment costs, the additional cost of frequent movement of scaffolding to reach isolated damaged regions and difficulties in spraying the foam into depressions. The cost of repair plus application of additional insulation for experimentation purposes at Gravesend was 11c/t after three years.

MOBILE GRAIN REFRIGERATION UNITS

The modular refrigeration unit concept permits the use of the treatment for other applications in the grain storage industry, by mounting units on trailers for transportation to a number of sites involving a wide variety of storage types and stored commodities.

Cooling of protectant treated grain was shown by Bengston *et al.*, (1980), Desmarchelier *et al.*, (1979) and Thorpe and Elder (1980) to prolong the effectiveness of the insecticide, and a trial was conducted at Murtoa, Victoria in collaboration with the Stored Grain Research Laboratory and the Grain Elevators Board using a mobile refrigeration unit. On the basis of the results, an economic analysis has been presented by Hunter (1981).

Mobile refrigeration units of similar capacity as used at Lah, Gravesend and Murtoa are being used by the State Wheat Board in Queensland for cooling high moisture grain sorghum, and by Co-operative Bulk Handling Limited for cooling wheat in Western Australia.

These and other applications facilitate the gradual introduction of the technology into the industry as relatively modest amounts of money are made available.

CONCLUSIONS

Trials using refrigerated air to cool grain stored in large bulks have shown that the treatment is feasible and effective as an insect control measure, and that the cost is reasonable. Early work showed that insulation was necessary to maintain all of the grain below 15°C, the insect population growth threshold adopted for design. Adequate control of grain temperatures near the wall was achieved by using peripheral air distribution ducting. In sheds, ducts placed close to the wall provide adequate cooling of the central regions of the bulk only if the storage is substantially full. An additional central duct system would cater for small heaps of grain particularly in drought seasons, and also enhance the initial cooling of the peaks of the bulk.

Although live insects may be detected at the peaks of refrigerated grain bulks, their prescence does not necessarily indicate that the grain will not pass export inspection. The movement of cold waves up to the warmer peaks concentrates live insects at the surface where they can be treated appropriately. During transportation, the temperature of refrigerated grain remains low thus providing a residual protection from insect problems.

Spray-applied closed cell rigid polyurethane insulation can be put successfully on the outside of the structure without including long term moisture accumulation from the adverse water vapour pressure gradients. The thickness must be such that rodents cannot form burrows, and care must be exercised in ensuring that fillets of insulation against structural members are avoided. Minor repairs to the insulation resulting from damage by birds will be necessary in most circumstances. The best coatings are in general the most expensive. A mastic is preferred to an elastomeric coating which can be peeled off the insulation. However, the long-term effects of crazed cracking of mastic by hail, on the degradation of the insulation under the cracks is yet to be observed. Hail has not caused any damage of a serious nature to date.

The capital cost of retro-fitting refrigeration systems to existing silos is a noticeable proportion of the cost of the storage facility and cannot compete with alternative insect control measures. It can however be justified if the alternatives are banned, or unsatisfactory facilities can, by becoming refrigerated, be upgraded to an acceptable standard. In the future, refrigeration may be regarded as an integral part of new storage facilities and its additional cost would then be a small proportion of the overall project budget. Operation and maintenance costs will then become the most noticeable feature of the system. It appears this is presently of the order of $1 per tonne per annum. Refrigeration cost is lower the larger the storage structure, and therefore this treatment is more worthwhile for the large horizontal storages common in Australia which may not otherwise provide satisfactory protection against insects.

The plant for grain refrigeration has been developed on a unit module for cooling about 2000 t of grain. This has been shown to facilitate the use of these cooling units for a number of pseudo-refrigeration applications involving various criteria other than direct insect control. this will lead to the gradual acceptance by the industry of the technology and in due course will enable a smoother transition to full scale refrigeration when necessary.

ACKNOWLEDGEMENTS

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The many co-operating bodies involved in the trial included the Australian Wheat Board, the State Wheat Board, Queensland and the Grain Elevators Boards of Victoria and New South Wales and CSIRO Divisions of Entomology and Food Research.

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SESSION 11.

CHEMICAL CONTROL

Papers by:

M. Bengston	–	Australia
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DEVELOPMENT OF GRAIN PROTECTANTS FOR USE IN AUSTRALIA

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ABSTRACT

Since the early 1960's Austalia's high standard of pest control in stored grain has depended chiefly on the application of grain protectants. Malathion was used until 1976-79, malathion-resistant strains then became predominant so alternative insecticides, chiefly fenitrothion but also pirimiphos-methyl and chlorpyrifos-methyl, were substituted progressively in the various States. Addition of synergised bioresmethrin was required in areas, initally the Eastern States, where prevalent strains of *Rhyzopertha dominica* were cross-resistant to the newer materials.

Development of appropriate protectant treatments commences with surveys of insecticide-resistant strains in storages and establishment of laboratory cultures of significant strains. Initial screening of candidate compounds is done in the laboratory using impregnated-paper assays and treated-grain assays. Field testing is then carried out in commercial storages with periodic grain samples tested in laboratory bioassays and chemical assays.

Compounds under development which have potential to control all species include methacrifos, deltamethrin, cyfluthrin and cypermethrin. Fenvalerate, permethrin and phenothrin have potential to control *Rhyzopertha dominica*. Data generated include residue levels both on grain and in major grain products e.g. bran, germ, flour and bread. Data are also generated on the rate of breakdown of these compounds at a range of grain temperatures and grain moistures. All residue levels must conform to maximum residue limits recommended by the Codex Alimentarius Commission.

INTRODUCTION

Grain protectants have been widely used in Australia for the control of pests of stored grain since the introduction of malathion for the purpose in the early 1960's. The standard of pest control required is high. Over 60% of production is exported and is subject to an export inspection, the standard of which is nil tolerance for live insects.

Most of the grain is stored in bulk in central premises and the grain protectants are sprayed into the grain stream during intake into storage. In unaerated storages the grain is typically hot and dry, and grain temperature averages around 30°C during the storage interval. Approximately 25% of storages have aeration, and in these the grain temperature is reduced to 15°C after 5 months storage. The Co-operatives and state run Instrumentalities receiving grain are empowered to prevent the growers from delivering commodities with high moisture contents to central premises. Currently, the maximum permissible moisture content for wheat is 12% and for sorghum 13.5%.

By the 1970's malathion-resistant strains of grain storage insects had developed both in Australia and in many overseas countries (Champ and Dyte, 1976). Research in Australia established that chlorpyrifos-methyl, fenitrothion and pirimiphos-methyl were effective against most of the malathion-resistant strains. Synergised bioresmethrin, carbaryl and synergised natural pyrethrins were shown effective against *Rhyzopertha dominica* (F.) including strains not effectively controlled by the organophosphorus compounds listed. All of these compounds have been cleared for use as grain protectants within Australia and use of malathion progressively declined form 1976 to 1979. The major usage has been of fenitrothion and this is combined with synergised bioresmethrin in the Eastern States and South Australia where resistant *R. dominica* predominate.

Further development of resistance must be anticipated and recent testing of additional alternative insecticides for use as grain protectants will be described.

TEST PROCEDURES

Resistant Strains

Test insects to be used in evaluation of newer grain protectants need to be representative of insecticide-resistant strains of the major pest species. Extensive surveys were carried out using the techniques developed by Champ (1968) to determine the insecticide-resistant status of insects collected from storages throughout Australia. Cultures of significant strains are now maintained in the laboratory under continuous selection with insecticides. Surveys continue and new strains are added to the battery of test insects as appropriate.

Laboratory Assays

Acute toxicity of candidate compounds is estimated initially using impregnated-paper assays adapted from the FAO method for measurement of resistance (Anon., 1974). Promising compounds are further evaluated using treated-grain assays. Test criteria include mortality of test adults at 3 and 26 days and reduction in progeny in F_1 and F_2 generations. These assays are carried out on samples of treated grain taken at intervals over the period of a year. Chemical residues are determined simultaneously.

Field Testing

Promising compounds are field tested at application rates suggested by laboratory studies. Field testing provides the ultimate assessment of the performance of a protectant under industry conditions. Important aspects include stability and ease of mixing of formulations, residues in grain dust and in grain products and acceptability of the compound to industry.

Laboratory bioassays and chemical assays are carried out on samples of treated grain and these are the most important criteria. The presence of natural field infestation is evidence of failure of a treatment but absence of field infestation is not strong evidence of success unless testing involves numerous (more than 20) storages.

This field testing has involved extensive industry co-operation throughout Australia. The Australian Wheat Board's Working Party on Grain Protectants undertakes field testing in each of the mainland States and other boards and authorities co-operate in work with other grains. These groups receive excellent co-operation from the chemical industry.

COMBINATIONS OF GRAIN PROTECTANTS

By the early 1970's common strains of *Tribolium castaneum* (Herbst), *Sitophilus oryzae* (L.) and *R. dominica* in eastern Australia were malathion-resistant. Significantly, the malathion-resistant strains of *T. castaneum* and *S. oryzae* could be controlled at acceptable application rates by available organophosphorus materials:chlorpyrifos-methyl, fenitrothion and pirimiphos-methyl. Unfortunately the common strains of *R. dominica* were cross-resistant to all of these compounds and for convenience these strains here will be termed multi-resistant. The multi-resistant strains were nevertheless susceptible to bioresmethrin, the most active of the available synthetic pyrethroids.

The response of some important strains to bioresmethrin and fenitrothion is shown in Table 1. The responses to chlorpyrifos-methyl and pirimiphos-methyl were essentially parallel to those for fenitrothion.

TABLE 1 - KC 99.9 (mg kg^{-1}) in newly treated grain based on significant response of test insects after 3 days in treated grain at 25°C and 70% RH.

Species	Strain	Resistance status	Malathion resistance factor	Bioresmethrin	Fenitrothion
Sitophilus oryzae	QSOLS2	Susceptible	–	15.8	0.2
	QSO56	Resistant	x 3.5	10.4	0.4
	CSO231	Highly resistant	x 8.9	352.0	2.0
Rhyzopertha dominica	QRD14	Susceptible	–	0.6	7.7
	QRD2	Resistant	x 5.7	0.8	12.4
	QRD63	Multi-resistant	x 78.0	0.6	10.9
Tribolium castaneum	QTC39	Susceptible	–	7.5	0.5
	QTC34	Highly resistant	x 39.4	26.2	0.5

In the absence of a single insecticide capable of controlling all pest species, combinations of insecticides provided the obvious pragmatic solution (Bengston *et al.*, 1975). Bioresmethrin has been widely used in combination with one of the organophosphorus materials and results have been excellent (Desmarchelier *et al.*, 1981).

PROTECTANTS TO CONTROL MAJOR PESTS (EXCLUDING MULT-RESISTANT *R. DOMINICA*)

Extensive testing established that all significant grain storage pests in Australia, excluding multi-resistant *R. dominica*, were controlled by chlorpyrifos-methyl, fenitrothion or pirimiphos-methyl. Current work indicates that etrimfos will be equally effective. Species tested include *S. oryzae*, *S. granarius* (L.), *R. dominica*, *T. castaneum*, *T. confusum* Jacquelin du Val, *Oryzaephilus surinamensis* (L.), *Ephestia cautella* (Walker) and *Plodia interpunctella* (Hubner).

PROTECTANTS TO CONTROL MULTI-RESISTANT *R. DOMINICA*

Insecticides of both the carbamate and synthetic pyrethroid groups are generally potent against multi-resistant *R. dominica*. Carbaryl (a carbamate) and bioresmethrin, cyfluthrin, cypermethrin, deltamethrin, fenvalerate, permethrin and phenothrin (synthetic pyrethroids) have all been shown effective in field testing (Bengston *et al.*, 1980 a,b, 1983 a,b).

PROTECTANTS TO CONTROL ALL MAJOR PESTS

Methacrifos and synergised cyfluthrin, cypermethrin and deltamethrin, have been shown effective against all the currently prevalent strains of major pest species. However, the synergised synthetic pyrethroid materials when formulated as emulsifiable concentrates all produced a nasal irritation to workmen with inhalation exposure. The problem was resolved by substitution of an unsynergised suspension concentrate formulation of deltamethrin. Alternative unsynergised formulations of cyfluthrin and cypermethrin are currently being tested.

SYNERGISM OF SYNTHETIC PYRETHROIDS

Available synthetic pyrethroid insecticides have been relatively expensive and synergism with piperonyl butoxide has been an important means of reducing application rates and hence costs.

Data on synergism of important compounds are given in Table 2.

TABLE 2 - Synergism of synthetic pyrethroid insecticides with piperonyl butoxide 10 mg kg^{-1}, based on mortalities of test insects after 3 days in treated grain at 25°C and 70% RH.

Insecticide	Species	Strain	Synergism Factor	(95% Limits)
Bioresmethrin	*Rhyzopertha dominica*	QRD63	2.24	(1.76 - 2.91)
Cypermethrin	*Sitophilus oryzae*	QS056	4.74	(4.35 - 5.16)
Deltamethrin	*Sitophilus oryzae*	QS056	2.33	(2.10 - 2.60)
	Rhyzopertha dominica	QRD63	6.60	(5.00 - 9.42)
Fenvalerate	*Rhyzopertha dominica*	QRD63	1.93	(1.78 - 2.08)
Permethrin	*Rhyzopertha dominica*	QRD63	2.07	(1.87 - 2.29)
Phenothrin	*Rhyzopertha dominica*	QRD63	2.38	(2.02 - 2.81)

COMPARISON BETWEEN GRAINS

Most of the detailed research of the efficacy of grain protectants in Australia has been carried out in relation to wheat which is the major cereal species produced. Recent studies have shown differences of surprising magnitude in the responses to insecticides of test insects in wheat and sorghum (Bengston *et al.*, 1983 a,b). The relative potencies of three insecticides are presented in Table 3.

TABLE 3 – Relative potencies of insecticides on sorghum compared to wheat, based on mortalities of test insects after 3 days in treated grain at 25°C and 70% RH.

Insecticide	Species	Strain	Relative Potency	(95% limits)
Bioresmethrin	*Rhyzopertha dominica*	QRD63	0.27	(0.25 – 0.30)
Carbaryl	*Rhyzopertha dominica*	QRD63	0.54	(0.42 – 0.69)
Fenitrothion	*Sitophilus oryzae*	QS056	0.45	(0.39 – 0.50)

Data suggest that to control an established infestation in sorghum an insecticide application rate 2 to 3 times that of wheat would be required. At the same time experience in the grain industry in Australia is that the frequency of pest problems during storage of sorghum is less than that for wheat. Factors such as lower mean grain temperature for stored sorghum in comparison to wheat (25°C compared to 30°C) are probabbly important. Currently the same application rates are used on the major cereal species.

SUMMARY OF APPLICATION RATES

In Australia, application rates are calculated so as to give effective protection from infestation during storage of from 3 to 9 months duration. In general these rates are halved for storage from 0 to 3 months and retreatment is considered appropriate for longer term storage. Application rates which have been shown to be effective for 9 months storage are shown in Table 4.

TABLE 4 - Grain protectants for control of malathion-resistant insects in Australia during 9 months storage.

MAJOR SPECIES EXCLUDING MULTI-RESISTANT *RHYZOPERTHA DOMINICA*

Chlorpyrifos-methyl	10 mg kg^{-1}
Fenitrothion	12 mg kg^{-1}
Pirimiphos-methyl	4 mg kg^{-1}

RHYZOPERTHA DOMINICA ONLY

Bioresmethrin 1 mg kg^{-1} plus piperonyl butoxide 10 mg kg^{-1}
Carbaryl 8 mg kg^{-1}
Deltamethrin 0.1 mg kg^{-1} plus piperonyl butoxide 10 mg kg^{-1}
Fenvalerate 1 mg kg^{-1} plus piperonyl butoxide 10 mg kg^{-1}
Permethrin 1 mg kg^{-1} plus piperonyl butoxide 10 mg kg^{-1}
(1)-phenothrin 2 mg kg^{-1} plus piperonyl butoxide 10 mg kg^{-1}

ALL MAJOR SPECIES

Cyfluthrin 2 mg kg^{-1} plus piperonyl butoxide 10 mg kg^{-1}
Cypermethrin 4 mg kg^{-1} plus piperonyl butoxide 10 mg kg^{-1}
Deltamethrin 1 mg kg^{-1}
Methacrifos 20 mg kg^{-1}

RESIDUES

Extensive studies have enabled the residue levels likely to arise from use of these compounds to be predicted with an adequate level of accuracy. Desmarchelier (1978) provided an explanation of the rate of decay of organophosphorus materials on various grains at different grain temperatures and moisture levels. Noble and Hamilton (1981) and Hargreaves *et al.* (1982) have provided recent data on the residual behaviour of pyrethroids. Information has also been obtained on residue levels in major grain products e.g. bran, germ, flour and bread.

Australia has actively supported consideration of residues of these compounds on grains by the Codex Alimentarius Commission of the United Nations and maximum residue limits in Australia are based upon Codex recommendations. We envisage that support by Australia for the work of the Codex Alimentarius Commission will continue.

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FUMIGATION TRIALS WITH CARBON DISULPHIDE:CARBON TETRACHLORIDE (20:80) IN SILO BINS

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Abstract: Trials were carried out in Shanghai, China to develop a simple fumigation system for silo bins. Preliminary experiments in the laboratory and on a pilot scale showed that carbon disulphide: carbon tetrachloride (20:80 % w/w) was an effective fumigant against *Sitophilus zeamais, Tribolium castaneum* and *Cryptolestes pusillus* at a rate of 331 g m^{-3} for a 7 day exposure period at about 29°C grain temperature. A dosage of 220 g m^{-3} under similar conditions gave incomplete control. It was found that the fumigant mixture applied to the grain surface in two sealed test bins (height 30m) at 331 g m^{-3} gave complete control of test insects placed in the grain close to the surface (test insects included immature stages of *T. castaneum* and pupae of *S. zeamais*). Both fumigant components rapidly penetrated the grain mass and were retained at substantial concentrations for more than ten days.

1. INTRODUCTION

The main type of grain storage structure in China are rectangular warehouses ('flat storage'). These have been widely used in the past and there is much experience in pest control in such structures. However, recently silo bins have been built in coastal areas for storage of imported grain and for export. This paper describes trials carried out in Shanghai between June 1981 and October 1982 to develop expertise in insect control in such unfamiliar structures. The treatment investigated used $CS_2:CCl_4$ (20:80) as a fumigant under gravity penetration.

2. LABORATORY TESTS

Wheat (4kg, 13.6% m.c.) was treated with $CS_2:CCl_4$ (20:80) (2.136 g) in glass bottles of 6.45 L capacity at 26°. The tests were carried out in 3 replicates. Each test included 100 adults, 20 pupae and 20 larvae of *Sitophilus zeamais* Mots. The adult insects started to be knocked down after 1.5 hours exposure. After 72 hours all adult insects were dead*. There was no mortality in

* Editor's Note: No information is given on the response of the immature stages to the treatment.

untreated control samples. The grain was normal in colour and odour.

3. PILOT SCALE TESTS

Two wheat bulks, each of 75 tonnes, were treated with $CS_2:CCl_4$ (20:80) applied by watering can to the grain surface. The surface of each bulk was covered with 3 layers of hemp mats and then sheeted with polyethylene sheet (0.23 mm thickness), leaving a headspace of 3 m^3. The grain temperature was 28 to 29°C and the air temperature 31.7°C One bulk, of 90m^3 total volume and 5 mm depth, was treated with 19.8 kg (220 g m^{-3}) of fumigant mixture. The other of 83 m^3 volume, was treated with 27 kg (331 g m^3) fumigant mixture. The wheat piles remained covered for 7 days to ensure effective fumigation.

Caged insects were placed in the upper, middle and lower part of the treated grain bulks. There was no survival of adult insects in the bulk treated at the higher dosage. The test insects included *S. zeamais, Tribolium castaneum* Herbst and *Cryptolestes pusillus* Schon. There was some survival of *T. castaneum* adults in the bulk treated at 220 g m^{-3}. The higher dosage seems more appropriate.

Caged rats were placed at distances of 5, 10, 15 and 20 m from the treated piles to indicate leakage of toxic gas. There were no deaths in these test rats, showing that the plastic sheeting was effective in producing a sealed structure.

4. FULL SCALE TESTS

Full scale tests were carried out in June and September 1982 on imported wheat (13% m.c.) in two silo bins. The larger bin, No. 101, was 8 m diameter and 29.3 m high, volume 1180 m^3, and contained 863 tonnes of wheat. The smaller bin, No. 201, volume 534 m^3, contained 394 tonnes. The grain temperature at the time of treatment ranged from 26 to 30°C in the upper layer and was at 25°C in the middle and lower layers of the grain mass.

4.1 Method of bin sealing

The bins were not gastight. The top and bottom of the bins were sealed with paper stuck to the structure with resin plaster. Some small leaks still remained in the wall, base and outlet. A pressure test gave a pressure decay time of 4.5 mins for 500 mm H_2O to 200 mm H_2O.

4.2 Test insects

S. zeamais (adult and pupae), *C. pusillus* (adult), *Rhyzopertha dominica* (F.) (adult) and *T. castaneum* (all stages) were used as test insects. Six probe tubes, 3 m long, were inserted into the grain. The probes contained cages of each different species with 20 insects and feed in each. Probes were removed at daily intervals after the first 48 hours after treatment. The test cages were examined, incubated and then re-examined after 15 to 30 days to determine insect mortality.

4.3 Fumigation method

CS_2 and CCl_4 were weighed out appropriately and mixed in a 200 kg capacity tank to give an 20:80 (w/w) mixture to be applied at 331 g m^{-3}. The mixture was sprayed onto the surface of the grain in the silo bins from the tank which was pressured with CO_2 at 0.5 kg cm^{-2} from a gas cylinder.

4.4 Gas sampling positions and frequency

Gas samples were taken at five points in the bin: in the head-space 0.05 m above the grain, 3 m below the surface, 12.5 m below the surface, 28.5 m below the surface and at the base of the bin. Semirigid polyethylene tubing attached to the temperature sensor cable was used for gas sampling. Gas concentrations were determined three times within 48 hours of dosing and then daily until the storage was vented.

4.5 Method of ventilation

The fumigant was removed by forced ventilation in bin No. 101. The bin was already equipped with cross flow ductwork and a large fan (5.5 kW, 350 mm H_2O d.p., 2400 m^3h^{-1}). Fumigant was pumped from bin No. 201 with an old vacuum pump as no ventilation system was fitted.

4.6 Results

4.6.1 *Effectiveness against insects.* There was no survival of any test insects for 7 to 10 days exposure with a dosage of 66 g m^{-3} CS_2 + 265 g m^{-3} CCl_4 (See Table 1).

4.6.2 *Gas distribution.* With the fumigant mixture applied to the grain surface, high concentrations of CS_2 and CCl_4 were detected throughout the bin after 24-32 hours with peak concentrations reached after 32-48 hours.

4.6.3 *Operation.* The method of treatment was easy and safe. The total of mixed fumigant used, 400 kg, was prepared in 11 mins.

4.6.4 *Fumigant residues.* Fumigant residues were determined after airing. 0.2 ppm CS_2 and < 4.1 ppm CCl_4 remained.

4.6.5 *Environmental safety* Gas samples were taken from each floor in the silo. Only two of the 32 samples exceeded the standard level.

5. DISCUSSION

5.1 The mixed fumigant (CS_2:CCl_4) was effective. The use of the mixed fumigant reduces the fire hazard.

5.2 Table 2 shows the penetration and spread of the fumigant. The distribution achieved was good with the mixed gas penetrating more than 30 m downward.

5.3 In China, the gastightness of silos is not good. Although some preventative measures are used, leakage still occurs. It is expected that there will be improvements made in gastightness.

TABLE 1.

Mortality (%) and location of test insects in bin No. 101.

Location in bin	east	west	north	south	centre	centre
Period of exposure (days) [a]	2	3	4	5	6	7
S. zeamais						
adult	100	100	100	100	100	100
pupa	100	100	100	100	100	100
T. castaneum						
adult	100	100	100	100	100	100
egg	100	100	100	100	100	100
larvae	100	100	100	100	100	100
pupa	100	100	100	100	100	100
R. dominica						
adult	100	100	100	100	100	100
pupa	100	100	100	100	100	100
C. pusillus						
adult	100	100	100	100	100	100
pupa	100	100	100	100	100	100

a Fumigation started on June 26, 1982.

TABLE 2.

Fumigant vapour concentration (g m^{-3}) at various times (bin 101).

Fumigant	Location in bin	6 hr	24 hr	32 hr	72 hr	96 hr	120 hr	144 hr	168 hr	192 hr	216 hr	240 hr	264 hr
CS_2	headspace	271	34	28.5	9.9	9.9	4.9	2.8	2.6	1.1	0.55	0.55	0.55
	upper	70	90.2	89.7	51.2	58.3	35.8	30.3	32.5	19.5	19.8	22.8	17.6
	middle	138.6	121	121.6	78.1	88	59.4	31.4	50.6	45.1	34.1	42.6	33.8
	lower	1.7	61	62.2	46.8	60	46.2	42	49	37.7	38.5	46.5	38
CCl_4	headspace	361	52.8	47.3	19.8	17.6	6.6	3.3	3.3	2.2	2.2	2.2	2.2
	upper	39.6	128.7	124.3	67.1	84.7	46	41.8	39.6	30.8	29.7	30.8	24.2
	middle	92.4	223.3	212	124	151.8	85.8	48.4	70.4	63.8	57.2	69.3	51.7
	lower	2.2	104.5	114	79.2	111	73.7	74.8	71.5	61.6	61.6	77	59.4

SESSION 12.

INTEGRATION

Papers by:

C.P.F. de Lima	–	Swaziland
H.G. Greening	–	Australia
D. Chantler	–	Australia
J.A. Conway and G. Mohiuddin	–	Bangladesh
E.J. Bond	–	Canada
J.R. Wiseman	–	Australia

MINIMAL FUMIGANT REQUIREMENTS FOR LONG-TERM AIR-TIGHT STORAGE OF GRAIN

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ABSTRACT

In large air-tight storage structures, insect control is slow because the relatively large volume of oxygen is not depleted quickly enough to exert control. In Kenya, practical use of large hermetic bins during the past 10 years has shown that effective insect control can be obtained through the use of phosphine gas at the lower limit of the effective concentration determined in laboratory experiments. Complete insect control is achieved at 0.0303 mg/l dosage applied on the whole bin volume.

In developing countries where grain may be stored for relatively long periods of time to meet domestic requirements and long-term famine reserves, the use of low phosphine concentrations in hermetic containers may have economic advantages over controlled atmosphere obtained from the use of nitrogen and carbon dioxide gases.

INTRODUCTION

Traditionally, underground storage of grain under semi-hermetic conditions has been practised in Asia, Africa, Europe and America from pre-historic to recent times (Sigaut, 1980). Practical use of modern large scale long-term semi-underground storage has been made in the past 40 years in Argentina, Cyprus and Kenya.

One of the problems of the use of large scale structures has been the relatively slow rate of insect control under cool conditions (because physiological development is slower and so oxygen consumption less) and under conditions where initial insect populations are small and so consumption of oxygen is negligible in comparison to the large volume of the storage container.

When such conditions prevail, practical experience in Kenya has shown that effective insect control is achieved with the minimal use of phosphine gas.

AIRTIGHT STORAGE IN KENYA

The successful use of 68 large (1500 tonne) hermetic bins in Kenya to store over 100,000 tonnes of maize as a famine reserve has been described elsewhere (De Lima 1980a, b). Maize has been continuously stored in excellent condition in individual bins for up to 4 years. The bins have been used under practical operating conditions for over 10 years. Total gross weight losses have been below 0.3% per year. After 3 to 4 years of continuous storage under hermetic conditions the grain has been sold as top quality, Grade 1. The 68 bins have been used 121 times over a 9 year period. Oxygen depletion in the bins was associated with mould growth in the apical portion of the bin. Maintenance requirements were small and economically attractive in comparison with conventional storages.

EFFECTIVENESS OF MINIMAL USE OF PHOSPHINE

In initial filling (1972) 30 g of hydrogen phosphide were generated by the release of aluminium phosphide tablets (Phostoxin, Degesch, Co., West Germany) or 17.7 g dichlorvos releasing strips were placed at the top and side hatches of the bins before sealing. These were meant to control superficial infestation by *Sitotroga cerealella* and *Ephestia cautella*.

In subsequent sampling some bins were shown to have a residual insect population as may be seen in Figures 1 and 2. The insects were mainly *Sitophilus zeamais*. *Sitotroga cerealella* and *Tribolium castaneum* Samples were taken down the central axis of the bin and gave a higher than normal value of average insect numbers, but the figures are good indications of potential insect problems. As can be seen in Figures 1 and 2 the insects were not controlled by the low oxygen atmospheres and phosphine concentration was below 0.015 mg/l. A second treatment of 60 Phostoxin tablets, 30 at the top and 30 at the side hatch, applied after approximately 26 and 28 months of initial filling gave effective control. This second treatment raised the phospine concentration by 0.0303 mg/l. It is doubtful if sufficient phosphine from the initial treatment remained to augment the second dose.

In later fillings (1973 onwards) 60 Phostoxin tablets were used as described above. The effectivenes of these treatments which gave a long term concentration of 0.0303 mg/l phosphine can be seen in Figures 3 and 4. There was only one isolated record of insects in grain samples (Fig 3) and none in the other bin (Fig 4).

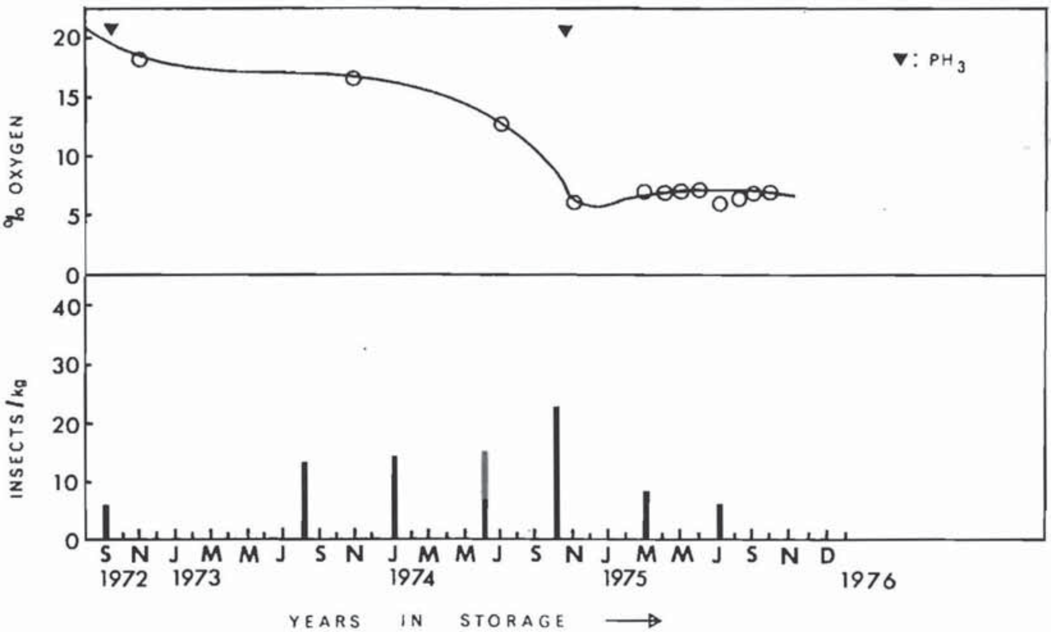

Figure 1. Initial 0.015 mg/l phosphine at filling, followed after 26 months with 0.0303 mg/l phosphine for complete control (Bin No. 40, Kitale).

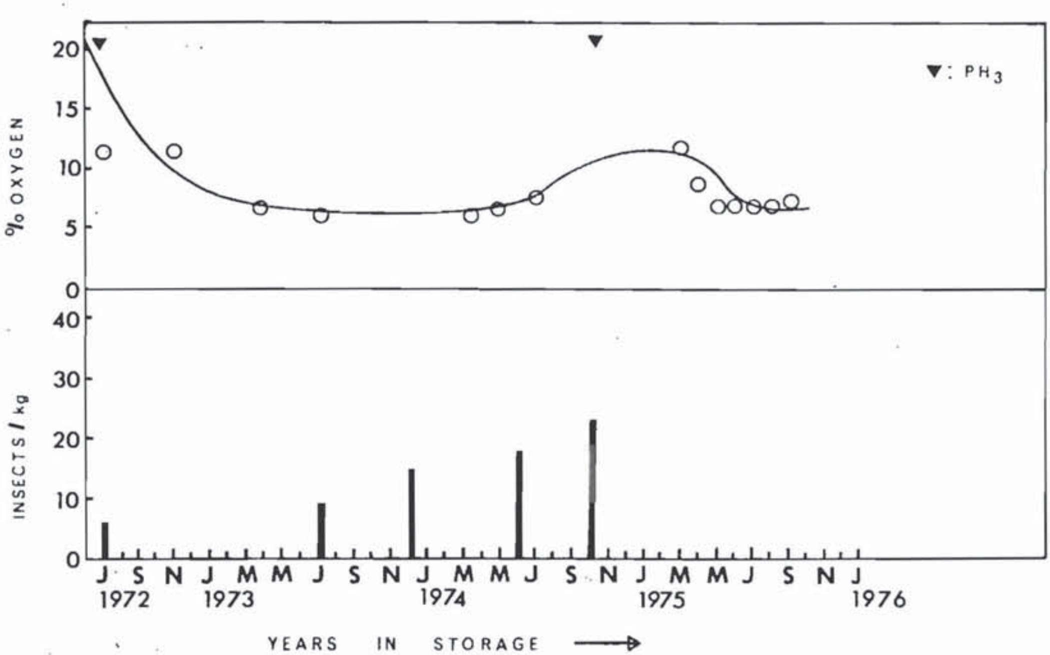

Figure 2. Initial 0.015 mg/l phosphine at filling, followed after 28 months with 0.0303 mg/l phosphine for complete control (Bin No. 33, Kitale)

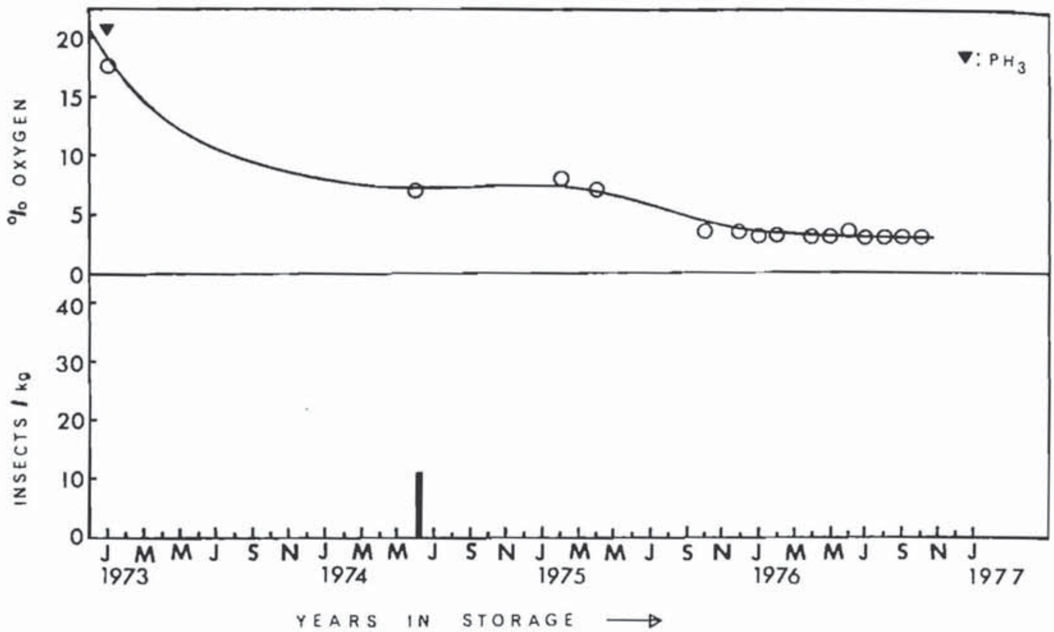

Figure 3. Effective control with single initial dose of 0.0303 mg/l phosphine. Isolated insect record after 18 months (Bin No. 12. Nakuru).

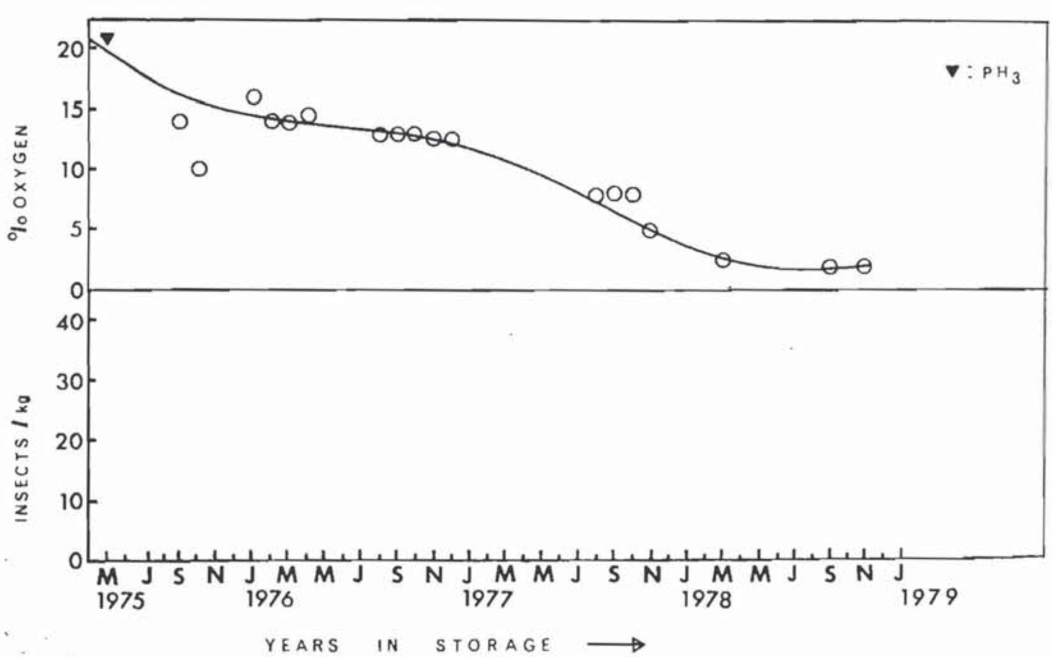

Figure 4. Effective control with single initial dose of 0.0303 mg/l phosphine (Bin No. 20. Nakuru).

DISCUSSION

Howe (1974) has reviewed at length various aspects of laboratory investigation of the toxicity of phosphine to stored product insects in an attempt to explain variations in results from field experiments. Part of the variability in laboratory and field trials is a result of varying susceptibility to phosphine by the different stages of the insect's life cycle (Bell 1976). This may be compounded in field trials with variations in daily temperatures especially if the trial is for a short period of time. Nakakita et al. (1974) obtained 100% mortalities of adult *Sitophilus zeamais* in the laboratory (at 25°C) within a 24 hour period at 25 ppm phosphine (approximately 0.035 mg/l) a value only slightly above that achieved in the Kenya hermetic bins. The presence of oxygen in the hermetic bins may be an advantage in enhancing the toxicity of phosphine to the insect pests. This has been demonstrated under laboratory conditions by Bond et al. (1967, 1969) and Chefurka et al. (1976). In a study of the toxicity of phosphine to diapausing larvae of *Ephestia elutella*. Bell (1979) found that (at 20°C) phosphine achieved peak efficiency at concentrations of 0.04 - 0.01 mg/l using a concentration - time product of 14 mg hr/l for 100% kill.

The length of exposure time for phosphine has not received much experimental attention. The need for this and the difficulties involved have been discussed by Howe (1974). Hole et al. (1976) have shown that long exposures are necessary for the control of tolerant insect species. Higher concentrations of phosphine produce narcosis (causing paralysis) permitting insects to survive longer because the fumigant inhibits metabolism (Nakakita et al. 1974).

In general however, Bell (1979) states, work over the past several years has shown that with phosphine, duration of exposure is more critical than gas concentration for ensuring insect control.

In the hermetic bins in Kenya the phosphine concentration used is 0.0303 mg/l and the time of exposure is several months. The experimental evidence in the literature discussed shows that the concentration of 0.0303 mg/l is at the lower end of the range required for control. However because of the hermetic conditions and the long exposure periods very large concentration x time products are achieved enabling complete control of insect pests.

In recent work, the use has been advocated of carbon dioxide and nitrogen atmospheres to artificially reduce the oxygen content of a storage container before finally sealing it. This approach has some economic merits in european countries where large quantities of grain are required to be harvested and stored at high moisture levels. The approach is advantageous in countries that are primary exporters of grain and are limited by nil tolerance requirements of insects and pesticide residues on their exported commodities. In developing countries such restrictions do not apply to locally consumed produce and the requirements are practically impossible to achieve because grain is received from large numbers of primary producers and is already infested at harvest. Under such conditions the use of minimum levels of phosphine in air-tight structures is an attractive option.

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FUMIGATION TRIALS WITH FARM-STORED GRAIN

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ABSTRACT

Fumigation of grain stored in bolted-iron farm silos was investigated in trials at Temora, Condobolin and Tamworth, 1976-82. Fumigant leakage from unsealed bins reduced the effectiveness of carbon disulphide, applied at dosages that were effective for 24 h fumigation in bins enveloped in plastic sheeting. Fumigation with 2, 6 or 12 g phosphine per tonne, applied as tablets containing aluminium phosphide probed into the grain, was effective both in unsealed bins and in bins enveloped in plastic sheeting for 7 to 10 days. Another tablet formulation containing aluminium phosphide, urea and ammonium bicarbonate, tested in conjunction with the aluminium phosphide, ammonium carbamate formulation used previously in these trials, gave comparable results at 6 g phosphine per tonne after 7 - 8 days in unsealed bins.

INTRODUCTION

The large quantity of grain stored on farms in eastern Australia (Lipton, 1980) includes grain for animal feeding that may be kept for long periods, depending on the availablity of natural fodder. It is usually held in iron silos of 25-80 t capacity, and is a potential source of insects (Greening, 1969, 1973) which may spread to harvesting equipment and contaminate newly harvested grain (Sinclair *et al.*, 1980).

Many farmers rely on periodic fumigation of the grain to prevent its destruction by insects. Carbon disulphide is commonly used in New South Wales. It is easily applied by pouring the liquid fumigant onto the surface of the grain in a bin. The need to confine the fumigant vapour in the bin (Hinds, 1924) is often overlooked. This prompted an investigation of the effectiveness of carbon disulphide fumigation in silos constructed from bolted iron sheets. The importance of distributing the fumigant as a spray over the grain surface (Walkden *et al.*, 1951) was also tested in these trials.

They were followed by trials of phosphine fumigation of grain stored under similar conditions. Phosphine became a popular fumigant for use on farms after the Grain Elevators Board of N.S.W. adopted it in 1955. For farm-stored grain, particularly in bulk sheds, phosphine was recommended for many years at dosages up to 10 g phosphine/t and the 1978 trial was conducted with that in mind. In later trials the choice of dosages was influenced by a review of grain fumigants used on farms (Williams *et al.*, 1980) in which 6 g phosphine/t was the maximum dosage recommended.

MATERIALS AND METHODS

Trial sites and formulations

Fumigation trials with carbon disulphide were conducted at Temora Agricultural Research Station (southern N.S.W.) in March, 1976 and April, 1977 and at Condobolin Agricultural Research Station (central N.S.W.) in May, 1977. Carbon disulphide manufactured by Stauffer Chemicals Ltd., Tomago, N.S.W. (1.2 kg carbon disulphide per litre) was used.

Phosphine fumigation trials were conducted at Condobolin Agricultural Research Station in April, 1978 and March, 1980 using Phostoxin (R) fumigation tablets (active ingredients, aluminium phosphide 56%, remainder inert ingredients including ammonium carbamate) manufactured by Degesch GMBH, Frankfurt/Main, FGR.

In May, 1982 at F. Klepzig & Sons' farm at Nemingha northern N.S.W. a trial was conducted to compare the efficacy of Phostoxin with that of a new material - Gastion (R) tablets (active ingredient, aluminium phosphide 57%, remainder inert ingredients including urea and ammonium bicarbonate), manufactured by Casa Bernardo Ltda, Santos, S.P. Brazil.

Silos

Grain was fumigated in silos made of curved steel sheets (galvanised iron) bolted together without sealant, a type of bin commonly used on Australian farms. They had flat concrete floors, except at Tamworth where both silos had conical concrete bases. The silos used at Temora in April, 1976 ranged in size from 37 to 113 m^3. In later trials at Temora and Condobolin, silos of total volume 83 m^3 each were used. Their nominal capacity was 50 t (73 m^3) and they held aproximately 40 t grain. At Tamworth silos of 50 t capacity holding approximately 45 t grain were used.

Fumigation sheets and exposure periods

In each trial, except in 1982, fumigation of silos completely covered with nylon-reinforced polyvinyl chloride sheeting was compared with fumigation of unsealed silos. After the first trial, when separate plastic sheets covering roof and wall were joined around two silos, a silo cover, tailor-made from the same type of plastic sheeting was used. The covering was removed 24 h after dosing in trials with carbon disulphide. In the trials with phosphine the covering was removed after 10 days in 1978, 7 days in 1980.

Insects

Steel cages, containing wheat infested with rice weevil, *Sitophilus oryzae* (L.), were inserted into the grain in the fumigated silos and in an adjacent unfumigated control silo for each trial, except in 1982. Each cage contained approximately 60 g wheat in which there were approximately 600 adult *S. oryzae* in 1976, 1977 and 1978 and approximately 200 adults in 1980. The latter was a composite equal parts of cultures aged 9, 12, 16, 26, 28 33 and 36 days incubated at 25°C, 70% r.h. Stainless steel wires attached to the cages enabled them to be retrieved for examination after fumigation. The contents were sieved immediately to determine the adult mortality, then the wheat was incubated at 27°C, 55% r.h. for 8 weeks before it was examined again to determine if there had been any survival of immature insects.

Sampling

Grain samples from the surface and outlets of each silo were taken before and after fumigation and examined for insects. In 1982, results were based on more extensive grain sampling: surface samples from 5 points, sub-surface samples, obtained with a Corcoran spear probe, from 0.75, 1.5, 2.0, 3.0 and 4.0 m deep, and samples from the grain outlet of each silo. Sub-surface samples were taken from a control bin also. Each sample was approximately 500 g.

Dosing

Carbon disulphide was hand-pumped from a bucket at ground level to the top hatch where, in the first trial, it flowed onto sacks hanging from the hatch rim inside the silo. In later trials carbon disulphide was sprayed over the grain surface. A stirrup pump fitted with petrol-resistant hose was used. A shallow layer of water, covering the carbon disulphide in the bucket, reduced evaporation during pumping. Carbon disulphide is highly flammable. Precautions necessary against accidental ignition of carbon disulphide liquid or vapour include earthing the fumigant container and spray nozzle against static electricity (Cotton, 1963).

Tablets containing aluminium phosphide, each generating 1 g phosphine on exposure to moisture, were probed into the grain, using a tubular steel probe designed for that purpose. At 14 positions on the grain surface (approximately 1.2 m apart) tablets were placed at 6 levels vertically (approximately 0.6 m apart) using 85, 252 and 480 tablets to achieve fumigant dosages of 2, 6, and 12 g phosphine/t respectively. In 1982, approximately 300 tablets were used in each silo (a dosage equivalent to 6 g phosphine per tonne capacity of the bin) including 10 tablets probed into grain in the conical base of the bin through the auger chute.

Temperature and moisture

Grain temperature was measured before fumigation with a thermistor probe, moisture content was determined with a "Motomco" meter.

RESULTS AND DISCUSSION

The effectiveness of carbon disulphide or phosphine for grain fumigation was measured in 5 trials by the mortality produced in *S. oryzae* cultures exposed in cages in the grain (Table 1). S. oryzae was chosen as the test insect because the occurrence of its immature stages inside the grain kernels provides a measure of the penetration of fumigant gas. It is recognised that immature stages of *Sitophilus spp.* are most difficult to control by fumigation with phosphine (Reynolds et al., 1967). These results were supplemented by observation of the effect of fumigation on naturally occurring insect infestation of the grain.

In the trials with carbon disulphide, fumigation was effective only in covered silos and when the fumigant was distributed over the grain surface by spraying. In 1977, adult insects were controlled in 24 h fumigations with applied dosages of 0.06 L m^{-3} at Temora and 0.12 L m^{-3} at Condobolin. Few progeny developed from the caged wheat fumigated at Temora and none from that fumigated at Condobolin. The dosage was altered to suit prevailing atmospheric temperature (16-40°C at Temora, 5-33°C at Condobolin), although the mid-bulk grain temperature did not differ correspondingly. Because of insect infestation, grain in the covered silo at Condobolin (33°C) was warmer than grain in the covered silo at Temora (29°C). The natural infestation at Condobolin, mainly lesser grain borer, *Rhyzopertha dominica* F., and rust-red flour beetle, *Tribolium castaneum* Herbst, was controlled by carbon disulphide fumigation.

In trials with phosphine, fumigation was effective in both sealed and unsealed silos. This was indicated by the mortality of adult *S. oryzae* caged in the grain (Table 1) and by the incubation of the cage contents, which showed that immature stages of *S. oryzae* did not survive fumigation. Control mortality of *S. oryzae* caged in an unfumigated silo, 0.1, 1.5 and 3.0 m deep in grain, was 5% or more in 1978 but $<1\%$ in 1980. Supplementary results obtained from grain samples (Table 2) indicated effectiveness of fumigation against *R. dominica* , *T. castaneum*, long-headed flour beetle, *Latheticus oryzae* Waterhouse and flat grain beetle, *Cryptolestes sp.* in 1978 and against *T. castaneum* after fumigation, in grain samples from bins that were not covered for fumigation was probably due to re-invasion of those bins from infested grain in adjacent silos. *T. castaneum* is noted for its mobility at atmospheric temperature >25°C which prevailed then. Mean sub-surface grain temperature in the 1978 trial was 41°C in the covered silo (barley, 11.0-12.9% moisture) and 32°C in the silo fumigated without covering (wheat, 9.4-9.7% moisture). In the 1980 trial it was approximately 30°C in all silos (wheat, 10-12% moisture).

In the phosphine fumigation trial at Tamworth the results achieved by the 2 fumigant formulations were similar. *T. castaneum*, occuring throughout both silos, was a convenient indicator. Very few insects of other species occurred alive in the grain. Complete mortality of insects on the grain surface and in the centre of the grain bulk was found in both silos 7-8 days after fumigation (Table 3). Some insects were found alive then in samples from the grain outlets of both silos. They were *T. castaneum* and *S. oryzae* that may have invaded the bins recently, as supposed for similar occurrences at Condobolin. Additional samples from the grain surface of each silo, taken close to the silo wall at the 4 compass points, contained no living insects after fumigation. Corresponding samples taken before fumigation from Silo 1 contained numerous *T. castaneum* alive. Similar samples were unobtainable then from Silo 2 because it was full. Wheat removed later to make headroom for probing was held in a transportable bin as a control. *T. castaneum* remained alive in the control bin during the trial. Grain temperature 0.75-5.0 m sub-serface was 27-31°C in Silo 1 (wheat, 11.4-12.2% moisture), 26-36°C in silo 2 (wheat, 10.3-12.3% moisture).

Probing the tablets into grain was a convenient method of obtaining accurate placement of fumigant in these trials. In practice, the working headspace might not be available, and it could be necessary to add the tablets to grain as it is turned from one bin to another. An effort to distribute the fumigant formulation evenly amongst the grain may be necessary to achieve the same results as were obtained in these trials. Their is a possibility that some insects may survive fumigation in the grain because of lack of penetration of phosphine at low doses (Lindgren *et al.*, 1958).

CONCLUSION

The trials with carbon disulphide, at dosages commonly recommended for farm-stored grain, demonstrated that fumigation in unsealed bins is unreliable, and that distribution of the fumigant over the grain surface in sealable bins is desirable. The danger of using this highly flammable fumigant is minimised when fumigating grain in isolated farm silos.

Trials with phosphine demonstrated that this fumigant can be used effectively in farm silos without sealing. Even distribution of the fumigant formulation in the grain is considered necessary in this instance. The dosage used in the initial phosphine fumigation trial was slightly greater than the

Table 1. Mortality (%) of caged *Sitophilus oryzae* in fumigation of grain in farm silos at Temora and Condobolin

Trial	Depth in grain (m)	Carbon disulphide 0.06 L m^{-3} for 24 hours		Carbon disulphide 0.12 L m^{-3} for 24 hours		Phosphine 12 g t^{-1} for 10 days		Phosphine 6 g t^{-1} for 7 days	Phosphine 2 g t^{-1} for 7 days	
		Silo covered	Silo not covered	Silo covered	Silo not covered	Silo covered	Silo not covered	Silo not covered	Silo covered	Silo not covered
Temora March 1976	0.1	87	0	100	0					
	2.0			100,88	0					
	3.0	<5	100		100					
	4.5	100,100	100	100	100					
Temora April 1977	0.2	100,100	65,100,100							
	3.0	100,100	78							
	4.5	100,100								
Condobolin May 1977	0.1			100,100	100,100					
	2.0			100,100	100,100					
	4.0			100,100	100,97					
Condobolin April 1978	0.1					100,100,100	100,100,100			
	1.5					100,100,100	100,100,100			
	3.0					100	100			
Condobolin March 1980	0.1							100,100	100,100	100,100
	1.5							100,100	100,100	100,100
	3.0							100	100	100

Table 2. Adult insects alive in grain samples from farm silos, before and after phosphine fumigation at Condobolin

Sampling site	Insects	1978 trial 12 g phosphine/t SILO COVERED before	after	SILO NOT COVERED before	after	6 g phosphine/t SILO NOT COVERED before	after	1980 trial 2 g phosphine/t SILO COVERED before	after	SILO NOT COVERED before	after
Surface	*R. dominica*	140		315		1					
	T. castaneum	50		13		2	1	2			
	L. oryzae	850									
	Cryptolestes sp.	12						5			
Grain outlet A	*R. dominica*	640		132							
	T. castaneum	832		86	3	70	1	74		1	
	L. oryzae	1080									
	Cryptolestes sp.	38									
Grain outlet B	*R. dominica*	540		4							
	T. castaneum	640		7		30		18		1	
	L. oryzae	16									
	Cryptolestes sp.							8			

Table 3. Number of adult *Tribolium castaneum* alive in grain samples from farm silos fumigated with phosphine 6 g t^{-1} at Tamworth, 1982 (without covering)

Sampling depth (m)	Silo 1 Dosed 4th May with 'Gastion(R) fumigation tablets"		Silo 2 Dosed 5th May with "Phostoxin(R) DEGESCH fumigation tablets"	
	Pre-fumigation (28th April)	Post-fumigation (12th May)	Pre-fumigation (28th April)	Post-fumigation (12th May)
0	39		45	
0.75	23		6	
1.5	17		6	
2.0	15		6	
3.0	39		6	
4.0	17		7	
grain outlet	15	6	169	+
auger chute	*	8	*	21

* not sampled pre-fumigation + 3 *S. oryzae*

maximum then recommended because it seemed appropriate to use the whole contents of a tin of 480 tablets (phostoxin), once it was opened, for each silo. Now that the maximum dose recommended is 6 g phosphine/t, a pack of 300 tablets (Gastion) is an appropriate size for fumigating a 50 t silo. Results obtained with 2 g phosphine/ t indicated that there is considerable margin of efficacy when this fumigant is used at dosages currently recommended for farm-stored grain.

These fumigants play a major role in the control of grain insects on farms. With the very large number of old silos and grain sheds in use, it is impractical to restrict their use to storages that are ideal for fumigation.

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THE ADOPTION OF SILO SEALING BY WESTERN AUSTRALIAN FARMERS

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ABSTRACT

The paper describes the circumstances which led to an interest in sealed storage for the control of stored grains insect pests in Western Australia and the way in which the technique was introduced to farmers. Four groups are identified as being associated with the development of on-farm sealed stores; sealant manufactureres, silo manufacturers, contractors and farmers. The problems faced by each group in achieving a satisfactory product are outlined, together with some of the solutions including the acceptance of a static pressure test to measure the efficiency of sealing. The way this development has been adopted by Western Australian farmers is reviewed, together with the present situation and prospects for future development.

The Development of Silo Sealing in Western Australia

In the wheatbelt areas of Western Australia temperatures in the upper thirties °C are quite usual in the three months following harvest (January-March), creating favourable conditions for the development of stored grain insect pest infestations in farm silos.

In recent years the prinicpal theme in extension programmes for insect control has been to promote a high level of farm hygiene and the concept of a 'clean pipeline'. This involves the elimination, as far as possible, of storage pests entering bulk receival points from farms. Farmers are urged to pay special attention to cleaning and disinfection of all grain handling equipment (headers, augers, field bins, trucks) prior to harvest. Whilst this policy has been largely successful, the problem still remained of keeping grain stored on-farm, free of insect pests.

Only two options are presently available to Western Australian farmers for the on-farm control of insect pests of stored grains.

(a) Treatment of the grain with Malathion as a protectant. No other insecticide is registered for this purpose in Western Australia.

(b) Fumigation - usually using phosphine gas generating tablets (Phostoxin, Gastoxin, Detecia gas-XB).

Increasing resistance to Malathion by some insects, notably *Rhyzopertha dominica* and *Tribolium castaneum*, is becoming a problem. However, Malathion is currently the only protectant registered under the Western Australian Health Act (pesticide regulations) for use on grain stored on the farm. This restriction has been imposed in an effort to delay for as long as possible the development of resistance to other pesticides that may be used elsewhere for the control of stored grain insect pests. Fenitrothion and other insecticides used elsewhere are reserved for use only by the Grain Handling Authorities in Western Australia at the request of the Grain Weevil Liaison Committee. This body includes representatives from the Western Australian Department of Agriculture, Co-operative Bulk Handling, Agriculture Protection Board, Australian Wheat Board, Primary Industry Association (formerly the Farmers Union) and the Pastoralists and Graziers Association. This policy appears to have been justified, although Co-operative Bulk Handling (CBH) are now finding some instances of resistance to Fenitrothion, especially in *Rhyzopertha dominica* and *Oryzaephilus surinamensis*. As farmers are permitted to treat empty grain storage areas and grain handling machinery with fenitrothion, this resistance may have developed initially on-farm, however CBH do not rule out the possibility of this resistance becoming established in bulk handling operations off farms. Nevertheless since these restrictions on the use of alternative insecticides are likely to continue in Western Australia, farmers are left with fumigation as the only effective protection against insect infestations in stored grains. The problem facing farmers is that the average farm silo is not gastight and is completely incapable of maintaining fumigants at an adequate concentration for a sufficiently long time for total insect kill. The problem would be solved if the silo were made gastight.

A leading role in research and development of sealed and controlled atmosphere storage was undertaken by Dr Banks and his team at CSIRO. The results obtained from their work led to studies in the application of their techniques in Western Australia.

In the period 1972-77 the Western Australian Department of Agriculture conducted trials with grain held in underground stores. In one trial at Salmon Gums, 120 tonnes of grain were stored in a plastic lined pit. Grain temperatures and moisture were maintained at a satisfactory level for three years and the low oxygen concentrations obtained early in the trial were maintained. Further trials were also held at the Merredin Research Station. Whilst the trials showed that grain could be stored in a sealed condition, it was considered this method was not practical for farmers because of the difficulty in handling grain into and out of underground stores. CBH also conducted trials. Some of their bulk storage cells were sealed in 1979 which led to the development of materials suitable for sealing both bulk and on-farm silos.

In 1977 the responsibility for enforcing the control of stored grain insect pests on farms was transferred from the Western Australian Department of Agriculture to the Agriculture Protection Board (APB), although the Department continued with research and resistance testing.

The APB has over 100 officers in the agricultural areas of Western Australia. Staff visit all landholders on a regular basis; carry out inspections for declared plants and animals; advise on their control and, if necessary, enforce and carry out control measures. This activity permits a close monitoring of conditions on farms. However, this responsibility for storage insect pests was quite new to the APB, whose only previous involvement with insects of any species had been in the control of small plague grasshoppers and Australian plague locusts. An intensive training course for APB officers was undertaken and new control strategies were evaluated.

Under the Agriculture and Related Resources Protection Act, the APB 'declared' eleven species of stored grain pests which makes the landholder, or person leasing the land on which these insects are found, legally responsible for carrying out control operations.

The species declared were:

Lesser grain borer	(*Rhyzopertha dominica*)
Rust-red flour beetle	(*Tribolium castaneum*)
Confused flour beetle	(*Tribolium confusum*)
Sawtooth grain beetle	(*Oryzaephilus surinamensis*)
Flat grain beetle	(*Cryptolestes* spp.)
Rice weevil	(*Sitophilus oryzae*)
Granary weevil	(*Sitophilus granarius*)
Indian meal moth	(*Plodia interpunctella*)
Angoumois grain moth	(*Sitotroga cerealella*)
Warehouse moth	(*Ephestia* spp.)
Warehouse beetle	(*Trogoderma variabile*)

In addition to farm visits for inspections and to monitor implementation of control programmes, a series of promotional ventures was carried out by the APB. One was a competition to find the farmer who had the most effective level of farm hygiene. Another was for a silo design including such features as: ease of emptying, cleaning and fumigation; efficiency of fumigant dispersal; airtightness; elimination of grain residues collection areas. Prizes were donated for these competitions by various organisations with an interest in reducing levels of insect infestation and in co-ordinating control strategies over the entire grain industry. The results of the initial sealing trials carried out by CBH were combined with improved farm silo designs originating in part from the APB competition. In consequence, the on-farm sealed silo was launched.

Promotion of Farm Silo Sealing

In 1981 CBH invited all silo manufacturers to submit a silo or silos for an exhibition or non-competitive demonstration at a site near Perth. The silos were sealed using Wastolan, Envelon and Formrok, products previously tried on CBH bulk storages. Because it was felt many farmers were unable to visit the display APB organised a silo sealing 'roadshow' to visit 24 centres in the northern and central wheatbelt and hold field days on selected farms.

This roadshow consisted of 3 demonstration silos mounted on trucks or trailers; (a) a normal 300 bushel (6 tonnes) silo sealed with Formrok; (b) a silo sealed during manufacture with Envelon, constructed so it could be opened and the internal sealing inspected; (Fig 1)(c) a small model silo sealed externally with Wastolan.

The format of this roadshow included:

I. An introductory talk by the APB officer on the reasons for sealing silos, emphasising the need for total insect control to reduce grain damage on farms and to protect export markets, problems of Malathion resistance, reduction in insect control costs through fumigation in sealed silos.

II. A demonstration of the inability of a conventional unsealed farm silo to retain fumigant by placing a coloured smoke bomb in the silo. (Fig 2)

III. Presentation of a film illustrating the practical methods of sealing silos.

IV. Practical demonstrations of the application of silo sealing materials. Agents of the companies supplying the products were invited to demonstrate them, but it was generally carried out by an APB officer. (Figs 3 & 4)

V. To conclude there was a quiz on aspects of silo sealing. At the end of the tour the respondent with the most correct answers received a prize of sufficient sealant for a 56 tonne (2,000 bushel) silo.

The roadshow was supported by wide publicity and proved so popular the tour was extended to cover the entire agricultural area, including over 40 venues, between July and November 1981. The demonstrations were attended by more than 3,000 people.

In addition to on-farm demonstrations, silo sealing was promoted at country shows and machinery field days throughout the grain growing areas. The APB also arranged for the sealing of one silo on selected Department of Agriculture research stations. Sealing costs were met by the Department of

Agriculture whilst the APB covered the contractors travel and accommodation costs. The purpose of this exercise was three-fold:

(a) To give all APB officers the opportunity to see and familiarise themselves with the complete sealing of a farm silo.

(b) To give interested farmers the opportunity to see the operation being carried out.

(c) It was hoped that research stations having a sealed silo would encourage more farmers to seal their own silos.

The promotion of silo sealing by APB and CBH generated a great deal of interest in the farming community, which in turn stimulated activity amongst:

I. Sealant manufacturers;

II. Silo manufacturers;

III. Contractors offering an on-farm sealing service;

IV. Application of sealants by farmers.

Sealant Manufacturers

The development of sealants suitable for use on on-farm silos resulted largely from work on CBH bulk stores. From these it was evident sealing materials had to meet minimum specifications:

(a) Flexible, to accommodate at least 200% elongation without breaking and to retain this flexibility for a number of years;

(b) Adhesion to both metal and concrete surfaces;

(c) Non-toxic when cured and not to contaminate foodstuffs;

(d) Not to break down in sunlight if used on the outside of a silo and to resist abrasion from grain if used internally;

(e) Not react with, or break down in the presence of, any fumigants or insecticides used in the control of stored grain insect pests;

(f) May be applied either as a spray or by brushing without any special skills or equipment.

Initially three products met these requirements:

ENVELON - produced by Dominion Plastic Industries of Melbourne and marketed in Western Australia by Liquid Membrane Supplies of 7 Malcolm Road, Maddington 6109.

FORMROK - produced by Hitchins Australia and distributed in Western Australia by Unitex Coatings of 20 Rio Street, Bayswater 6053.

WASTOLAN - produced by VAT in Hamburg, West Germany and imported and distributed by Woodkon Pty Ltd. of 140 Great Northern Highway, Middle Swan 6056.

During the last year a further two products have been developed and marketed in Western Australia. These are:

ACRONYL and ACROLYNE - produced by Rochelle Chemicals, PO Box 42, Wembley 6014.

SILOFLEX - produced by Crommelin Chemicals, 72 Division Street, Welshpool 6106.

Of these, Wastolan and Siloflex are basically similar products being water based modified acrylic emulsion formulations. Acronyl is also an acrylic but is slightly different in that it is solvent based but water soluble. Acrolyne is an acrylic-styrene membrane that is also used to provide an overall reflective white coat when using Acronyl to seal the joints. Envelon is an organic solvent based polymeric vinyl and Formrok is a two part urethane-elastamer which requires mixing in correct proportions immediately prior to using.

All are excellent for sealing on-farm silos and the decision on which material to use is a matter of price, convenience and personal choice. In general the costs of all products would be around A$1.00 per linear metre of joint covered.

The advantages of the acrylic based materials is that they are completely safe to use, there are no toxic fumes or fire risks when applying them. Daily cleaning of equipment is easy as water only is required (a thorough periodic cleaning with solvents is necessary however). A disadvantage is that in cold, damp or high humidity conditions these products can take some hours or even days to cure. A 2mm thickness of these materials will cure in less than 45 minutes when temperatures are not less than 20^{o}C at 30% relative humidity. At higher temperatures drying time is reduced to a few minutes.

Envelon, which is organic solvent based, dries within two minutes of application under all conditions. A disadvantage is that when used in a limited space it produces toxic and inflammable fumes. An air-wash mask must be used when applying Envelon in a confined space. In general Envelon tends to be used more by the manufacturers producing sealed silos because of its quick drying properties making it suited to production line techniques. Acronyl, Wastolan and Siloflex are the popular choice of contractors because of the ease of application and enhanced safety factor. However this is not an inflexible rule since some large silo manufacturers use acrylics and some contractors use Envelon, especially in the winter months.

Formrok is a product with maximum adhesion and elasticity and forms an excellent seal when used on farm silos. However, as this is a specialised two part product usually needing to be applied by trained operators, it has not as yet obtained an appreciable share of the Western Australian farm silo market.

Silo Manufacturers

In Western Australia over 95% of the silo market is supplied by factory produced silos, ranging in capacity from 8 to 60 tonnes (300 to 2,250 bushels). Few small prefabricated silos (less than 64 tonnes capacity) are sold and some specialist farmers, such as large scale pig producers, use much larger silos up to 1,200 tonnes (45,000 bushel) capacity.

The APB competition in 1979 for a silo that made pest control easier was won by Walker Engineering. Walkers further developed their original design and in 1980 became the first manufacturer in Western Australia to offer a range of sealed silos. However, less than 3% of their production for 1980 was of sealed silos. Following the CBH exhibition at Midland and during the APB roadshow, other manufacturers changed their production designs and added sealed silos to their range, including Moylan's of Kellerberrin, Becker's of Wyalkatchem, Stearne's of Esperance and Bird's of Popanyinning. It is interesting that these manufacturers were in the rural areas of Western Australia. It was not until 1982 that a metropolitan manufacturer offered a sealed silo.

One of the early problems of promoting sealed silos was the lack of support from some agents and distributors because they had considerable stocks of unsealed silos on hand. Many changes had to be made to silos to obtain a satisfactory level of seal; the design of inlets and outlets to enable silos to be easily resealed on closure; modifications to the construction of the roof; the joint areas between the roof and wall and the wall and the bottom cone.

The sealing of vertical and horizontal joints is usually carried out by spraying the sealant directly over the joint and for approximately 30mm on each side. A primer may first be applied to provide good adhesion of the sealant, particularly newly galvanised surfaces to which sealants do not usually adhere. Where there maybe movement or there are gaps between the metal sheets, tape or filler must first be applied to act as a filler and backing for the sealant and allow movement or stress to be spread over a greater area.

Not all manufacturers seal their joints in this way. Birds of Popanyinning use a silicone sealant (Selleys 780), a thin band of silicone is run between the metal sheets of the wall prior to riveting them together. Rivetting compresses and spreads the sealant between the joints to give an effective seal.

Many manufacturers have changed the design of the roof from a number of individual panels to a one piece welded unit, (in larger capacity silos, this type of roof may require additional strengthening), which avoids the need for taping or filling and sealing. Boyd Metal Industries of Welshpool produce a model with a glassfibre roof which entirely eliminates the sealing of roof joints.

One major problem is that of making all inlets and outlets easily resealable. In the early days of sealing it was suggested that these could be covered with tape, painted over with the sealant to seal the silo and this would be cut away to open it again. This was not a practical option. Re-opening was extremely difficult due to the strength and adhesion of the sealant which also built up around the inlets and outlets after several applications. In many cases farmers simply did not get around to re-taping and re-sealing these areas. The problem with sealing the roof entry points and hatch covers was solved by the use of spun metal discs pulled down onto rubber faced collars by means of springs or levers (Fig 5). Alternatively one manufacturer fits his sealing lids into the inside of the silo and these are pulled up tight to the opening by means of a clamp or screw.

It now remains a problem to find a suitable rubber material to form the seal round the hatches which will not deteriorate in the high temperatures of Western Australia summer. Attempts to use polyurethane screw down spray tank lids were not entirely successful as these tended to warp and become brittle after exposure.

The designs of silo discharge outlets show considerable variation from simple sliding gate valves to butterfly valves, and the problem of fitting seals is, in some instances, very difficult and complex. (Figs 6,7 & 8) Some manufacturers use rigid rubber backed plates that can be pulled tight onto the opening by springs, screws or clamps, others a hinged flap with an overcentre lock to hold the plate tight. A cam type lever or clamp is used to hold a rubber backed slide against the outlet. In most cases the edge of the outlet is ground to a sharp angle to prevent any grain being trapped between the outlet and the rubber of the sealing plate.

Another alternative method of sealing the bottom outlet is by a totally enclosed boot that has an access door, also rubber backed, that can be held tight against the outlet by springs or clamps. Some boots are fitted with a slide with a removable handle which, because it is enclosed within the boot the slide does not have to form a seal.

When introducing sealed silos manufacturers also had quality and marketing problems. Farmers had to be pursuaded to pay the additional costs of a sealed silo, about 10-15% or A$300 for a 56 tonne (2,000 bushel) capacity silo. However farmers quickly realised pest control costs were reduced in sealed silos by up to 90% for fumigation and the grain was stored in optimum condition.

There was also concern over testing the efficiency of the seal. Together CBH and APB have promoted the idea of an air pressure decay test for sealed silos. The test standard now accepted is that an inflation pressure of 250 pascals (1000 pascals is equivalent to the pressure of a column of water 1 metre high. 250 pascals is equivalent to a water gauge of 25mm) should not fall to less than 125 pascals in five minutes for new silos (or to fall to 125 pascals in 3 minutes for silos sealed on farms by contractors). This variation is because of the greater problems in sealing silos that have been used. It is difficult to carry out this test reliably because of climatic conditions such as wind, temperature and especially sunlight. The usual procedure is for the manufacturer to test the silo in the factory and for him to give an on-farm test if requested by the farmer. There was some concern that the seal would be damaged during transportation to the farm, but to date there are no known instances of breakdown of the seal during the road journey.

There is also a problem in meeting the CBH/APB recommendation that sealed silos be given a reflective coat during transport. It has now been left to the individual manufacturers whether or not they apply this reflective white coat. Some apply the coat at an extra charge and touch up any transit damage on arrival, others recommend to the farmer that he applies a coat of white acrylic paint after two years when the surface of the galvanising has weathered. Although not nearly so efficient as white, a newly galvanised surface does have an acceptable reflective level. One manufacturer is experimenting with a white colourbond steel sheeting in an attempt to overcome this problem.

Of the twelve silo manufacturers in Western Australia, only two do not include a sealed model in their current range and one is currently working on a sealed silo for release next year. Sealed silos now account for over 60% of the total market which, in under two years since they first became available, is beyond all expectations. Some manufacturers are producing only sealed silos and others report sales are 60-90% of total production.

Contractor On-farm Sealing

A number of contractors have become established to offer a service to seal existing silos on farms. In the main these contractors were already in the agricultural field in pest control, fencing, spraying, painting or building and the sealing of silos is an extension of these activities.

Initially contractors faced many problems in a new industry and using new materials. These were generally similar to those faced by the silo manufacturers - choice of sealant, application techniques, curing times, replacement or sealing of inlets and outlets, pressure testing and an equitable and acceptable price structure. Many of these problems were discussed with manufacturers and a common solution was found. For instance, many contractors now simply remove all the old inlets and outlets and replace them with new ones supplied by the silo manufacturers. There were some cases of fraudulent practice as some contractors saw substantial gains to be made. In one case a contractor simply painted the silo white, fitted a pressure relief valve and charged for 'sealing'.

In order to overcome these problems and to create an opportunity for the exchange of ideas between all those involved in this field, the APB organised a conference early in 1982.

One outcome of this conference was the formation of the Silo Sealing Association of Western Australia in December 1982. Its aims are to act as a forum, to set and maintain standards and co-ordinate and promote activities in connection with on-farm silo sealing. It has recently adopted a procedure for dealing with disputes on workmanship or any complaints laid by farmers who have had their silo sealed by members of the Association. Contractors are able to take up Business Membership of the Association and nearly all contractors are now members. Associate Membership is offered to suppliers of goods and services used by the industry, such as sealant materials, and to manufacturers producing sealed silos. The Association is now a sectional association of the Confederation of West Australian Industry.

It can be said that solutions can be found to all problems of sealing and contractors can effectively seal any type, model or size of grain silo. By the end of 1982 an estimated 1000 on-farm silos had been sealed by contractors. Approximate charges for sealing an average 56 tonne (2,000 bushel) capacity silo is between A$750 - A$1,000, including all materials, labour, cement floor treatment if applicable, replacement of all inlets and outlets, pressure relief valve (Fig 9) and application of a reflective white coat.

A contractors' specification for sealing on-farm silos should include:

I. Surface preparation. One of the most important and time consuming operations of silo sealing, essential to ensure good adhesion of the sealants and a reliable seal. Strict attention to cleaning surfaces applies equally for sealing applied internally or externally.

II. Replacement of hatches and outlets usually replaced with new re-sealable units obtained from silo manufacturers. Fittings for the pressure relief valve are added, usually at the top of the silo adjacent to the ladder. A car tyre valve is often fitted into the wall or base cone of the silo to enable the silo to be pressure tested when sealing has been completed. Farmers can also check the seal at regular intervals. With most silos it is necessary to strengthen certain areas, individual roof panels, the eaves and around inlets and outlets usually by rivetting extra metal strips or bands over the joints to hold them together.

III. Filling or taping of the longer gaps using a suitable filler. Alternatively cloth or plastic tape may be used and many contractors successfully use good quality masking tape. Most contractors now tend to use fillers in preference to tape as it is quicker to apply, is equally effective and equivalent in price.

IV. Application of the sealant to all joins in the silo, whether welded or bolted, to a suitable width and thickness. (For acrylics this would be not less than 1mm in thickness and extending at least 30mm each side of the joint). The choice between internal and external sealing is one of personal preference, both will give satisfactory seal if carried out properly. Most contractors now seal externally because it allows them to utilise a greater degree or mechanisation such as the use of self-propelled hydraulic ladders of 'cherry pickers'. It is usually easier and more pleasant to work on the outside of a silo, especially in summer, and it allows greater continuity of work since silos can be sealed externally even when they are full. Most contractors using the water based modified acrylics apply these with an airless spray operating at a pressure of around 7,000 kPa (or 1,000 psi), but some of the smaller contractors apply the sealant with an ordinary paint brush.

V. Application of a reflective acrylic white paint coating to all external surfaces of the silo.

VI. Pressure testing when sealing has been completed. Even the most experienced contractor has a success rate of less than 10% at the first test so any silo not tested is probably not sealed. The minimum acceptable standard for existing silos sealed on-farms is that a pressure of 250 pascals should not fall to less than 125 pascals in three minutes. (This is less than the five minutes for new silos because of the additional problems involved in sealing these structures). As with factory built silos, this test must only be carried out when climatic conditions are stable. It is important not to exceed a pressure of 300 pascals in the silo to avoid possible damage to the sealant or structure.

Farmer Application

The degree of attention to detail and the dedication required to seal a farm silo to the recommended standard is daunting to most farmers. 50 farmers only are believed to have completed the job and saved about A$300-A$400 per silo. Some farmers are known to have been unable to complete the sealing to a satisfactory standard and a few even employed contractors to finish the job.

A recent development is the sale of complete silo sealing kits by members of the Silo Sealing Association to farmers who want to carry out the sealing work themselves. The kits contain the correct amount of sealant for the farmers particular type of silo, together will all necessary fittings, outlets, inlets, filler, tape, etc. The member will also give advice on sealing technqiues.

Trends in Silo Sealing for On-farm Use

The acceptance by farmers of the concept of sealed storage for efficient on-farm fumigation has exceeded all expectations. Sales of new sealed silos are well ahead of unsealed models and prior to November 1982 it was impossible to arrange for any contractor to carry out on-farm sealing of silos for at least four months because all had full order books. However, there are some areas of Western Australia with only a few farms with sealed silos. The reasons for this are not fully understood. It may be due to the costs of having an existing silo sealed or the extra cost of a sealed model together with a lack of understanding of problems of stored grain insect control and the financial benefits of sealed storage. There were initial difficulties with both manufactured and contractor sealed silos due to lack of experience in new techniques and these difficulties may be used to discredit sealed silos by agents and manufacturers still selling unsealed models. The most likely cause may be the uneven distribution of contractors throughout the State so that in some areas contractors are unavailable, or travelling and accommodation charges make the cost prohibitive.

It is now a major concern that some silo manufacturers are advertising "semi-sealed" silos. These are not tested to APB/CBH recommendations, often the outlets and inlets are not re-sealable and unproven materials have been used to cover joints. One manufacturer used car underbody sealer. Unfortunately, there is little than can be done to prevent this practice although officers from the APB and Silo Sealing Association have visited these firms in an attempt to get them to voluntarily come into line. Our fear is that if farmers use recommended dosage rates of fumigant for sealed silos in a so-called "semi-sealed" silo, only a low level of control will be achieved and farmers' faith in the efficacy of sealed storage will weaken.

It is claimed that grain is damaged due to moisture migration within the silo. Whilst it is accepted that this phenomenon may be accentuated in a sealed silo, if the grain is at an acceptable moisture content (less than 12%) migration is unlikely to occur. However, farmers often store grain, rejected at the CBH receival point because of high moisture content, in their on-farm silo. This practice makes migration inevitable with the risk of associated grain spoilage. It is in this instance that sealed storage will be blamed. The solution appears to be in educating farmers in the dangers of storing damp grain, particularly in a sealed silo.

Some farmers believe that because grain is stored in sealed silos it is safe from insect attack, and no other control measures are needed. The extension programme has now to return to the elements of stored grain insect control to re-stress the importance of the clean pipeline concept, to increase its efforts to pursuade farmers to pay more attention to farm hygiene and to emphasise that silo sealing is only part of good farm hygiene, not a substitute for it.

In the past two years the picture of grain storage in Western Australia has changed dramatically. For the first time there appears to be an opportunity to maintain stored grain free of insect pests. Looking to the future it seems likely there will be wider farmer acceptance of sealed storage. The sealing, in 1982, of a further 19 country bulk stores by CBH has illustrated dramatically to farmers that organisation's commitment to these techniques and this has led to an increase in demand for sealed silos in these areas.

Farmers have accepted sealed storage to enable them to undertake efficient phosphine fumigation. If, in the future, it becomes necessary, because of increasing insect resistance or public health requirements, to change to using inert gases and controlled atmosphere storage, the Western Australian farmers who have previously adopted sealed storage will be able easily to adopt these new techniques.

I am convinced that the development of sealed storage has been adopted readily, not so much because Western Australian farmers are vitally concerned with the problem and are by nature progressive, but because this technique has been researched, developed and promoted by all those in the grain industry, farmers, farmer organisations, Department of Agriculture, Co-operative Bulk Handling and Agriculture Protection Board. These bodies have worked together to solve a common problem, unacceptable insect damage in stored grain.

ACKNOWLEDGEMENTS

I should like to acknowledge all those who have assisted me in the preparation of this paper; Mr Ken Carpenter and Mr Fred Swithenbank of the Silo Sealing Association; the sealant manufacturers and agents; silo manufacturers producing sealed silos and my colleagues at the Agriculture Protection Board, Mr Mike Sexton for his advice, Mr Frank Smith for his assistance in composition and Mr Jeremy Dixon for the artwork.

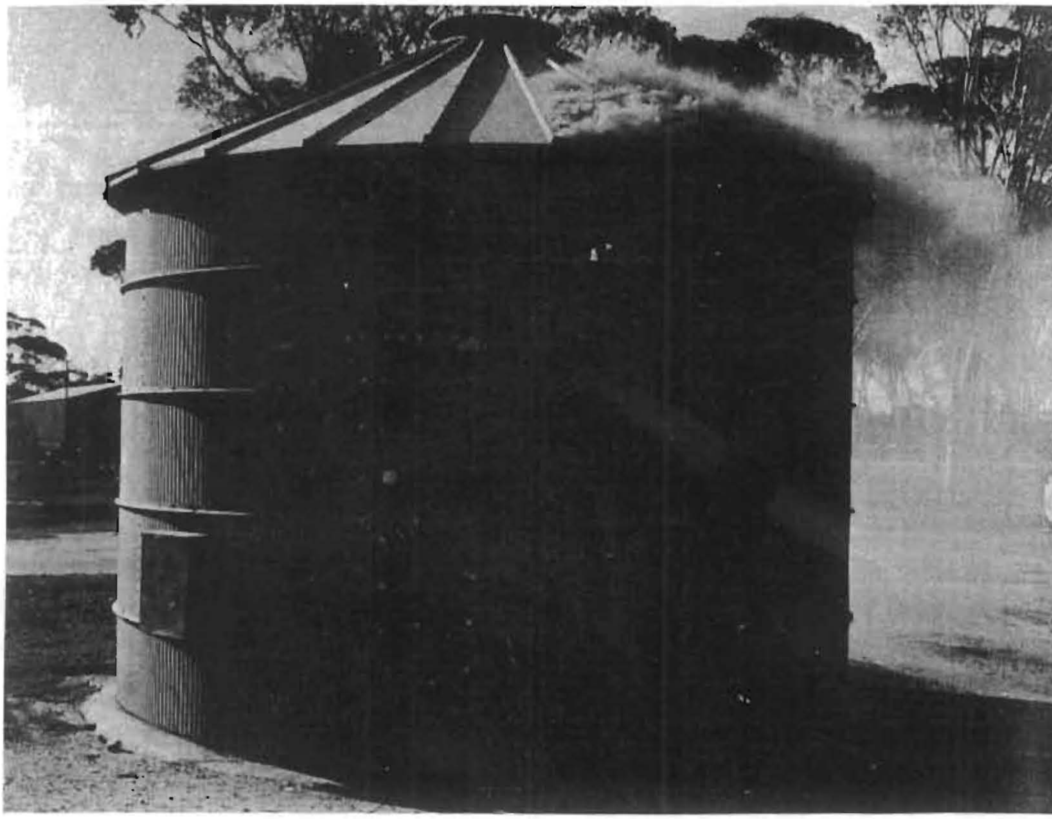


Fig. 2 Smoke escaping from joins of an unsealed farm silo.

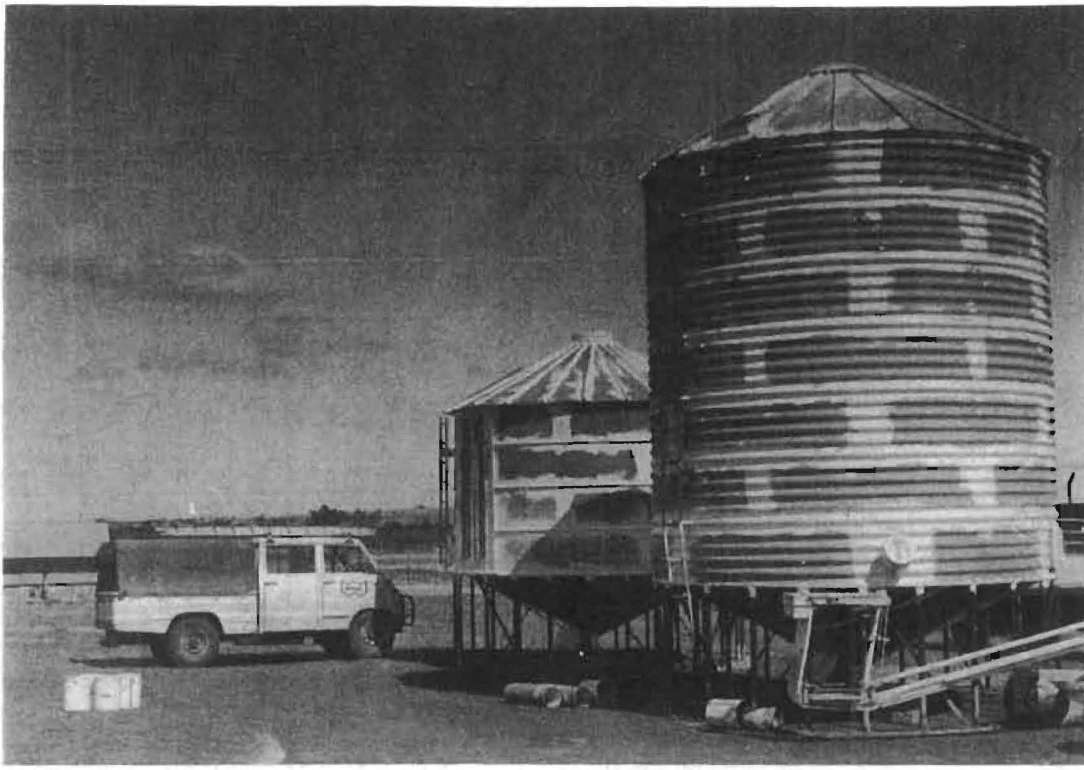


Fig. 3 Sealing of joins in on-site farm silos.

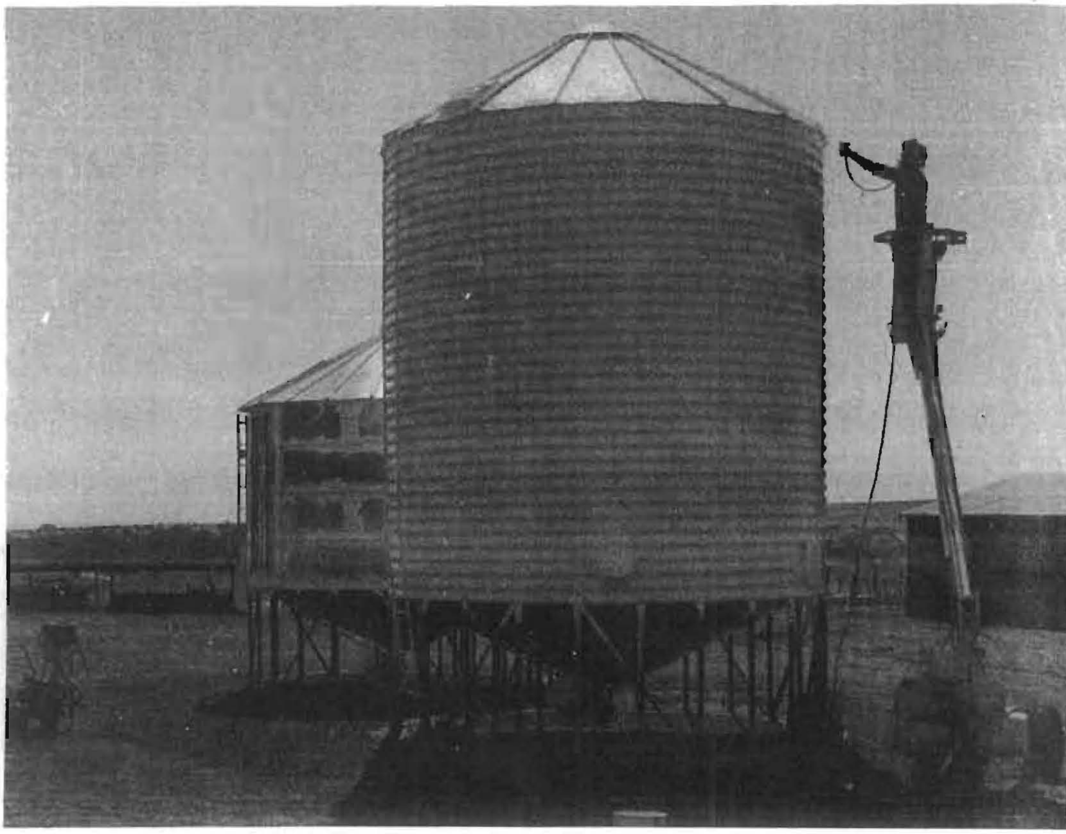
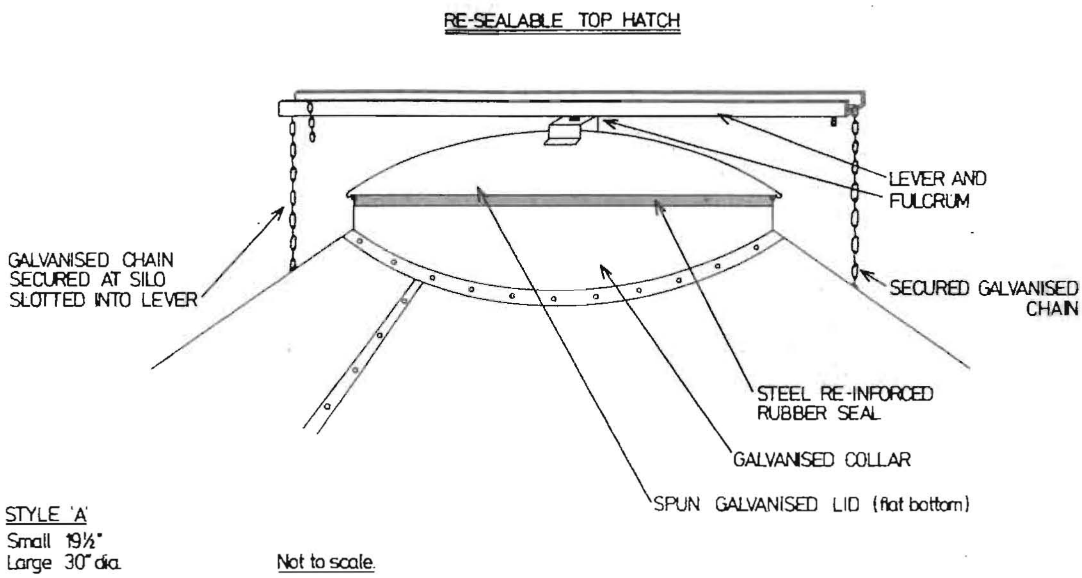


Fig. 4 Application of reflective coat to sealed silos.



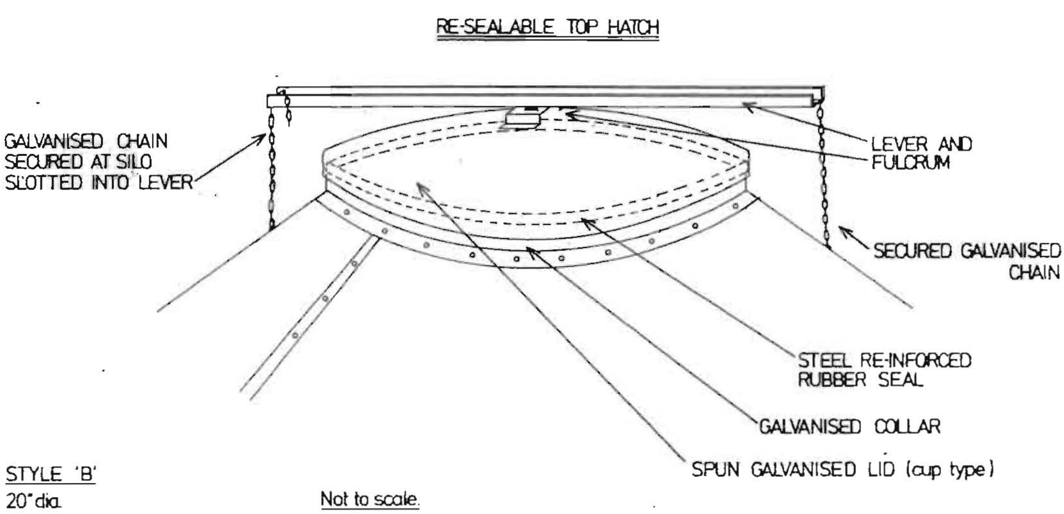
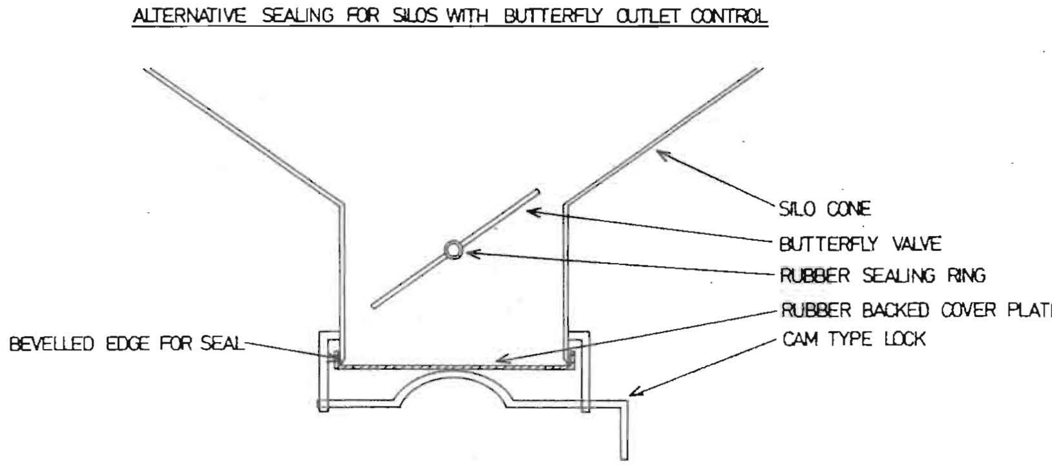


Fig. 5 Designs for sealable top hatches for farm bins.



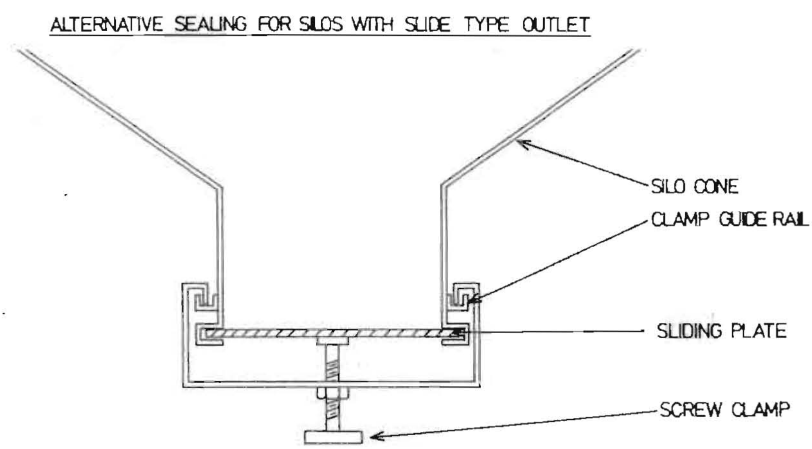


Fig. 6 Sealing systems for outloading chutes for farm bins.

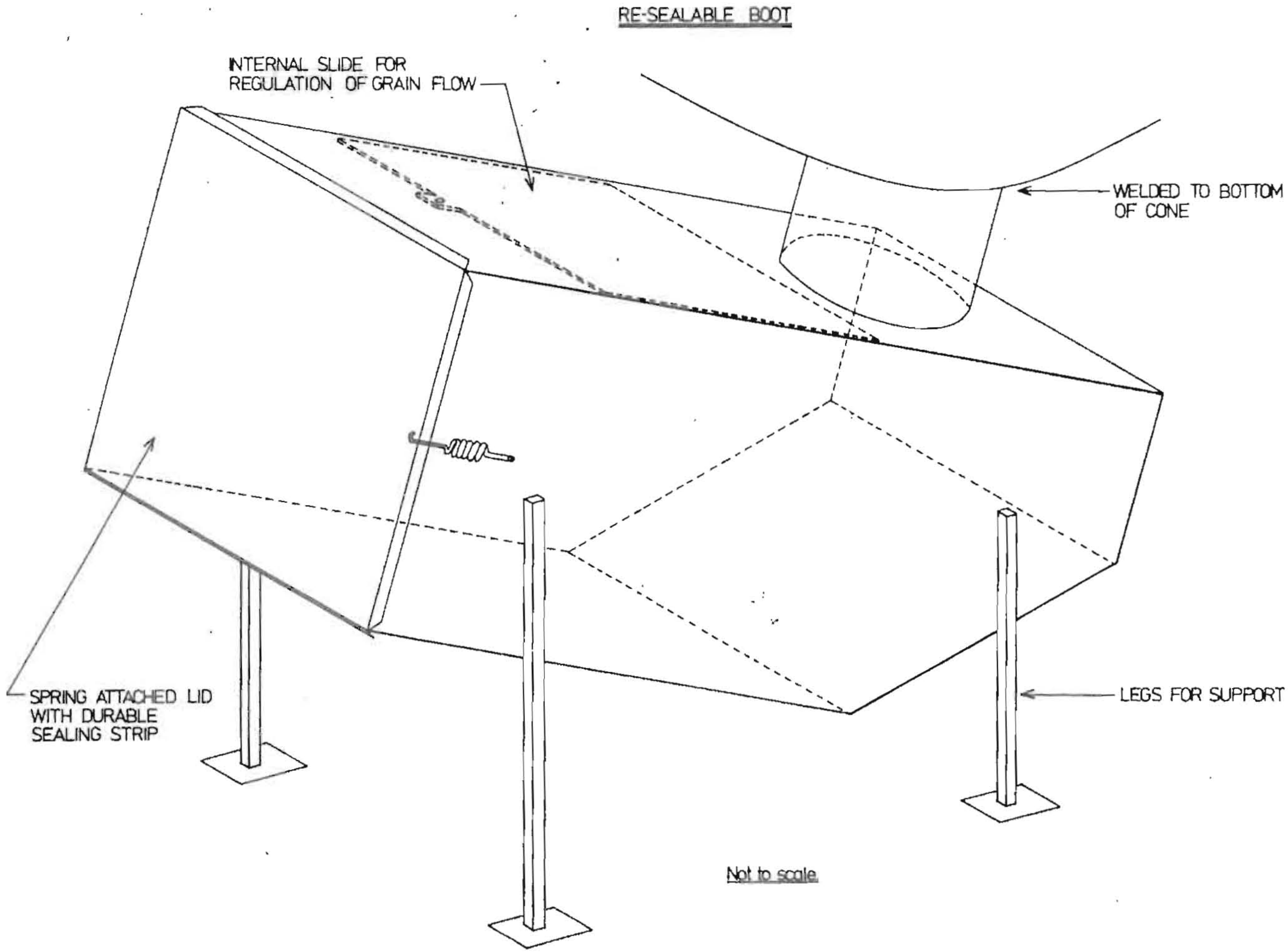


Fig. 7 Design for a resealable auger boot.

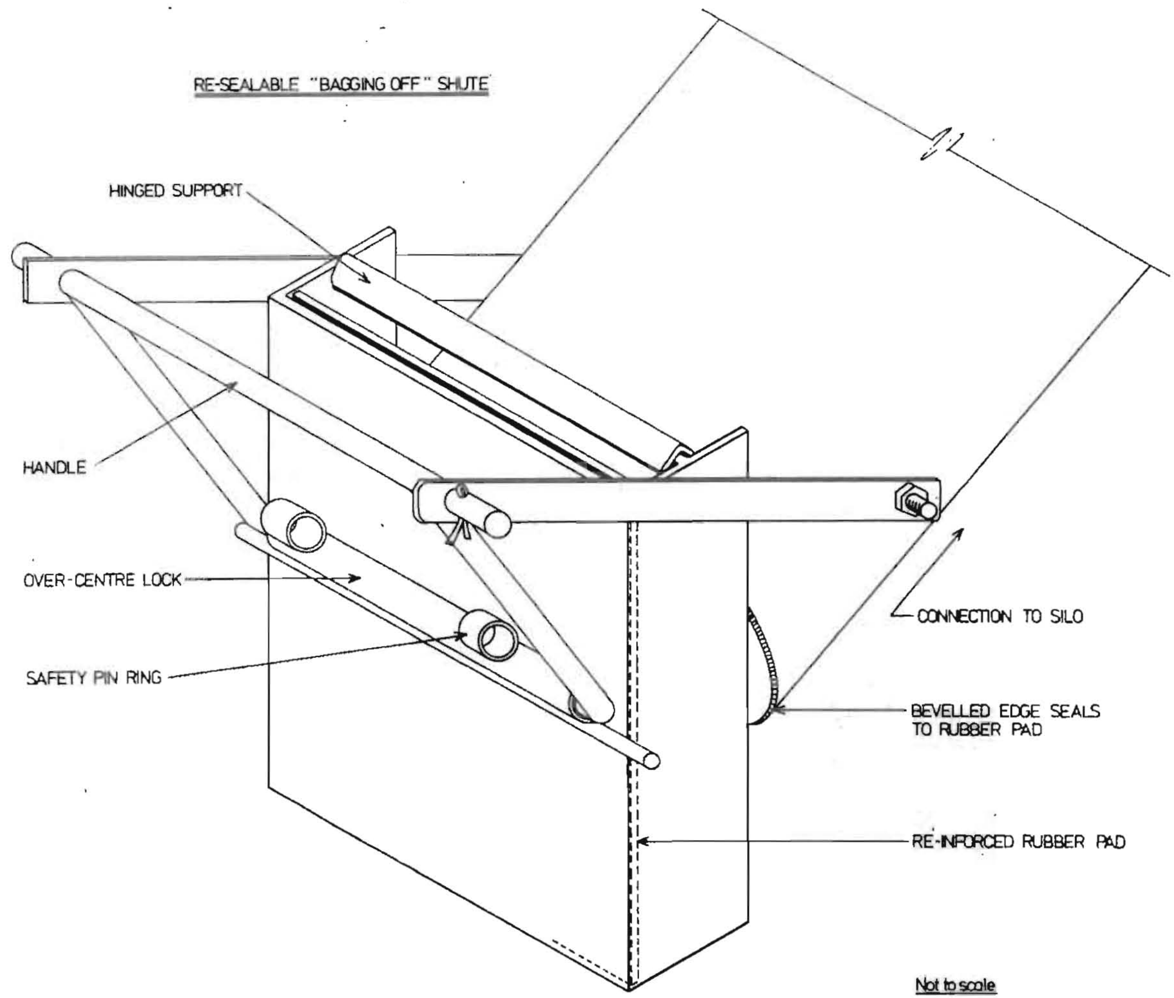


Fig. 8 Design for a sealable bagging-off chute for a farm bin.

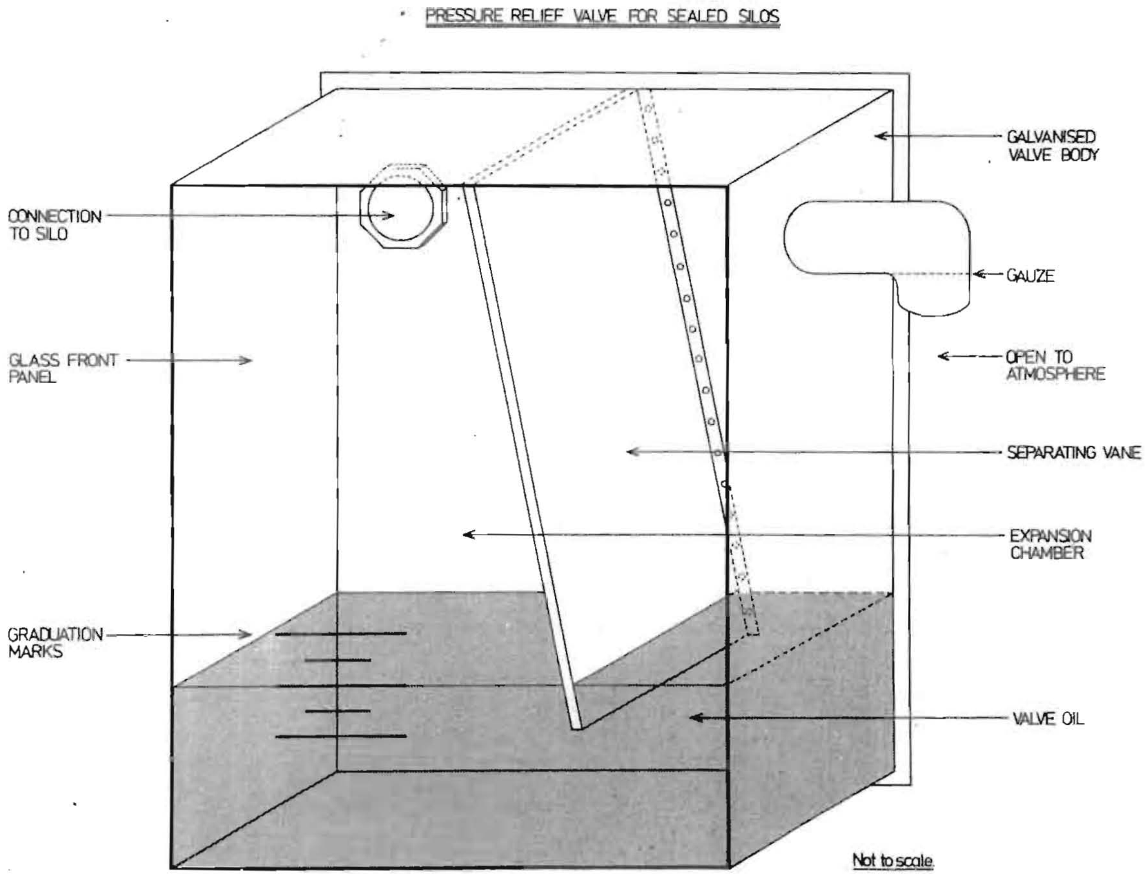


Fig. 9 Design of an oil-filled pressure relief valve for a farm bin.

FUMIGATION OF BULK WHEAT IN CONCRETE SILOS IN BANGLADESH USING ALUMINIUM PHOSPHIDE PREPARATIONS

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Abstract: Trials were carried out to determine the distribution of phosphine and *Ct*-products achieved in concrete wheat storage cells when fumigated with aluminium phosphide preparations using various application patterns. With aluminium phosphide tablets added to the grain stream on loading in batches at half-hourly intervals, adequate *Ct*-products were achieved throughout the grain bulk after 7 days, but at some points no phosphine was observed until 1.5 days after application. With the total dosage of tablets ($\equiv 2gPH_3\ t^{-1}$) added to the grain surface when the bin was half-full, with the remainder of the load added after dosing, the gas evolved dispersed downward only, with no phosphine observed above the band of tablets. With the complete dosage added to the grain surface, the phosphine dispersed throughout the bulk but inadequate *Ct*-products for insect control were obtained after 6 days at 30 m depth and it appeared unlikely that a satisfactory *Ct*-product would be attained even after 10 days. In all of the trials substantial leakage of phosphine into adjacent bins was noted.

With the threat to phosphine fumigation posed by the resistant insect strains present in Bangladesh, it is important that fumigations be carried out effectively in silos. It is concluded from these trials that a dosage of $2gPH_3\ m^{-3}$ (i.e. 3 tablets per tonne) is satisfactory but a 7 day exposure period is required to ensure adequate gas distribution. The fumigant preparation should be added so that it is distributed at least at several levels in the bulk. Application as a single layer, either at the surface or in the bulk, is not a satisfactory method.

1. INTRODUCTION

The four Ministry of Food silo complexes were constructed under the first IDA Grain Storage Project during 1968-70. The complexes are of 100,000, 2 x 50,000 and 25,000 tonne capacity and are based on multiples of 750 tonne circular bins, 450 tonne star bins and 175 tonne semi-circular bins. The silos were constructed using the conventional slip-form technique. Recent reports from Australia (D. Ellis, personal communication) assert that concrete silos of similar vintage and identical construction in that country, particularly in coastal areas, have physically deteriorated to a considerable extent and can no longer be considered as suitable for fumigation processes.

The Bangladesh silos were equipped at the time of construction with automatic dispensers for aluminium phosphide pellets, but these were never used. Fumigation of imported wheat has, however, been carried out on a routine, if somewhat *ad hoc,* basis over the years using hand-dispensed aluminium phosphide tablets added to the grain stream. However these procedures do not appear to have been effective. Very high insect populations (in excess of 2,000 per kg) were present in residues from silo hopper bottoms in Chittagong and observations on wheat stocks issued from silos to other storage depots in many parts of the country suggested that infested wheat was not uncommonly being despatched from silos.

In view of the foregoing, it was decided to monitor a series of fumigations in order to assess the technical efficiency of the procedures recommended and to determine whether fumigation of grain in silo bins with phosphine was any longer an appropriate approach. The well-established phenomenon of phosphine resistence in Bangladesh would obviously have an influence on this determination. This paper gives details of five experiments undertaken to study the distribution and retention of phosphine in silos.

2. TRIALS

2.1 Trial 1

This trial was carried out to determine to what degree phosphine concentrations could be maintained in an empty concrete silo bin and to determine whether leakage into adjacent bins was significant.

2.1.1 *Methods:* A 750-tonne empty round bin at the Narayanganj silo complex, some 20 km south-east of Dhaka, was selected for this trial. The bin under test, No. 108, was situated at the extreme north-west corner of the complex which is aligned roughly on a N/S axis. This bin had some 60% of its external circumference permanently exposed to the elements and the moderate, prevailing NNW wind. The internal dimensions of the bin were: height 35 m, diameter 6 m, volume 1,000 m^3.

Grain could be loaded into the bin from a belt conveyor via a 65 x 65 cm hatch in the concrete floor over the bins and loaded out via a conventional steel hopper at the base of the silo, leading into a delivery chute with chain operated slide control. A 35 x 35 cm inspection and access hatch was cut into the bottom hopper. A cowl-type ventilator was present on the flat exposed cap of the bin. Two 20 x 20 cm ventilation ports led into the two adjoining star bins at the very top of the bin wall (Fig. 1).

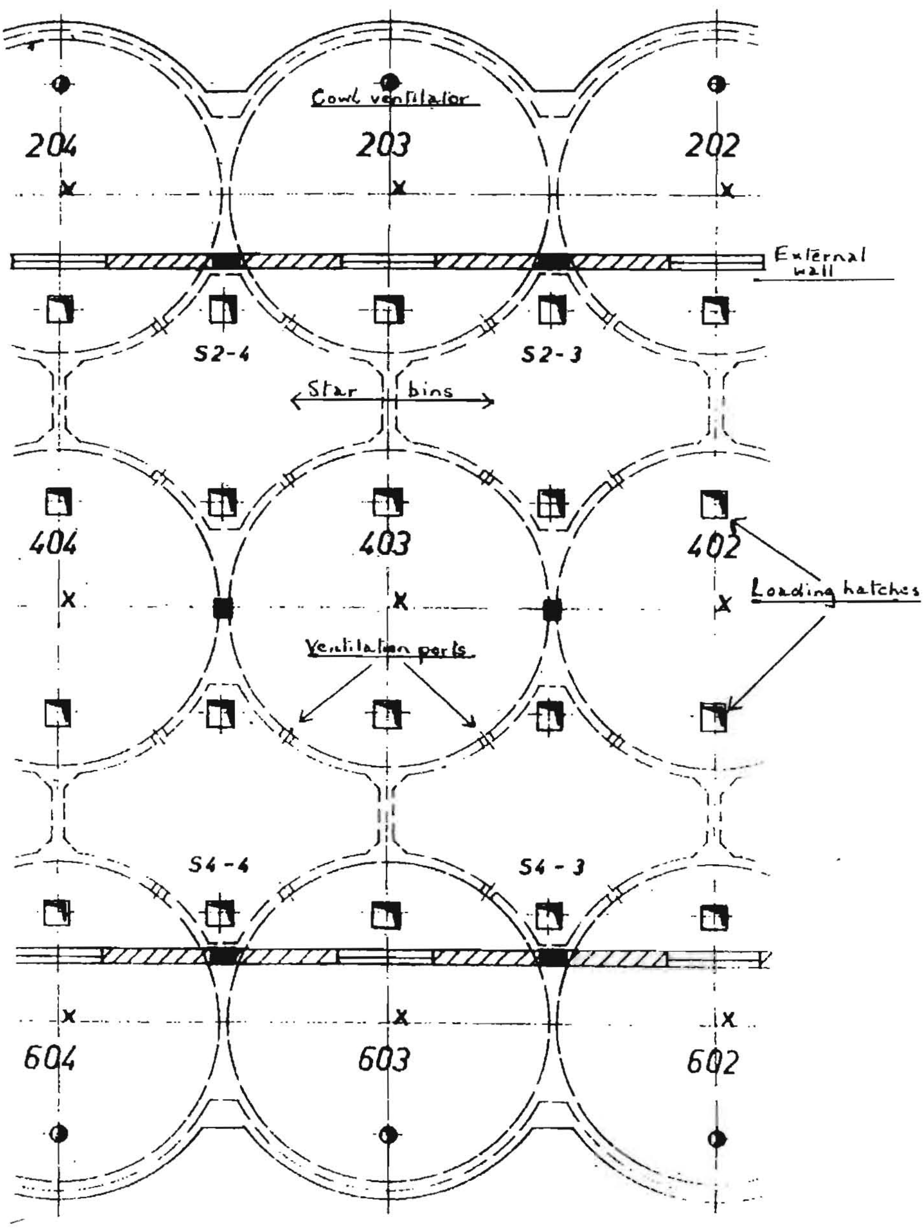

Fig. 1. Narayanganj silo complex - section of bin layout as seen from above.

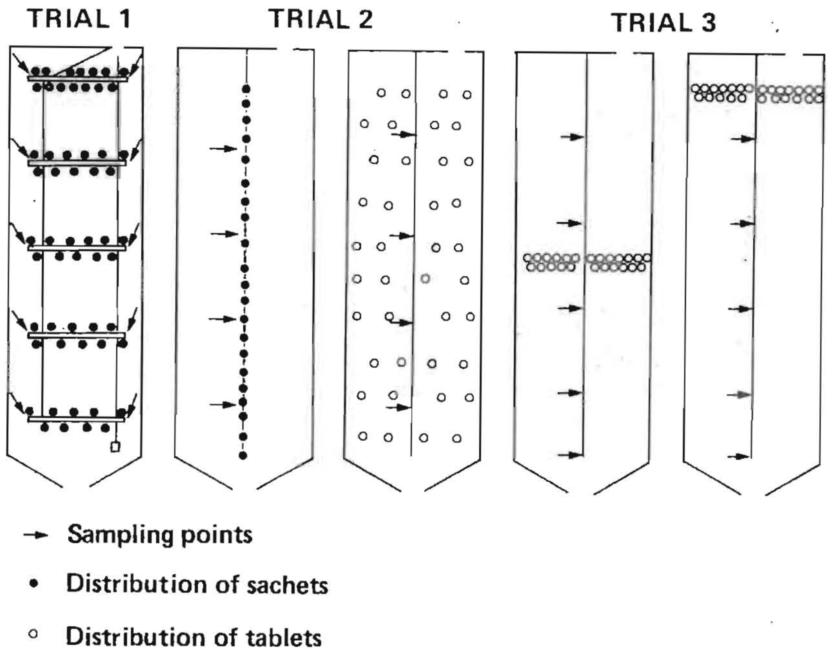

Fig. 2. Position of gas sampling lines and fumigation preparation in trials to investigate phosphine distribution in silo bins.

A rope and bamboo pole framework was assembled on the bin-deck floor. Polyethylene tubing (1.8 mm diam.) was taped to this framework to provide six pairs of gas sampling points at various depths in the bin (see Fig. 2). After assembly, sachets of an aluminium phosphide preparation (Detia Gas-Ex-B) were tied to the six bamboo poles at 30 sachets per pole (180 x 34 g sachets in total, equivalent to 2gPH_3 m^{-3} in the bin). The framework was lowered into the bin through the loading hatch and was held in place by the use of weights and a combination of fine and coarse supporting ropes. The cowl ventilator was sealed with polythene sheet and both top and bottom hatches were sealed with tape. The bin atmosphere was at 23°C, 55% relative humidity at the start of the trial.

Gas sampling lines were also lowered to the centre of the empty adjoining round bin 107 and star bin S1-8.

2.1.2 *Gas concentration measurement:* All twelve lines were cleared, using a footpump, some eighteen hours after placement of the fumigant. Dräger Model 31 multi-gas detector pumps were used with CH 21201 PH_3 60/a detector tubes for normal readings. For trace readings in adjacent bins and hatch and hopper areas CH 31101 PH_3 0.1/a detector tubes were used. Throughout the trial, lines were cleared with a used detector tube prior to taking any reading and two separate readings were taken at all points. Readings were taken at roughly twelve-hourly intervals for the first three days and thereafter every twenty four hours.

2.1.3 *Results:* The mean concentration of phosphine observed in Bin 108 is shown in Fig. 3. Table 1 summarises the *Ct*-products achieved after various times. The phosphine concentration observed and *Ct*-products attained in adjacent bins is given in Table 2.

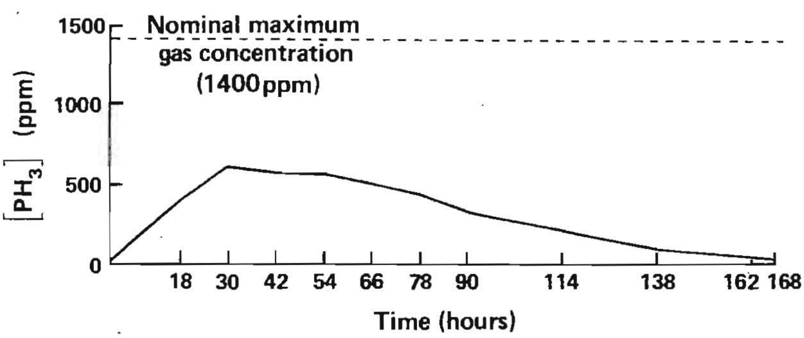

Fig. 3. Phosphine concentration (mean of 12 sampling points) observed in Trial 1 - Bin 108 treated empty at $2gPH_3\ m^{-1}$.

TABLE 1

Ct-products achieved in Bin 108 treated at $2gPH_3\ m^{-3}$ when empty (Trial 1).

Time after dosing (hours)	*Ct*-product achieved (g h m^{-3})		
	Mean of 12 points	Maximum	Minimum
72	48	51.3	45.2
96	59	62.8	54.5
120	65	69.8	60.1
144	69	77.2	63.4
168	70	79.1	64.8

2.1.4 *Discussion:* The evolution, build-up and dissipation of phosphine were extremely uniform throughout the bin. There was nothing to indicate that significant air movements were influencing gas concentrations nor were there any readings suggesting layering or

TABLE 2

Phosphine concentrations (ppm) and *Ct*-products (g h m^{-3}) in regions adjacent to Bin 108 during Trial 1.

Time after dosing (hours)	Location			
	Bin 107	Bin S1-8	Hatch Area	Hopper Area
12	16	28		
24	31	99	15	10
48	38	128	4	10
72	30 (3.2)[a]	50 (9.2)[a]	0	10
96	21 (3.9)	36 (10.7)	0	5
120	6 (4.2)	22 (11.5)	10	5
144	2.5 (8.6)	17 (12.1)	0.5	10
168	4.0 (8.7)	35 (13.1)	-	-

a *Ct*-products

sinking of gas even by the end of the seven-day trial period. It is evident that significant leakage either through the bin wall or the ventilation ports took place, since at no time at any sampling point did the phosphine concentration exceed 43% of the nominally available 1400 ppm (2.0 g m^{-3}) and readings as high as 190 ppm were achieved in the adjacent star bin (S1-8). Readings in the star bin were consistently higher than those in the round bin (Table 2), reflecting either the much greater area of common bin wall with Bin 109, the trial bin under fumigation, or the influence of the ventilation port leading directly into the star bin from Bin 108. There was no direct ventilation port connection between Bin 108 and the round bin (107) but there was a port connection between the star bin and the round bin (see Fig. 1).

The *Ct*-products recorded for the 3-7 day period (Table 1) indicate a barely adequate fumigation result for some susceptible species. The dosage rate of 2gPH_3 m^{-3} is equivalent to 3 tablets per tonne of grain, the standard recommendation for a full bin.

2.2 Trial 2

Trial 2 was carried out to determine whether similar gas concentrations were achieved in a treated bin filled with wheat as were achieved in Trial 1 and to replicate the treatment both in a round bin with no external wall surface exposed to the elements and in a star bin with a greater relative surface area of bin wall.

2.2.1 *Methods:* In this trial three bins, Nos 108, 403 and S1-7, were treated at a rate equivalent to $2gPH_3\ m^{-3}$ with aluminium phosphide preparations. Gas sampling lines were attached to the central thermocouple cable at four depths in each bin; the gas sampling lines in the bins adjacent to Bin 108 from Trial 1 remaining in position. Brass gauze filters were fitted over the ends of the lines to prevent blockage by grain. Bin 108 was filled with 750 tonnes wheat (22°C, 12.2% moisture content) and fumigated using sachets (Gas-Ex-B) in order to duplicate the gas evolution pattern of Trial 1. The sachets (180 x 34 g each) were tied to the central thermocouple cable in groups of 10. Bin 403 was filled with 760 tonnes of wheat (21°C, 12.0% moisture content) and treated with 240 x 3 g aluminium phosphide tablets (Phostoxin) added in batches to the grain stream every half-hour over the four hour loading period. Bin 403 was a round bin in the central row of the complex without any exposed external wall surface, but with ventilation ports leading into all four ajoining star bins. Bin S1-7 was filled with 450 tonnes wheat (22°C, 12.4% moisture content) and treated with a total of 1,200 x 3 g aluminium phosphide tablets added to the grain stream in batches of half-hourly intervals during filling of the bin. After filling and treatment, the top and bottom hatches of each bin were sealed with tape.

2.2.2 *Readings:* Readings were taken as in Trial 1.

2.2.3 *Results and Discussion:* Phosphine concentrations observed for the four individual gas lines in Bins 403 and S1-7 are shown in Figs 4 and 5. No such presentation of results is possible for Bin 108. The initial, heavy concentrations of phosphine evolving in close proximity to the gas sampling points did not dissipate to the point at which meaningful readings (i.e. < 3000 ppm) could be obtained until after six days from the start of the trial (see Table 3). At day 12 there was still an average concentration of 755 ppm. Therefore, this fumigation told us little about actual concentrations achieved in a normal fumigation. Some movement and leakage of phosphine into adjacent bins through the bin walls and/ or ventilation ports took place (Table 3).

Table 4 shows the *Ct*-products acheived in Bins 403 and S1-7 for the 3-7 day fumigation period.

It can be seen that with this form of treatment phosphine

TABLE 3.

Phosphine concentrations (ppm), at four depths in Bin 108 after treatment when full with $2gPH_3\ m^{-3}$, with phosphine concentrations in the ajoining star and round bins (Trial 2, phosphine-releasing sachets close to the sampling points).

Time after dosing (hours)	Bin 108				Bin S1-8	Bin 107
	9 m	15 m	21 m	27 m		
24	> 3000	> 3000	2700	> 3000	1.0	0
48	> 3000	> 3000	> 3000	> 3000	10.0	10.0
72	> 3000	> 3000	> 3000	> 3000	8.0	8.5
96	> 3000	> 3000	> 3000	> 3000	5.0	5.0
120	> 3000	> 3000	> 3000	> 3000	6.5	6.5
144	3000	> 3000	2600	> 3000	4.0	4.0
168	2400	3000	1700	> 3000	3.0	4.0
192	1950	2500	1100	2700	3.0	3.0
216	1500	2000	850	1800	line cut	2.0
288	850	1200	170	800	-	1.0

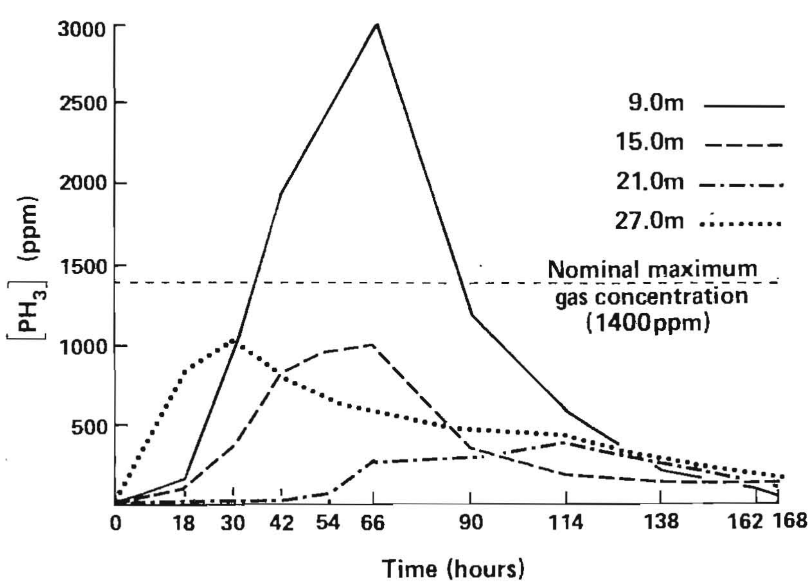

Fig. 4. Phosphine concentrations in 760 t wheat in Bin 403 treated with $2gPH_3\ m^{-3}$ added as tablets during loading - Trial 2.

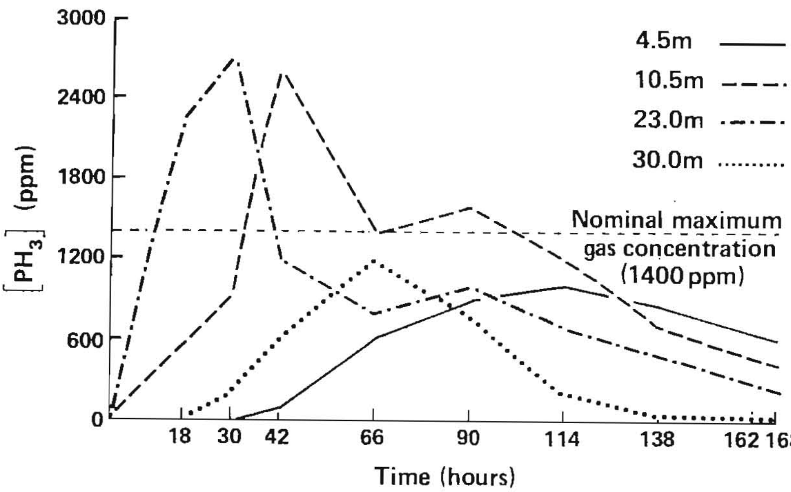

Fig. 5. Phosphine concentrations in 450 t wheat in star Bin S1-7 treated at 2gPH_3 m-3 added as tablets during loading - Trial 2.

TABLE 4

Ct-products (g h m^{-3}) achieved in Bins 403 and S1-7 fumigated when full at 2gPH_3 m^{-3} (Trial 2, phosphine-releasing tablets added to the grain stream at half-hourly intervals).

Time after dosing (hours)	Bin 403				Bin S1-7			
	9 m	15 m	21 m	27 m	9 m	15 m	21 m	27 m
72	161.1	60.3	N/R	74.4	N/R	138.4	146.9	N/R
96	204.4	73.7	N/R	90.6	53.0[b]	188.8	178.5	72.7[c]
120	224.9	80.3	31.7[a]	109.1	85.6	229.1	202.7	88.3
144	223.0	84.8	42.3	113.2	113.9	254.6	219.5	92.5
168	235.7	89.0	48.0	118.9	136.2	271.3	229.4	94.0

a Computation based on readings commencing at 48 hours.
b Computation based on readings commencing at 36 hours.
c Computation based on readings commencing at 24 hours.
N/R Not calculated, *Ct*-product zero or very low.

concentrations varied widely with the depth in the bins. No gas was recorded at all at the 21 m depth in Bin 403 for the first thirty six hours. Even after 7 days from dosing the *Ct*-product (Table 4) achieved there would have been inadequate for complete control of susceptible *Sitophilus oryzae* and resistant strains of *Rhyzopertha dominica*. However, the 120 hour *Ct*-product at 21 m (48 g h m^{-3}) may have been just adequate to achieve complete control of these species. At all other points in both bins a seven day exposure would have produced *Ct*-products (> 80 g h m^{-3}) which were probably quite adequate for complete control at these temperatures (Hole, 1981).

The high concentrations and *Ct*-produced recorded at other points indicate that leakage of gas is much reduced by the presence of the bulk wheat and that the increased surface area of wall in the star Bin S1-7 did not apparently give rise to reduced gas concentrations within the bin. Nor did the presence of four unsealed ventilation ports above the grain at the top of Bins 403 and S1-7 appear to exert any significant effect of gas levels within the bulk.

The distribution of the fumigant is important for short duration fumigations (Heseltine, 1973) but is not critical if sufficient time can be allowed to permit phosphine concentrations to equilibrate throughout the bin. Since instructions for application of fumigant even at half-hourly intervals will probably not be followed rigorously at the silo complexes in Bangladesh in practice, it remains to be seen whether the application can be yet further simplified without compromising the desired result.

2.3 Trial 3

This trial was carried out to determine to what degree and over what period of time phosphine, evolved from aluminium phosphide tablets, would penetrate a grain bulk in a silo from a central position in the grain bulk or when applied at the top of bin.

2.3.1 *Phosphine evolved from tablets placed half-way down a full wheat bin.*

2.3.1.1 *Methods:* Gas sampling lines were taped to the central thermocouple cable in round Bin 403 at 4.5 m, 10.5 m, 16.5 m, 23 m and 30 m depths, as previously described. 375 tonnes of wheat (24°C 14.5% m.c.) were loaded then into the bin. Aluminium phosphide tablets (1920 x 3 g, Phostoxin) were deposited onto the grain surface in one operation through both loading hatches. A further 375 tonnes of wheat from the same source were added to the bin. Both top hatches were then closed.

2.3.1.2 *Gas concentration measurements:* Readings, as described in Section 2.1.2, were taken daily at each point and the mean of two readings was recorded.

2.3.1.3 *Results and Discussion:* The variation in gas concentration at 16.5 m, 23.0 m and 30 m is shown in Fig. 6. No phosphine was recorded at 4.5 m and 10.5 m depths. *Ct*-products for the 3-8 days after dosing are given in Table 5.

TABLE 5

Ct-products achieved in Bin 403 treated with aluminium phosphide tablets applied at $2gPH_3\ m^{-3}$ in a band half way down the grain bulk (Trial 3).

Time after dosing (hours)	Depth in bin [a] 16.5 m	23 m	30 m
72	53.8	156.0	N/R
96	63.4	256.5	N/R
120	66.7	350.6	26.0[b]
144	68.2	411.2	68.7
168	69.1	445.5	117.4
192	69.5	463.3	164.1

a No phosphine detected at sampling points at 4.5 and 10.5 m.
b Computation based on readings commencing at 48 hours.
N/R Not calculated, *Ct*-product zero or very low.

The phosphine evolved from tablets placed half-way down the silo passed rapidly, within 36 - 48 hours, down to the 23 m depth and thence down to the bottom of the bin in 96 - 120 hours. This penetration concurs with that recorded by Banks (1977). In our case, however, the gas dispersed downward through the bulk, not upwards as observed by Banks (1977). After eight days from treatment there was virtually no gas that the point of application but still 1300 ppm at the bottom of the bin. After ten days, this level had fallen to 400 ppm. As gas concentrations did not rise above the 'trace' level in the hopper free space below the bin and, as there was obviously no question in this trial of leakage through the

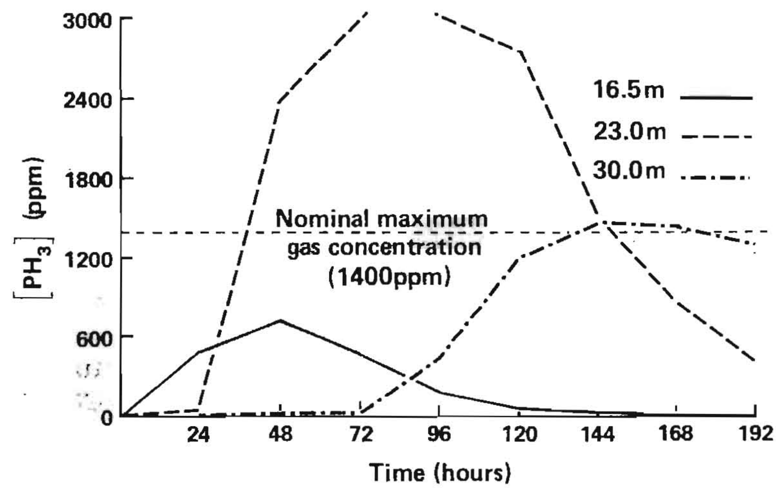

Fig. 6. Phosphine concentrations in 750 t wheat in Bin 403 treated at a rate of $2gPH_3\ m^{-3}$ with tablets added in a layer after half-filling the bin - Trial 3.

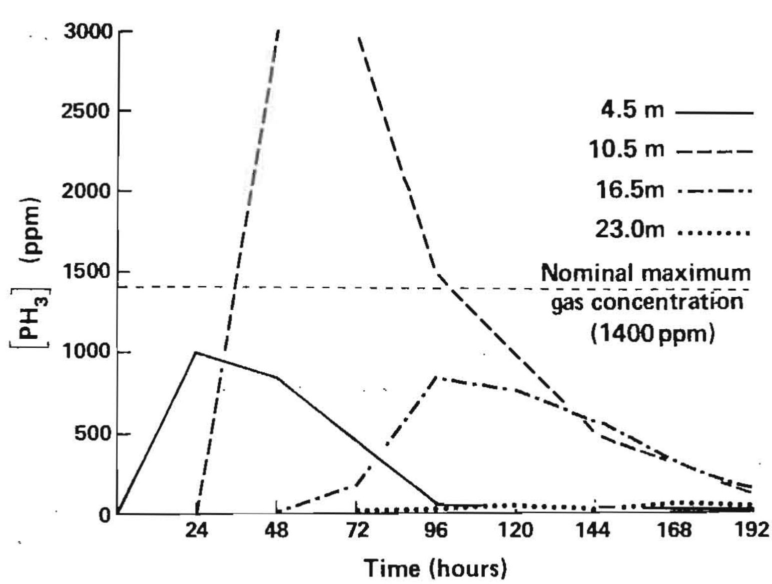

Fig. 7. Phosphine concentrations at 760 t of wheat in Bin 403 treated at a rate of $2gPH_3\ m^{-3}$ with tablets added to the grain surface after filling.

ventilation ports at the top of the bin, it must be concluded that leakage through the bin wall is the most important gas loss factor in a normal fumigation.

Ct-products achieved were more than adequate for insect control at 23 and 30 m and possibly just adequate at 16.5 m depth. A six or seven day exposure period would be required for effective results, but gas levels at the bottom of the bin would still be possibly undesirably high to permit grain movement immediately.

2.3.2 *Phosphine evolved from tablets placed at the top of a full wheat bin.*

2.3.2.1 *Methods:* After an interval of five days to allow remaining phosphine to dissipate, Bin 403, with wheat and gas lines intact, was again treated. Tablets of aluminium phosphide (1920 x 3 g in total, Phostoxin) were tipped onto the surface of the grain through both loading hatches. Ten tonnes of wheat were loaded into the bin to cover the tablets.

Gas sampling lines were suspended at a depth of 15 m in all four adjacent star bins sharing a common wall and ventilation ports with Bin 403. Top and bottom hatches of the empty star bins were closed but not sealed. Temperatures in the star bins were 26-28°C with relative humidities of 55-64%.

2.3.2.2 *Results and Discussions:* The phosphine concentrations observed at 4.5 m, 10.5 m, 16.5 m and 23 m depths are shown in Fig. 7. Readings were not sufficiently high at the 30 m point to permit a similar presentation but are given, together with *Ct*-products achieved at all points between 3 and 8 days after closing, in Table 6. Gas concentrations observed in the adjacent star bins are given in Table 7.

The phosphine did reach the 30 m depth of the grain bulk within 4 - 5 days but not in a sufficient amount to give an effective *Ct*-product at this depth.

The *Ct*-producs achieved after 8 days from dosing at 23 and 30 m were quite inadequate, even for control of the most susceptible species. By prolonging the exposure to ten days it is difficult to see how the results at these depths could have been significantly improved. Concentrations would not have risen appreciably due to falling gas as little gas remained at higher levels.

It is clear that neither of these two simplified forms of fumigant application will give adequate concentrations of phosphine throughout the grain bulk for sufficient periods of time to offer

TABLE 6

Ct-products achieved in Bin 403 treated with aluminium phosphide tablets applied at $2gPH_3\ m^{-3}$ to the grain surface (Trial 3). Gas concentrations achieved at 30 m depth also shown.

Time after dosing (hours)	Depth in grain					Phosphine concentration (ppm) at 30 m
	4.5 m	10.5 m	16.5 m	23 m	30 m	
72	73.7	165.2	N/R	N/R	N/R	0
96	78.7	218.1	N/R	N/R	N/R	5.0
120	80.2	255.2	40.8[a]	N/R	N/R	5.0
144	81.5	276.0	64.8	3.3[b]	0.83[b]	20
168	82.4	287.4	82.1	5.3	2.00	40
192	83.3	293.6	90.9	7.0	3.5	45

a Computation based on readings commencing at 48 hours.
b Computation based on readings commencing at 84 hours.
N/R Not calculated, *Ct*-product zero or very low.

TABLE 7

Phosphine concentrations (ppm) observed in the four star bins ajoining Bin 403 while under fumigation (Trial 3, surface fumigation).

Time after dosing (hours)	Bin No.			
	S2-3	S2-4	S4-3	S4-4
12	75	5	20	8.5
24	15	10	40	17
48	15	10	50	30
72	11	1.0	30	15
96	1.0	1.0	35	20
120	0.2	0.2	18	10
144	trace	trace	16.5	11
168	0	0	4.0	5.0
192	0	0	1.0	1.0

an acceptable prospect of elimination of all of our target species whether susceptible or otherwise.

3. CONCLUSIONS

To date we have no evidence to show that insect populations in the Bangladesh silo system have developed resistance to phosphine. We do, however, have a considerable amount of data to show that resistance to phosphine has been developed in the godown system where only bagged grain is handled (Tyler *et al.*, 1983). Tests conducted so far show that *Tribolium castaneum*, *Rhyzopertha dominica*, *Cryptolestes ferrugineus* and *Oryzaephilus surinamensis* have all developed levels of resistance to phosphine that are of practical significance (C.E. Dyte, personal communication).

It could be argued that in the absence of evidence of resistance from silos we should adopt the most economic form of fumigation and simplify the application method where possible as we have attempted to do in these trials. It would however, be most unwise to assume that phosphine resistance is or will remain absent from silo insect populations. Therefore we should promote fumigation techniques which prevent the exposure of grain insects to possibly sub-lethal or marginal doses of phosphine. As bin temperatures during the winter months will drop to the 20 - 23°C range, as they were during the earlier part of these trials, and this will adversely effect the level of control which can be expected at low concentrations, this reinforces the conclusion that placing the fumigant formulation on top of the bulk grain should not be encouraged as a fumigation technique.

It seems quite clear that substantial gas loss does occur through the concrete bin walls, but it is also clear that quite satisfactory phosphine levels can be achieved and maintained in a silo complex of this age and type at the economically acceptable rate of 2gPH_3 m^{-3}.

There was no indication that the presence of the small ventilation ports above the grain surface at the top of the bins detracts in any significant way from the fumigation results. Normal closing of the bin hatch covers on the bin deck floor and hopper hatches at the bases of the bins, without any additional sealing measures, would seem to be adequate for a normal treatment. Concentrations measured in the bin hatch and hopper areas during fumigations showed no dangerous levels of phosphine and ventilation of these areas was adequate.

Future recommendations for a fumigation technique at these silos should aim at ensuring that the fumigant is dispensed at several levels throughout the grain bulk either by a simple manual application during the filling process or by bringing into commission the automatic pellet dispensers. The dosage rate should remain at $2gPH_3\ m^{-3}$ (equivalent to 3 tablets per tonne of grain for a full bin) and the fumigation period should be 7 days to ensure even and adequate distribution of gas at all levels.

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FUMIGATION AS PART OF AN INTEGRATED PEST MANAGEMENT PROGRAM

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The prevention and control of pest organisms in grain is achieved by a number of policies and procedures that are employed in food storage systems. Careful planning in the design of storage and handling facilities, so that infestation is impeded or prevented, is of great importance. Any peculiarity of the system that might allow the introduction and increase of pest organisms not only endangers the quality of the product but it can also allow cross contamination and undermine the integrity of an entire pest control program. Facilities that are amenable to sanitation and disinfestation procedures are likely to be most beneficial to food storage and handling systems. Best results may be attained by careful integration of all the procedures that will prevent contamination, suppress development or eliminate pest organisms.

Fumigation is a widely used procedure that has served an important role in pest control programs for many years. As fumigants can penetrate into materials to eradicate pest organisms, mainly insects, from sites where no other form of pest control is feasible, they are invaluable agents for use in food preservation programs. The continued utility of fumigants is dependent, not only on their penetrating properties and effectiveness as pest control agents, but also on the introduction of new and adaptable procedures that will meet the demands of an ever-changing food storage technology and on safety procedures that will allow them to be used without harm to human beings. Safety measures, both for workers handling fumigants and for consumers eating treated products, must progress to meet the requirements of present day health standards. Although a great amount of research has been done on fumigants and much information is available on their effectiveness, considerably more information is required to fully exploit the potential applications of these materials for pest control and to ensure that they are used with minimal hazard to man. Continued acceptance of the fumigants by health authorities and the general public is dependent on such information.

CURRENT STATUS OF FUMIGANTS AND FUMIGATION

Fumigants are one of the oldest groups of pesticides in use today. They have been employed on a commercial scale for the treatment of grain and other commodities for over 100 years. There are only a few chemicals that have the appropriate properties to be fumigants and even fewer that are suitable for use in treatment of food materials. Of the variety of compounds that have been tested over the years, less than a dozen have been extensively used as fumigants and many of these have very limited applications; two compounds, methyl bromide and phosphine have proven to be particularly suitable for many different kinds of fumigation treatments and are still extensively used.

Because the statistical possibilities of finding suitable, small molecule compounds, that have not been tested already are very remote, the fumigants that we now have are about the only ones that are likely to be available in the future. This means that the future of the fumigation technique is dependent on a very small number of chemicals and no substitutes are likely to be found. Furthermore, the continued acceptance of these few chemicals is dependent on whether or not the necessary research will be carried out to provide the information needed for them to be used according to modern health standards.

Hazards and Safety Precautions

Fumigants are toxic to man as well as to insects and any exposure before, during or after a fumigation treatment can be harmful to human beings. Safety from occupational hazards and from contamination of food has become a matter of increasing concern in the utilization of fumigants for treatment of food materials. The need for improved safety measures has become more evident as new technology for detection and analysis has become available and as new information on the toxic hazards of fumigants revealed. Both the applicator that uses fumigants and the consumer that eats treated goods must be protected from potentially harmful exposures. Regulatory agencies must move to provide guidelines that will adequately cope with these occupational and residue problems.

Occupational hazards for personnel that handle the toxic gases fall into two categories - acute and chronic hazards. The acute effects of fumigants have long been known and appropriate precautions are usually taken to avoid them. If proper care is taken, work with fumigants is no more hazardous than any other industrial or domestic technique that uses potentially harmful chemicals. Chronic or long-term effects, which may result from overdose to a single exposure of a toxic gas or from repeated exposure to low levels over a period of time, are less evident. The effects may not apppear until long after exposure to the fumigant has taken place and, in some cases, they may not be easily associated with it. Chronic effects may take the form of injury to liver, kidneys or other organs or tissues or there may be other delayed effects. Some of the fumigants have the ability to produce cancer in animals under experimental conditions and it is believed that they may be potential carcinogens for humans. Acrylonitrile, carbon tetrachloride, ethylene dibromide, ethylene dichloride and ethylene oxide are all "suspected" carcinogens (ACGIH, 1981, National Cancer Inst. 1978). Consequently, threshold limit values have been reduced to very low levels and instructions for their use have been revised accordingly, to reduce the hazards.

Residues of fumigants in food materials have also taken on increasing importance as awareness of their presence in food has come about. New instruments and new methods of analysis that can detect and measure residues down to low ranges of parts per billion or less have indicated the occurrence of residues that were previously unsuspected. Consequently the setting of meaningful tolerance levels has become extremely difficult. Acceptable daily intake studies for degradation products and, in some cases, for unchanged fumigant are incomplete and often lacking. Also the toxicological significance of exposure to low levels of fumigants or their residues for extended periods is not known. Authorities are reluctant to set tolerance levels for residues, particularly carcinogens where the long term effects have not been determined.

With the ultra-sensitive methods of detection that are now available, the possibility of fumigating a commodity without leaving some residue that can remain undetected is remote. The question of health hazards cannot be resolved until the toxic effects of these residues have been determined. For fumigants that are suspected carcinogens, the question of health hazards is further complicated by the dearth of knowledge of the cancer-causing process. The possible cumulative effects of repeated exposure to ultra-low levels of carcinogens are not known. Consequently the hazards brought about by exposure to very low levels of suspected fumigant carcinogens cannot be predicted. Further developments in the field of cancer research will be needed, along with additional toxicological data and new developments in safety procedures to adequately cope with this problem.

Health Standards for Fumigants

For the protection of human beings from the toxic effects of airborne substances, such as fumigants, threshold limit values have been established. These values are based on the best available information from industrial experience, from experimental human and animal exposure and, when possible, from a combination of the three. In recent years threshold limit values for many of the fumigants have been reduced to make allowance for known or suspected toxicological effects (Table 1).

Table 1. Threshold limit values for fumigants as listed by the American Conference of Governmental Industrial Hygienists (ACGIH) in 1964 and 1981.

	TLV	
	1964	1981
Acrylonitrile[1]	20	2
Carbon disulphide	20	10
Carbon tetrachloride[2]	10	5
Ethylene dibromide[2]	25	( )[3]
Ethylene dichloride	50	15
Ethylene oxide[2]	50	5
Methyl Bromide	20	5

[1] Listed by ACGIH (1981) as "human carcinogens"

[2] Listed by ACGIH (1981) as "industrial substances suspect of carcinogenic potential for man"

[3] No value assigned because of insufficient information

While threshold limit values are believed to allow adequate protection for customary working situations, i.e. an 8 hour day and 40 hour week, they do not apply to other situations. For fumigation treatments where personnel may be exposed for longer periods of time acceptable threshold values have not been established and the criteria for arriving at such values have not been developed.

In one extensively practised fumigation procedure, i.e. "in transit" ship fumigation, personnel are confined in close proximity to the fumigated areas for continuous and extended periods of time. If current standards for health safety are to be followed it is essential that appropriate threshold limit values should be established to cover such situations. Health authorities state that established threshold limit values based on intermittent exposures cannot be transposed nor related directly to continuous prolonged exposures. The toxicological effects resulting form prolonged exposure could be very different from short intermittent exposures. New information on long term dose-effect relationships is needed to establish meaningful tolerance values for these treatments. Also adequate methods for monitoring the low concentrations of fumigant occurring in such circumstances will be required to meet current health criteria.

Even the data on the toxicity and toxicology of fumigants that we have at present is inadequate as measured by present day health standards. Since fumigants were first used as pesticides in times when knowledge of the hazards of chemicals to human health was far less advanced than it is today, the requirements for health standards were less demanding. Fumigants were registered in those bygone times and are still being used without the information that is presently required for the approval of new pesticides. Regulatory agencies are faced with the dilemma of either by-passing legislative requirements to give continued approval for the fumigants or of de-registering invaluable materials that cannot be replaced.

A review and re-registration process is now underway in Canada and the United States to bring the fumigants in line with the health safety standards set for other pesticides. For this re-registration a considerable amount of toxicological data, that is presently unavailable, will be needed to establish apropriate safety regulations. The question of how to obtain the necessary data and who should underwrite the cost is a difficult one to resolve. Because of the high cost of gathering toxicological data that will meet present day standards, because of the limited markets for fumigants and because most of the fumigants can no longer be covered by patents, chemical manufacturers are reluctant to invest large sums of money in these materials. The benefits derived from the information would, however, be shared by users of fumigants through the world as well as by the manufacturers and suppliers.

This seems to be an area where an international co-operative effort would be invaluable. If several national governments could combine their efforts, together with the appropriate industries, to finance such an operation and carry out the research, the information could be obtained without excessive burden on any one group.

The lack of essential data on fumigant toxicology could jeopardize the development of realistic health standards and might imperil the future approval of fumigants for use on food materials. Regulatory authorities could be forced into the position of banning useful materials because of the fear of harmful effects. It would be unfortunate if some of the fumigants were lost simply because the required toxicological data were unavailable.

Detection and Analysis of Fumigants

Although many new possibilities for rapid detection and analysis of fumigants have become available through modern technology, the development of methods that will fill the needs of fumigators and public health officials has been slow. For several fumigants practical methods that will give instantaneous readings of concentration of the gas in air are not available. For instance there are no quick, easy and inexpensive methods for rapid detection of fumigants such as ethylene dibromide, ethylene dichloride and chloropicrin. For other fumigants like carbon tetrachloride, carbon disulphide, hydrogen cyanide, ethylene oxide and phosphine, where procedures using glass detector tubes or indicator tapes are available, analysis is somewhat cumbersome, often limited in range and the lower limits of detection are barely adequate. Even for the fumigants phosphine and methyl bromide, the lower limits of detection with glass detector tubes (0.1 and 3 ppm respectively) are just below the TLV of 0.3 and 5 ppm and consequently the margin of safety is minimal.

Some instruments that have been widely used in the past, such as the halide leak detector, are now considered to be inadequate to meet the demands of present day health regulations. The halide leak detector has given valuable service to fumigators, over a long period of time, particularly, for the fumigant methyl bromide. It is light-weight, economical and easy to use and it gives an instantaneous response when fumigant is present. It is invaluable as a detector for locating leakage of halide fumigants from treated areas and for rapid warning against toxic concentrations of these compounds. However, it is not sufficiently sensitive to detect the low concentrations that are now established for health protection. For methyl bromide, the instrument will show a response for concentrations down to 10-20 ppm but it will not detect the gas at the presently established TLV of 5 ppm. Therefore the halide leak detector should not be used as an indicator to declare areas safe for re-entry of personnel.

Fumigants in the atmosphere can be detected and analysed with great precision with equipment like gas chromatographs. A new instrument (Fig. 1) designed for field use and one that is portable, easy to use and has a high degree of sensitivity over a wide range of concentrations and down to ultra low levels has come on the market recently (Barker and Leveson, 1980, Bond and Dumas 1982, Dumas and Bond, 1982). This instrument is capable of giving rapid and reliable analysis of methyl bromide, phosphine, ethylene oxide and ethylene dibromide in parts per billion range and it can be adjusted to analyse the high concentrations used for insect control. An infra red analyser that will analyse fumigants over the range of concentrations used for insect control is also available (Webley et al 1981).

Figure 1.

Portable gas chromatograph for measuring low concentrations of fumigants at TLV levels and the high concentrations used in fumigation.

A number of new devices that are being developed for monitoring exposure of individuals to toxic gases (McCammon 1979) may have potential value for personnel using fumigants. They are small, light-weight and can be located in the immediate breathing area of the worker. A whole-air sampler known as "critical orifice personal sampler" has been tested successfully for a number of years and is available commerically. Several passive monitors that collect samples on a collection medium are becoming available. A pocket-size gas chromatograph that will provide real-time warning to acute exposures and will accumulate a workers 8-h time weighted average exposure is in the developing stages.

Methods of analysis for residues of fumigants in food materials have progressed to the stage where very minute quantities of compounds can be detected and measured. Information on many of these methods may be found in the following references: Alumot and Bielorai, 1969, Bielorai and Alumot, 1975, Dumas 1973, 1978, 1980, Dumas and Bond 1975, 1977, 1979, Fairall and Scudamore, 1980, Heuser and Scudamore 1968, 1969, 1970, Jagielski et al 1978, Majumder et al, 1965, Monro, 1969, Scudamore and Heuser, 1971, Stijve 1977. Some information is available on reaction products and the nature of residues remaining in food after fumigation, however, much more research is required to fully assess the significance of fumigation residues to human health.

As more knowledge about the long term hazards of fumigants becomes available and as new methods and equipment for detecting and measuring concentrations of fumigants, both in the environment and as residues in food materials, are developed, the whole procedure of fumigation can be made much safer. All of the facilities of modern technology should be employed to achieve this objective.

Effectiveness of Fumigants in Controlling Insects

The toxicity of fumigants to insects is influenced by environmental factors, particularly temperature, and by the innate characteristics of the insects themselves. Much information has been published concerning the toxicity of various fumigants to stored product insects. In general it can be said that tolerance varies considerably amongst different species of insects and, even within a species, various stages and conditions of the insects may exhibit a range of tolerances. LD_{50} and LD_{99} values for most of the fumigants have been established for most species, particularly for the stages of these insects that are easiest to obtain and treat. Unfortunately the stages that are difficult to procure for experimentation (often eggs and pupae) are the ones that are most difficult to control and toxicity data for these stages are lacking.

The effectiveness of fumigants on insects can also be altered by other characteristics of the insects. Certain stages of some species enter a state of diapause in response to extreme environmental conditions (Howe 1962) and this alters their tolerance to fumigants. For insects in this state, tolerance to methyl bromide and phosphine may be several times greater than for non diapausing insects (Bell 1977a, b).

Protective narcosis can also be a significant factor in fumigant toxicity. If insects are exposed to sublethal concentrations or to excessively high concentrations at the beginning of a treatment they may go into a depressed state where the fumigant is less effective than it would be at normally recommended concentrations. The tolerance of insects to the fumigant phosphine at concentrations in excess of 0.5 mg/l may increase considerably above that which occurs at low concentrations of 0.005 and 0.5 mg/l (Winks 1974a). This makes insect populations more difficult to control.

Several species of stored product insects have the ability to develop resistance to fumigants. Research has shown that strains of insects resistant to methyl bromide and phosphine can be easily produced in the laboratory (Monro et al. 1972, Winks 1974b, Bond and Upitis, 1976). In a global survey Champ and Dyte (1976) found resistance to both methyl bromide and phosphine in wild populations of insects in widely scattered areas of the world. In collections of 849 strains of insects from 82 countries, 5% had some resistance to methyl bromide and 10% to phosphine. It was concluded from this survey (which was made in 1972-73) that resistance to fumigants was, as yet, limited in extent and often at marginal levels, but that it was of some consequence as it posed a real threat to the future use of fumigants as control agents. Considering the facts that (a) further resistance has probably developed in diverse populations of stored product insects since the survey was made some 10 years ago, (b) the number of fumigants approved for use is declining (c) the few fumigants remaining are being used with increasing frequency, the future utility of fumigants will be very limited unless appropriate means are developed to counteract resistance.

The significance of insect resistance to fumigants can be determined best by continual monitoring of insect populations for tolerance. Comprehensive surveys such as that carried out by Champ and Dyte (1976) are needed periodically to estimate the magnitude and gravity of the problem. Much research is required to understand the basic mechanisms of resistance and to design effective countermeasures to cope with resistant population of insects. Information on the characteristics of the resistant insects - the tolerance of different stages of a species, the tendency of different stages to develop resistant traits, cross resistance to other pesticides, biochemical mechanisms conferring resistance, as well as genetic and behavioural features, are necessary to develop comprehensive methods to cope with resistance on a rational basis.

Properties of Fumigants

Although much is known about the properties of fumigants, more information is needed to use them most effectively and safely. For example, sufficient information on flammability and stability of some compounds and on possible relationships between fumigation and dust explosions is not available. With the fumigant phosphine, data on flammability are meager - the influence of temperature and pressure stability of the molecule under the variety of conditions that exist in fumigation milieu have not been defined.

For many years phosphine was not recommended for use in recirculation systems in grain storages for fear of explosion or fire that might occur if the fumigant was subjected to abnormal pressures. Recently practical trials with the fumigant have shown that treatments using a low-flow recirculation technique can be carried out successfully (Cook 1980). The interstitial air of a grain mass is slowly displaced with fumigant-air mixture so that the fumigant is not exposed to appreciable changes in pressure. In this way it can be uniformly dispersed in a few hours to increase the efficacy of the treatment. However, essential information on the parameters related to stability, flammability and explosion hazard of the compound is lacking. The pressure limits to which phosphine can be subjected without danger of fire or explosion, the influence of temperature (in the ranges found in grain storages) on the stability molecule and the possible influence of other factors as dusts, moisture and other gases on the reaction must be determined if the technique is to be used with complete confidence.

Concern also has been expressed over a possible connection between grain dust explosions and fumigant in grain. Some tests have given evidence to suggest that there may be significant interactions between fumigant vapours and explosible dusts. Tests with a non-flammable mixture of carbon tetrachloride and carbon disulphide showed that the explosible concentration of commercial flour dust could be lowered appreciably by the fumigant (Atallah, 1979). However, another investigation with three fumigant mixtures containing carbon tetrachloride and ethylene dichloride or carbon disulphide showed that there was no increase in the severity of grain dust explosions and in some cases the vapours actually suppressed the explosion (Tait et al, 1980).

The uncertainty about any connection between fumigation and dust explosions will remain until further information is available. In the meantime due precautions against creating a hazardous combination of circumstances may be warranted, particularly with fumigants that have relatively low flammability limits.

Another problem sometimes encountered with fumigants concerns corrosion of metals. Phosphine reacts avidly with the metal copper and sometimes causes considerable damage to copper components of equipment exposed to it. Very little information on this problem is available. Some experiments carried out in our laboratory recently have provided some data on the reaction and have shown that anticorrosive agents can reduce the effects of the fumigant to a very low level (Bond et al, 1984). Here again more research is required on the chemistry of the reaction to provide a rational basis for counteracting the effect.

FUMIGATION AND PEST MANAGEMENT PROGRAMS

Fumigation is just one of a number of methods that can be used for controlling pests in stored products. Best control is likely to be obtained when all appropriate measures are taken to eliminate pest organisms. In an effective pest management program, methods of prevention and control are integrated to give maximum protection of goods at the lowest possible cost. A number of other procedures that have been found effective in preventing and controlling infestations are as follows:

1. Sanitation.
2. Exclusion of pests.
3. Low temperature - "freeze-outs", refrigeration, aeration.
4. High temperature - heating of mills.
5. Moisture control - grain drying.
6. Aeration - cooling, drying, elimination of temperature gradients.
7. Protectants - chemicals, inert dusts, natural compounds.
8. Residual insecticide sprays.
9. Atmospheric gases - carbon dioxide, nitrogen.
10. Gamma radiation, radio and sonic waves, microwaves, infrared radiation.
11. Pheromones.
12. Insect growth regulators.
13. Insect pathogens.
14. Predators.
15. Insect resistant packaging.
16. Resistant varieties.

An effective integrated pest management system should begin with comprehensive planning to include all aspects of the problem, followed by the application of appropriate preventative and control methods. For example, the planning of pest management for a commodity like farm-stored grain may be divided into five major categories:

1. Exclusion of the pest organism.
2. Inspection procedures.
3. Good housekeeping and sanitation.
4. Physical and mechanical control.
5. Chemical control.

Infestation problems can often be reduced by careful planning so that the possibilities of pest organisms reaching the commodity will be minimized. Location of the storage relative to sources of infestation is important as well as quality of the structure. Well built storages with a minimum of sites where debris can accumulate and insects develop are desirable. Other features of the storage that should be considered include - facilities for conditioning such as aeration systems or driers, provision for proper inspection and cleaning and appropriate facilities for pest control procedures. An effective pest management program may include the following steps:

1. Use of sound structures for storage of commodities.
2. Maintaining clean conditions around storages.
3. Removal of residues of grain or other material from storage facility 4 to 6 weeks prior to storing newly harvested produce.
4. Spraying of storage with approved residual insecticide after removal of food residues.
5. Storage of commodity in a condition suitable for optimum storage e.g. grain is best stored at low moisture levels.
6. Treatment with appropriate insecticide protectant at time of storage may be desirable.
7. Use of aeration or other procedures to cool grain and maintain uniform temperatures below those favourable for development of pest organisms.
8. Regular inspection to determine:
   (a) evidence of insect activity or the development of micro-organisms
   (b) accumulation of moisture
   (c) changes in temperature
9. If insects are detected grain should be fumigated; where field infestation occurs grain should be fumigated within 6 weeks after harvest. If micro organisms are developing, further drying may be required.

Several fumigation techniques may be combined or incorporated with other practices such as controlled atmosphere techniques or aeration and drying procedures. Some information on the potential usefulness of combining fumigants with carbon dioxide has already been obtained (Calderon and Carmie 1973, Jones 1938, Kashi and Bond 1975). The toxicity of fumigants may be greatly enhanced by combination with treatments where the insects have been weakened by exposure to carbon dioxide or to an anoxic atmosphere. The use of fumigants in conjunction with aeration procedures has yet to be explored.

By careful planning and management, fumigation may be incorporated into food preservation systems so that fumigants can be used more effectively and safely than when used independently. They should never be used as a substitute for sound management and good sanitation procedures The benefits derived can include: reduced cost of storage with improved food quality, reduced residues in food materials, greater occupational safety and less environmental contamination. All of these benefits are of great concern to the general public and will be factors that have to be taken into consideration in the future use of fumigants. The ultimate goal in the control of pests in stored products should be to so improve the methods of handling, storing and processing commodities, that the need for pesticides will decrease. However, the protection of grain from the ravages of pest organisms, particularly insects, will still depend on the judicious use of fumigants for many years in the future. New approaches to effective fumigant utilization can only come through intensive investigation of all the factors that relate to insect control. To date, research on fumigants has trailed far behind other developments in science and technology and users of fumigants have failed to make maximum use of research data and technological innovations. Hopefully increased effort will be made in the future to provide and to employ the necessary information and instrumentation, so that these valuable materials can be utilized with greatest efficacy in comprehensive pest management programs.

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A METHYL BROMIDE MONITOR

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Abstract - A sensitive and accurate instrument is described for monitoring concentrations of methyl bromide in the range 1 - 20 ppm at numerous points in large work-areas. Its operation is entirely automatic, micro-computer management being used to continuously standardise and calibrate the instrument, monitor its performance, gather, process and record concentration data, and actuate alarms.

INTRODUCTION

Where toxic substances are used in industry there is a need to continually monitor their levels in the working environment. Methyl bromide (MeBr) is widely used as an insecticide in the grain storage industry, and current standards call for human exposure levels not to exceed 15 ppm, and average levels to be less than 5 ppm during each working day (American Conference of Governmental Industrial Hygienists, 1981). To be effective, such standards must be supported by suitable monitoring equipment, which, must itself meet rigorous standards.

To be suitable for monitoring MeBr in large workspaces, such as those associated with grain stores, instruments must operate continuously and be reliable, accurate, flame and tamper-proof, insensitive to likely contaminants and capable of monitoring at a number of representative points (or being cheaply replicated). Laboratory techniques (e.g. spectroscopic, photometric, chromatographic, infrared absorption) are generally too costly or their operation too complex for such industrial use. Some (e.g. thermal conductivity or interferometric) are not sufficiently sensitive or specific (Wohlgemuth, 1971). Chemical sensing using glass encapsulated or cassette packaged reagents, though ideal for spot checking, is unsuitable for continuous monitoring. Several instruments for detecting or measuring MeBr have been described in the literature (Call, 1952; Olah *et al.*, 1966; Avera, 1966; Nelson and Shapiro, 1971) but none adequately meets the above requirements.

A promising technique, combining economy and simple continuous operation with suitable sensitivity and robustness, uses platinum filament detectors, commonly used for detecting halogenated hydrocarbon refrigerants. The instrument described uses this sensing technique, coupled with frequent standardisation, to achieve high accuracy and reliability. It includes a sampling system whereby the concentration of methyl bromide may be determined at numerous points remote from the instrument. It meets the requirements listed above, with the exception that it responds to other halogenated hydrocarbons, including chloropicrin and dichlorvos.

THE MONITORING SYSTEM

The monitor uses a proprietry G.E.C. device (Catalogue number 1250K46700) consisting of an electrically heated platinum filament helically wound to form a cylinder approximately 2 mm in diameter and 4 mm long, surrounding an axial cathode maintained several hundred volts negative with respect to the filament (Anon., undated) Gas is passed axially through the sensor at a rate of 7.5-8.5 mL s^{-1} (a linear velocity of about 0.5 m s^{-1}) which conducts a small (microamp) current in the presence of gaseous halogens or halogenated hydrocarbons, including MeBr. Sensor current is proportional to halogen concentration, but the proportionality constant varies widely and is strongly influenced by previous exposure to halogenated compounds and to gas flow rate and filament power (Figs 1, 2, 3). Sensitivity is inversely related to cumulative exposure to halogens; new sensors display high sensitivity which deteriorates

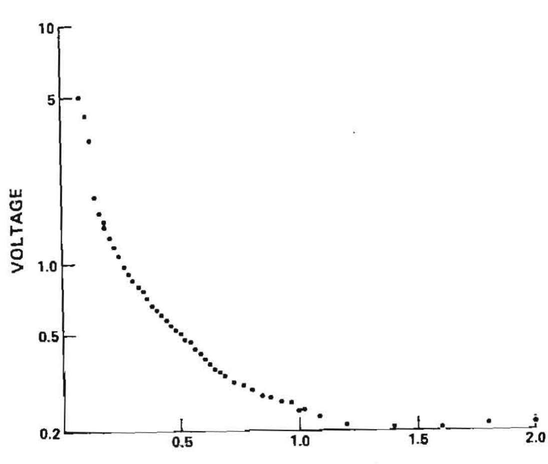

Fig. 1. Response of new sensor, to continuous exposure to 24 ppm MeBr v. time. (From Banks (1975) with permission)

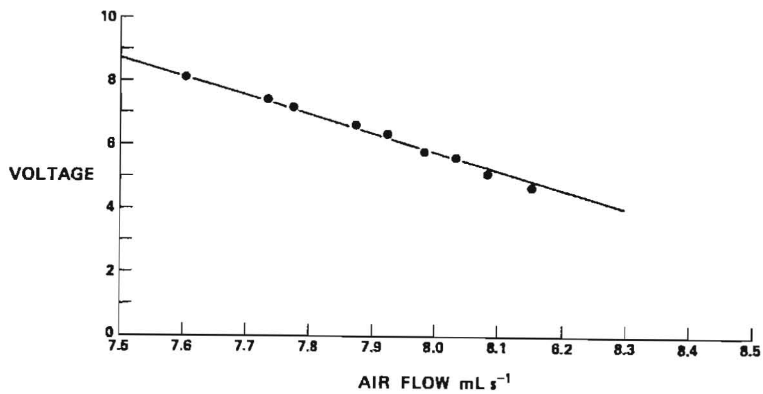

Fig. 2. Sensor response to constant 0.3 mL s^{-1} of 780 ppm MeBr diluted with varying air flow.

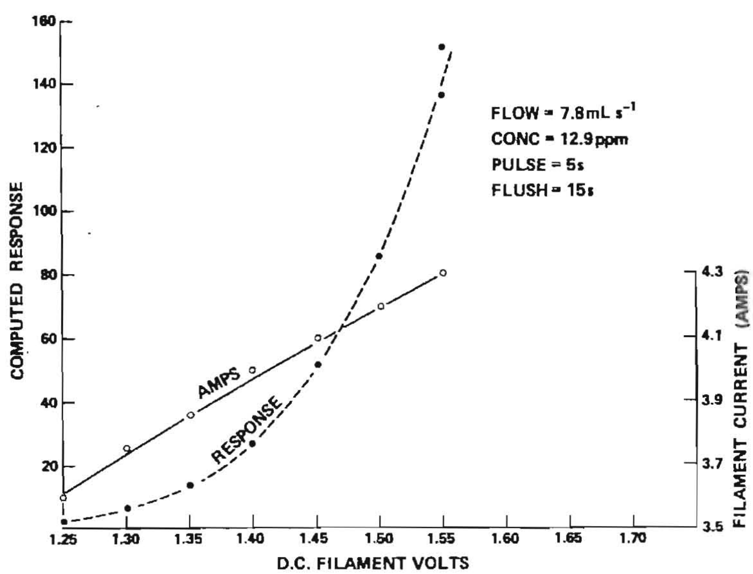

Fig. 3. Response and filament current v. filament voltage.

very rapidly with early exposure to MeBr (Fig. 1). After an initial stabilising period, short term sensitivity variation may be ignored if gas flow rate and filament power are held constant.

The detector is exposed cyclically to zero, sample and reference concentrations of MeBr. The measurement cycle consists of a period of 15 s during which the sensor and its plumbing are flushed with clean air, and a measure made of the sensor's response to this zero MeBr concentration, then a 5 s period in which it is exposed to the gas whose concentration is to be determined and a measure taken. This gas is drawn alternately through one of a number of sampling lines (12 in the prototype) leading to representative points in the work area or from a known reference concentration of MeBr used to calibrate the sensor.

Two diaphram pumps are used, one drawing samples through selected inlet lines, the other providing clean air for flushing the sensor and for use as a zero concentration MeBr standard. A reference concentration of MeBr is obtained by adding a small constant flow of a known high (around 1000 ppm) concentration to the air supplied by the latter pump.

Solenoid operated valves are used to switch gas streams. A bank of twelve selects the required one of twelve sample lines, and a bank of three selects sample, zero or reference gas. The configuration of valves and pumps is shown in Fig. 4.

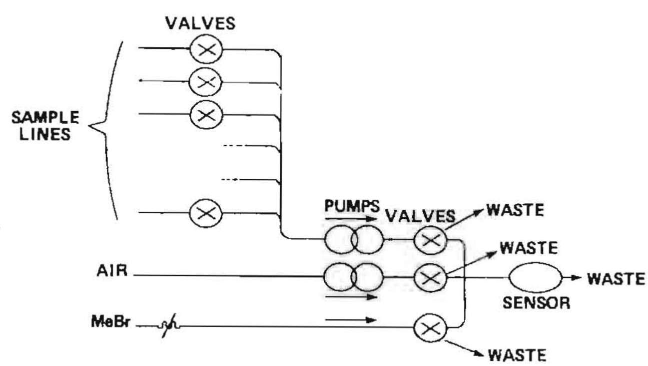

Fig. 4. Configuration of valves and pumps.

To correct for drift, the most recent 'zero' value is subtracted from each sample reading and from the most recent calibration value. The sample concentration is then calculated using the equation:

$$(\text{Sample concentration}) = \frac{(\text{Sample - zero})}{(\text{Reference - zero})} \times (\text{Reference concentration})$$

The system is managed by a Rockwell AIM 65 microcomputer which provides flexible control and calculating capability. It produces printed records, keeps time, generates alarms and monitors system performance. Upgraded from its minimum configuration by the addition of 1 K of random access memory (RAM) and the manufacturer's Maths Package chip, it costs around \$400 - \$500. The operating routines occupy part of an additional read only memory (EPROM) chip.

The AIM 65 controls a data acquisition chip (National ADC0816) comprising an 8 bit analogue to digital converter (ADC) and a separable 16 channel multiplexer. The ADC is connected without the multiplexer to the gas sensor, and the multiplexer used to operate the solenoid controlled gas valves through suitable drivers (Fig. 5). All switching is achieved using optically coupled solid state devices, so as to avoid the danger of mechanical switches arcing in potentially explosive environments.

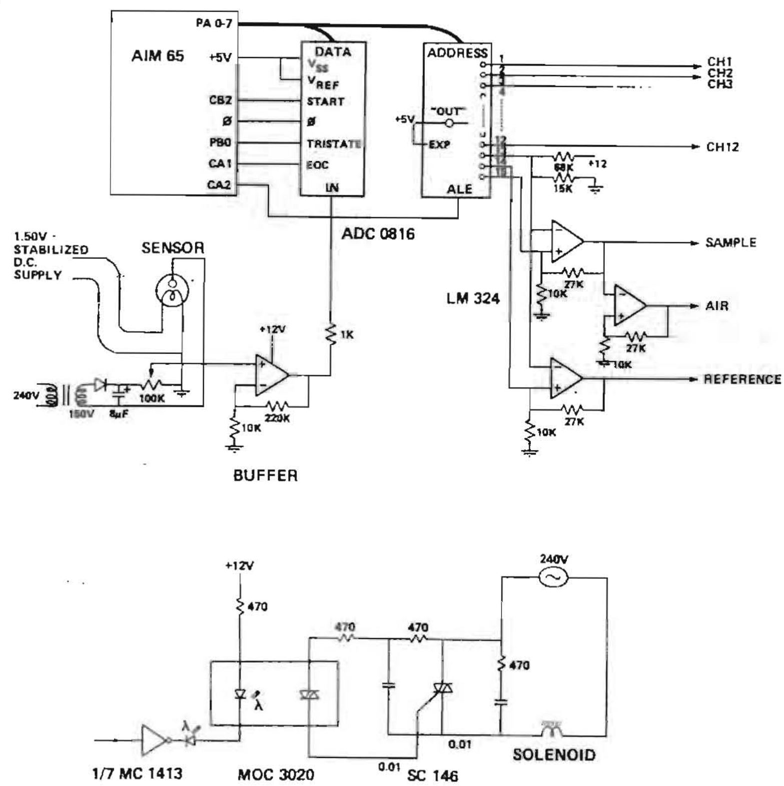

Fig. 5. (a) Measurement and valve control circuitry.
(b) Opto-isolated solenoid driver circuitry.

Various arithmetic and system checks are made:

1) To guard against the possibility that values lie outside the range of the ADC, the minimum and maximum ADC readings are considered invalid.
2) Since the sensitivity of the sensor varies widely, its response to the known reference concentration is checked to ensure that it is within acceptable limits. Out of bounds readings may also point to lack of reference gas or, because the reference

is obtained by dilution, improper adjustment of the dilution ratio.

3) Since the readings are obtained by subtraction of one ADC reading from another, and signals may be noisy, negative results are not uncommon when very low or zero concentrations are sampled. Slightly negative values of the unknown concentration are considered to be zero and printed as "-Ø". Significantly negative values are indicative of system malfunction, usually associated with variation in pumping speed.

4) Provision is made for two alarms, actuated when measured concentrations exceed predetermined thresholds.

Measured values of unknown concentrations are printed, together with time, day and channel number. Should system malfunction prevent the determination of a concentration, the nature of the malfunction is printed, details are retained in memory, and a system alarm actuated.

OPERATION

Only two mechanical operations are required to maintain the monitor, replacement of the sensor (perhaps weekly) and approximate adjustment of sensitivity (daily). Both are simple, the latter being guided by the AIM 65.

The prototype provides for sampling at up to twelve remote points, the present software allowing any of the twelve channels to be independently included or excluded from the monitoring programme. With additional valves, valve decoding and drivers, and after minor software changes, additional points may be monitored. The monitor takes 40 s to take each reading, so that monitoring large numbers of points may slow the reading rate of each unacceptably. Where greater channel capacity is required it may be preferable to use additional instruments rather than expanding a single unit. The Appendix lists the available machine commands, most of which are used to set operating parameters. If the initialising routines are amended to provide the parameters appropriate to each machine or fixed installation, the only input required from the operator will be the occasional setting of the clock.

The following are examples of a typical data printout (day number, time, channel number, measured concentration and alarm status), the range of system malfunction messages, and a listing of typical operating parameters:

128	12:14	3	8.7 *		
128	12:14	5	5.6 *	ADC TOO SLOW	REFERENCE CONC 10.0
128	12:15	6	-0	NEGATIVE REFERENCE	LO LEVEL ALARM 5.0
128	12:16	8	3.2	NEGATIVE SAMPLE	HI LEVEL ALARM 10.0
128	12:17	1	3.7	LOW SENSITIVITY	MIN SENSITIVITY 0.3
128	12:18	2	17.3**	HIGH SENSITIVITY	MAX SENSITIVITY 8.0
128	12:18	3	8.8 *		

PERFORMANCE

Fig. 6 is a plot of concentration measured by the system against concentration obtained by adding various small flows of 780 ppm MeBr to a constant flow of 7.8 mL s^{-1} of air. The flow rates of the two components were measured using variable area (gapmeter) flow meters. A MeBr concentration of 19 ppm was used as the reference.

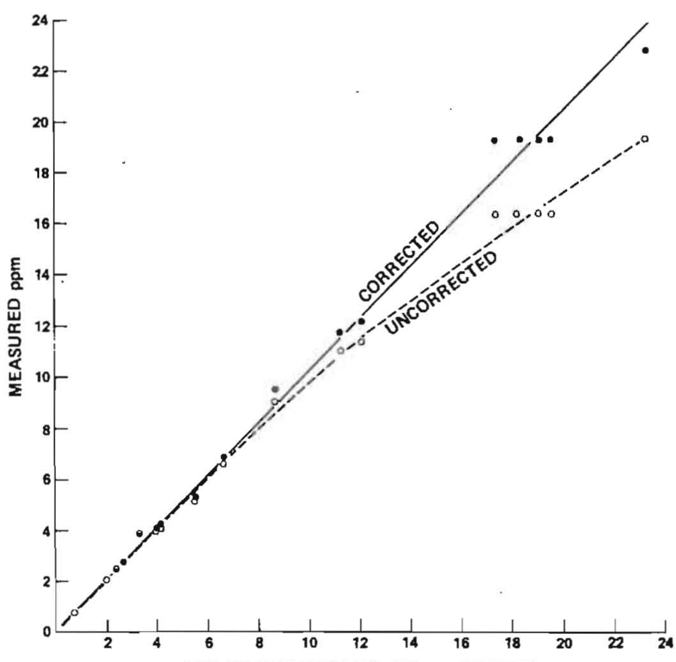

Fig. 6. Measured v. actual MeBr concentration.

The uncorrected curve in Fig. 6 illustrates the effect of the small change (less than 3%) in flow rate due to the varying dilution ratio. At these flow rates the detector response is reduced by about 6% for each 1% increase in flow (Fig. 2). The second curve in Fig. 6 is corrected for this flow variation and demonstrates well the sensitivity and accuracy of the instrument, even at concentrations below 1 ppm.

The ADC is capable of distinguishing between about 250 different levels, which for samples of up to 20 ppm limits accuracy when using a reference of around 10 ppm, to about $\pm$ 0.2 ppm. When the sensor has deteriorated to its worst acceptable level, accuracy is reduced to about $\pm$ 0.5 ppm. Although the sensor itself, given stable operating conditions, is capable of much better precision, its responsiveness to variations in flow rate and filament power renders this precision unattainable in a simple system such as that described here.

The life span of the sensor is dependent on cumulative exposure to halogenated compounds, and in low level monitoring applications is expected to be at least a working week. Various means described below may be employed to extend this life.

FURTHER DEVELOPMENT

A ratiometric measurement process, in which an unknown is compared with a reference using a linear instrument, is very robust. It does however rely on the linearity and short term stability of the sensing element. The sensor employed in the present instrument displays exceptional linearity, and sensitivity, but its stability is very poor, as indicated by Figs 1, 2 and 3. Cumulative exposure to halogens, increased flow rates and reduced filament power all reduce sensitivity.

It is likely that sensor temperature is a primary determinant of short term sensitivity to MeBr, and that flow rate and filament power affect sensitivity by varying sensor temperature. Since filament power may be readily controlled stability may be improved by maintaining constant temperature. Variation in filament temperature might be reduced by monitoring it using an infrared sensitive diode or other device positioned with a view of the filament, or using the very slight change in filament resistance with temperature, and varying filament current accordingly. (Although the effect of varying flow rate will be considerably reduced, the rate of delivery of halogenated molecules to the sensor will continue to be flow dependent.) Control of filament temperature may also permit the excessive sensitivity of new devices to be reduced to acceptable levels. The different cooling effects of alternative atmospheres, particularly those rich in carbon dioxide, will also call for control of filament temperature in some applications.

The quantities of reference gas (around 10 ppm) required dictate that it be obtained by dilution. The use of a dilute source (about 800 ppm in the prototype), further diluted in the instrument is unsatisfactory however, since both dilution ratios are subject to uncertainty. Adding the already dilute source to the zero concentration air stream also changes flow rate significantly, detracting from the instrument's standardisation. The use of a far smaller flow of 100% MeBr, precisely determined by a permeation device or calibrated leak, diluted with the existing air stream would enhance accuracy and reliability.

The sensor life is determined largely by cumulative exposure to halogens, the reference gas often constituting the major source. In such cases less frequent standardising will directly increase operating life. The sensor may need calibrating only rarely, other than when a significant level of MeBr is detected. Depending on the levels of MeBr in the samples gases, better calibrating strategies may increase operating life considerably.

In view of the excellent performance of the sensor under ideal conditions there may be advantage in diluting the sample gas stream and using a reference of far lower concentration. Sensor life span will be increased thereby, and stability enhanced.

CONCLUSION

The system described measures MeBr concentrations in the range from zero to greater than 20 ppm with an accuracy better than $\pm$ 0.5 ppm, and the measures are directly traceable to standards. It also monitors its own performance.

The system is suitable for monitoring concentrations at numerous points in large work areas, and actuating alarms when predetermined levels are exceeded. It produces permanent printed records of all measured concentrations, system malfunctions and alarm states, and is easily operated and controlled by unskilled users.

ACKNOWLEDGEMENTS

The sensor was initially appraised by H.J. Banks who also proposed the zeroing and referencing procedure, the subject of Australian Patent Application No 78722/75.

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APPENDIX I - OPERATING PROCEDURES

The software routines were written to operate on a standard AIM 65 microcomputer with 4k of RAM and the Rockwell AIM 65 Maths Package chip. The following operations are provided. More details, including program listings, are available from the author.

(1) Initialise
- Clock is reset to time zero, day zero
- Monitor reverts to sampling all twelve channels
- Operating parameters are set to: -
  - (a) Nominal reference concentration : 10 ppm
  - (b) Low level alarm : 5 ppm
  - (c) High level alarm : 15 ppm
  - (d) Minimum detector sensitivity : 0.3 times nominal
  - (e) Maximum detector sensitivity : 8 times nominal.

(2) Display and print current operating parameters
(3) Change nominal reference concentration
(4) Change low level alarm
(5) Change high level alarm
(6) Change minimum detector sensitivity
(7) Change maximum detector sensitivity
(8) Continue (Return to normal operation after any disruption. The system must have been initialised previously.)
(9) Set clock.
(10) Individual channels are enabled or disabled by storing ØØ or Ø1 respectively at address locations OFA1 to OFAC, corresponding with channel 1 to channel 12.

SESSION 13.

RAPPORTEURS REPORTS OF SESSIONS 9 TO 12 (2ND WEEK)

Reports by:

Dr E.G. Jay	-	United States of America
Dr H.J. Banks	-	Australia
Dr J. Desmarchelier	-	Australia
Dr D.E. Evans	-	Australia
Mr B.E. Ripp (Moderator)	-	Australia

Rapporteur Report

Session 9

The Bunker System

Edward Jay

Dr Evans, in opening this session, pointed out that bunker storage is an adaptation of the age-old principle of hermetic storage. We saw on the field trip connected with this symposium that it is an adaptation of this principle, but an extensive one, far removed by modern technology from basic hermetic storage. Dr Evans also pointed out that this technique has moved from underground to above ground and in the paper presented by Mr Yates and Mr Sticka we learned that storing grain in underground situations is a labour intensive and costly process, at least in a developed country where labour costs are high. This paper also provides an interesting summary of grain storage techniques practiced in New South Wales over the years. The need for increasing bunker storage capacity was caused by the large increase in grain production in this state during the 1970's. Fortunately, these authors had the foresight to conduct preliminary studies with bunker storage in combination with phosphine fumigation, and were therefore familiar with this method when it was needed for storage of this increased production. The successful practice of this method has led to the conclusion that at least in New South Wales all new storage constructed will be of this type. Certainly, the cost analysis presented in this paper would tend to reinforce this decision.

Mr Cameron's paper on bunker storage in West Australia also included interesting information on the history of grain handling in this state. The West Australian bunker system differs in some respects from the New South Wales system in that portable steel walls are used for bunker construction. These walls facilitate the rapid movement of bunkers to areas where the crop exceeds the available storage space. The two systems also differ in the method of securing the PVC cover onto the base of the structure. Both New South Wales and West Australia depend on phosphine fumigation for insect control and this technique is apparently successful because of the gastight nature of these bunkers.

Dr Navarro's report on research with bunker storage in the desert differed from the other two presentations in that his method depends on the principle of hermetic storage to protect the grain from insect attack. This paper contains much valuable biological data and the most important point presented was that this approach resulted in moisture migration and fungus growth on top of the grain mass. This occurred despite the fact that the grain was placed in storage at a relatively dry moisture content of 11.6%. This growth apparently contributed to the depletion of the oxygen in the storage and the creation of a "semi-hermetic" atmosphere which was lethal to insects in the bulk. A small portion of the grain was damaged and this may well be the beginning of current research on what was described earlier by Dr Banks as "sacrifical storage".

In summary, we can see from Australian research that the combination of bunker storage with phosphine fumigation is a valid and less costly alternative to conventional storage for maintaining the quality of DRY grain. However, the Israeli experience with storing relatively dry grain in a desert situation should act as a warning to anyone wishing to adopt this technique in areas having extreme temperature fluctuations and/or for storing grain at a moisture content of 12% or more. Obviously additional study is needed prior to initiation of large scale bunker storage when these conditions are present.

RAPPORTEUR'S REPORT

SESSION 10

PHYSICAL METHODS

DR H J BANKS

This session dealt with the application of heating and cooling for the control of insects infesting grain. These two physical methods of insect control are of interest as possible alternative tactics giving pesticide-free grain. With the concentration of attention hitherto in this conference on the use of fumigants and other modified atmospheres, this session provided a timely reminder that there are other weapons available which may well have an important place in the overall fight against insects to provide sound grain.

Dr Evans started his presentation by pointing out that heat disinfestation techniques could be complementary to other techniques by providing rapid instream methods of disinfestation-a capability which techniques such as fumigation lack. He then described the background and development work on the fluidised bed system as an efficient and controllable system for heating grain to insecticidal temperatures. The fluidised bed method provides a means of rapid heat transfer to grain without the formation of unevenly heated regions. Dr Evans said that his proving trials during the scaling up work towards a large prototype installation were carried out with late larvae and pupae of *Rhyzopertha dominica*. These developmental stages of this species were the most tolerant to heat and thus were the most stringent test of the process. The temperature required to eliminate *R. dominica* in continuous flow systems was substantially less than that causing quality damage to wheat (less than 70°C).

The level of mortality attained in a treatment was a function of both temperature and time of exposure. With the knowledge of the form of this function, gained from laboratory studies, it is possible to choose a temperature-time regime which ensures insect kill, but no commodity damage.

Dr Evans described a series of experimental rigs used to investigate fluidized bed heat disinfestation, progressing from a small scale batch system, through semi-industrial scale continuous systems to a large 50 t.p.h. pilot plant installed at Dunolly, Victoria. Slides were shown of this plant and its workings described. The plant has been used successfully to disinfest artificially infested grain and is currently undergoing trials to optimize its performance and rectify the few remaining problems. The chairman of the session noted that it was good to see the fluidized bed concept translated into a working animal.

In the following paper Mr Elder detailed the development of refrigerated aeration as a technique for grain preservation in Australia. The survey described early trials in Dalby in Queensland, later and larger trials at Lah, Victoria and Brookstead, Qld, and finally and, most recently, the large scale application of the system at Gravesend, NSW.

The first trials demonstrated that it was necessary to provide a high air flow rate through the grain and insulation of the storage in order to achieve the target value of less than 15°C throughout the storage. The later trials, carried out on storages fully covered with polyurethane foam for insulation, achieved this target value throughout the storage but demonstarated the crucial importance of correct duct work layout to provide suitable air and cooling distribution patterns. The Brookstead bin is still routinely used with refrigeration for the preservation of malting barley. The Gravesend installation has not yet been successful in providing grain without insect infestation, but has not yet been run with the newly-configured air input and distribution system. In the two treatments to date one was inadequate because of inadequate air flow and the other was carried out in a part-filled bin. The ductwork was not optimized for this. Insects survived only in the peak of the grain bulks.

Mr Elder also showed the use of a mobile chiller for reduction of the temperature of pesticide treated grain, so that the life of the pesticide and the protection it afforded was prolonged. He said that modular units, either of the portable type or as installed at Gravesend, provided a means of introducing refrigerated aeration technology progressively.

The papers provoked considerable discusssion. In response to Dr Evans' paper, Dr Bond asked if the grain moisture content influenced the time required to heat disinfest grain. Dr Evans replied that this was indeed so. Cold moist grain takes longer to heat to insecticidal temperatures than warm dry grain. Figures for the difference were given in one of his recent papers.

Mr Kemp then asked about the possibility of development of cold tolerance by stored grain pests. Dr Evans replied that while it was theoretically possible, there are several factors which work against tolerance development. In particular, reproduction is very slow at low temperatures and so the time required for selection of tolerance is great. Because of the lack of geographic isolation, with continued mixing of selected and unselected populations through trade, small changes towards tolerance are unlikely to be established. Dr Banks then asked if there might be a change in pest species with use of cold. Dr Evans replied that while there may be a change in balance, there may not be an increase in total numbers. Thus the less cold-hardy may die, leaving adults of the more tolerant species, such as *Oryzaephilus surinamensis*, which is probably present in low numbers, more obvious. However at the low temperatures these species would still not be able to multiply. Mr Ripp noted that grain in Canada may be at $-10^{o}F$ and apparently the rusty grain beetle can survive there. He asked whether this was through cold tolerance or because of change they make themselves to the grain. Dr Evans replied that *Cryptolestes* and other pests survive through physiological acclimatation to the lowered temperatures, as the grain slowly cools. The aridity of the environment affects ability to survive low temperatures. Nevertheless small populations of *Cryptolestes*, *Sitophilus granarius* and *O. surinamensis* can withstand the conditions produced in Australia by ambient air or refrigerated aeration for 6 - 12 months, depending on the species and moisture content of the grain. Mr Elder added that, when the refrigerated aeration programme was initiated, it was thought necessary only to achieve conditions under which the insect pests did not multiply. He said that the question now is whether, at increased cost, the refrigerated plant should be designed to reach a much lower temperature to prevent the possible emergence of cold tolerance.

These two papers provide insights into two of the physical processes available for grain disinfestation producing a residue-free and high quality product. We must ask where these strategies fit into the integrated system for pest control. Where the use of one process influences the use of another. Thus we should note that while refrigeration or natural aeration may be combined effectively with pesticide use, refrigeration in the short term is directly antagonistic to either fumigation and controlled atmosphere use and to heat disinfestation. Cold grain is more difficult to disinfest with many gas processes and requires more energy to heat to adequate treatment temperatures. It is clear that careful consideration must be given to use of the various techniques available for insect control so that the ones chosen are compatible through the system.

RAPPORTEURS REPORT

Session 11

Dr J Desmarchelier

What I will do is three things. I'll make two specific comments on the papers, and secondly I'll attempt to summarize general themes, and thirdly I will identify what I regard as the controversial points in these papers.

SPECIFIC COMMENTS

I think Dr Bengston the Queenslander was typically reticent of the achievements he had achieved. Particularly with regard to control of insects in the field situation. You will recall that he did at the final stage of the routine, 20 silos and these were stored for 9 months. I think that Dr Bengston could haved stated just a little bit more clearly that the insecticides recommended for use in table 4 were applied in 20 silos at 20 sites throughout Australia and at the end of 9 months the number of insects in these 20 silos was zero. That's a high level of achievement.

A comment on Mr Kemp's paper. You will recall that the residues they found initially were below the calculated values, and they went back a couple of days later and sampled in the bin and found the residue to be close to the calculated value. We've done a lot of work on this theme in the Working Party on Grain Protectants, we find it's very difficult to sample grain that has just been treated with insecticide. The reason is, the vapour pressure is high immediately after application and it drops extremely rapidly. What is happening of course, is that in bulk grain a bit of insecticide from one grain has been absorbed by another, and your overall loss is nothing. But when you get a sample, take it off the belt, run it down a few conveyors, put it out, spread it out, there is a lot of opportunity for vapour loss, and in fact I once showed that by varying the size of your container you could get any figure that you wanted for this stability of the compound. I am absolutely convinced that the residue from the probe sample, is in fact the true figure.

GENERAL COMMENTS

Look I hope that the author's won't be annoyed, but I've picked out some general themes. The first one is V.I.P.

V - for Versatile: here we have a lot of insecticides; They have different chemical properties, that is VERSATILE. The rate can be varied depending on what you want. That can be fitted in with other things. Your applications can be varied. We had three different types of applications discussed. A lot of versatility in that procedure.

I won't talk too much about INTEGRATION because Dr Evans will be very happy, but this came out in a lot of papers. Integration of hygiene with workers supervision is always an essential part of pesticide usage, and of course of fumigant usage. And P - Here is PRACTICAL: just one example is the ULV work. One of the reasons for it's adoption was the practical reason in that the worker is exposed to a little bit of insecticide. I think that this practical theme came through after these talks.

I've got another acronym. We can write this either as LEGS (Laboratory, Extension, General Use, Supervision) but to show that ones not more important than another, we can also write that as GELS. I prefer LEGS, I think it's something a little easier to grasp. What we have here, in all these talks, is work done in the laboratory (L), the extension (E), and the general use under supervision (G,S), I make the point that, in all these talks, and that was the impressive part, these things aren't in isolation. This is highly important and was brought out in an example by Mr Grant who is using all these things. But it is not good developing something in the laboratory and then forgetting about it. It is very much an ongoing programme, and we saw that in all talks, and of course it is not something that is restricted to pesticides.

CONTROVERSIAL POINTS

Now we have come to what I will call Controversial Points.

Dr Bengston as a Statistician actually gave us a number here - now I will quote him: "absence of field infestation is not strong evidence of success unless testing involves numerous, greater than 20, storages," and so that means that if you are going to do something, and say it works, you've got all your laboratory results, so it should work. If you are going to go out in the field and say this theme actually works in practice, you've got to do that 20 times. Well I think that is a very brave statement, a very interesting statement, it is also a statement I think that applies to pesticides, and it applies to other things as well. The second point I've put in here, the importance of supervision, the importance of the practical, and Mr Grant made some points, he will in no way be insulted when I say that that was a repetition of a point made by Mr Calverley, this work of motivation, this extension, this whole practical business is terribly important. Nobody involved in this work would deny that. But is that reflected in our literature, in our publications, do we ever see any publications of this type, is work motivation a sign, are we neglecting that in our own aid programmes? Is it something we should do, can we do anything to increase this awareness throughout the industry?

The third point again concerns Mr Grant's theme that grain protectants have no future. I made a list of Mr Grant's points as follows:

Nobody will accept the residues.

If they did, the insects would become resistant.

And in any case the chemical companies could not be bothered, - that's roughly the three points.

Mr Elder subsequently pointed out that these points were made 10 years ago, let's take the last two points. Insects resistance and the chemical companies won't do the work. The work outlined by Dr Bengston would seem to contradict the statement made by Mr Grant, certainly contradicts a similar statement made 10 years ago and we question if that applies to what is going to happen in 10 years from now. The residues - I'll make my comment then you can attack me. There are I think two aspects of the residue problem, not completely separated, and we might call them the sociological and the toxicological aspect. An example of the sociological aspect is 2,4,5 T (the herbicide) that is associated in everybody's minds with Agent Orange and the Vietnam War. We've got to the point that whatever the toxicology, sociological pressure is so strong, that it over-rides the toxicological. The toxicological with any chemical, yes let's stress chemical not grain protectant, with any chemical, is only weighted on the safety factor and let's say that the safety factor is roughly the toxic amount over the amount of actual residues. So there is a toxic amount, amount that causes harm, and the amount that you get in food. Now with the pesticides, that is roughly in the range of a thousandth to a billionth. When you come to something like Agent Orange, this figure can become irrelevant, does'nt really matter if it's 10^{-9} or 10^{-3}. At the moment, people in controlled atmospheres and phosphine say, yes, "chemical free" is the push for our product, but I think that the sociological pressure on protectants could equally well apply to sealers, could equally apply to Phosphine. Let me give you an example, sealers: we can think of vinyl chloride in PVC and that's being extensively reduced, and the safety factor has been pushed up, but it's never pushed up to infinity. I raise the point, I think that the chemical industry, whether it be in sealant or in protectant, is in the same boat, and that is certainly the case for Phosphine. Phosphine has a large safety factor. Sociologically, let me just give you one example, that does apply to fumigants, and of course that fumigant hydrogen cyanide was also the gas that was used in the concentration camp. There you have a sociological reason why hydrogen cyanide is not used on grain in Germany and Israel. With Phosphine it is not impossible to imagine a comedy of human errors that could lead, through bad formulations, terrible practice, to a few simultaneous disasters. You think of the record of human stupidity that has associated 2,4,5 T with the Vietnam War and Seveso, that is so improbable, has to be absolutely impossible, except that it happened.

The fourth point concerns the ULV application. There is a lot of literature for discussion on how much grain you have to treat. I think it is tied in with your Pesticide, how far it moves from one grain to another. It's tied in with the method of application, if the grain goes up an auger, or if you apply pesticide somewhere else, with your insects, how much your insects move around, this is a huge area, and is covered by a number of facts - so I will leave it at that.

DISCUSSION

Mr Ripp: Who would like to take up some of those points?

Mr C Sierakowski: Jim, it was very interesting to listen to your comments, but actually I would like you to commit yourself on one side or the other, because you gave us a broad view of implications, but you really didn't say, and I don't want any radical views, probably most of us are not interested in these, but are you speaking for chemicals and chemical control or against chemicals. Your comments were far reaching.

Dr J Desmarchelier: I think the world is a big place, there is a lot of grain. We are speaking for chemicals, yes I'm speaking for chemicals, silo sealants and plastics are also chemicals. They have in principal the same problem as grain protectants. I believe it is a matter of this safety factor if you like. Your toxic amount over your actual amount. There is this-ever-to-be-increased factor of infinity which will never be attained and there is also your cost benefit ratio. In these circumstances, whatever they be, the pesticide does so and so and so and so, your term does so and so and so and so, in this particular circumstance this or that pesticide has been used. For example on a ship or at a terminal, whether importing or exporting - and Kwinana is an exception here, - but I'm thinking of Hamburg or Sydney, or somewhere like that. There will always be a need for a quick method of disinfestation, and that is because your costs are so enormous, and we could never ignore costs, and this applies even more in developing countries than to the developed countries, where the amount of food is dependent on the costs. My personal view is that there will be a place for pesticides for a long time, and it's our job then to be increasing this safety factor. In other words we've got to be selecting pesticides that are more and more degradable, let's say the Methacriphos type where you can't analyse, you can't detect any residues on bread. They are certainly there at some level or other, true, or it's into your formulation type pesticide, the safety for those that work with formulations. The chemical industry is extremely versatile as we've seen in the sealant work, and I personally believe that in 10 years time we will be able to, if we want to, use almost any pesticide in such a way that it will leave no residues in the product. Now I can do that at the moment in the Laboratory, but can't in the industry at this stage. My theory is, let's adopt the Chairman's expression of horses for courses - pesticides for specific uses.

Mr Webley: I think the situation is that there is a lot more insecticides available than there were 10 years ago. I mean basically the insecticides we are talking about are the same insecticides that we were talking 10 years ago, because by 1973 most of the Pyrethroids were already in existence. I feel that Dr Bengston gave us a list of pesticides but many of them were equivalent. I mean Chlorpyrifos-Methyl = Pirimiphos-Methyl = Fenitrothion basically, and Cypermethrin = Cyfluthrin = Deltamethrin. I think that when you look at it from that point of view, you will see the number is in fact very small, and it is the same insecticides that we've been talking about for a long time, so I don't feel that, with the knowledge I have, there is any optimism, but there are a lot of new compounds out there waiting if the compounds that are used at the present moment should fail.

Dr J Desmarchelier: I think there are two points, you did leave out the two that were different, Methacrifos and Carbaryl. No way in the world could you equate methacrifos with actellic for example. Pyrethroids, yes there are similarities, the difference is, of course, I'd say that Permethrin is the same as some other Pyrethroid, but then the insects don't necessarily see it that way. We've had a lot of specific resistance to Malathion, and somehow or other it doesn't turn up to resistance to Fenitrothion, and in other types of course it does. We certainly haven't developed an unlimited amount of insecticides, but there are at least more than you would have thought. Okay, we are not in a position to judge, and I think it is also a point that applies to atmospheres, you know you are sealing the storage and this can be used for phosphine or it can be used for carbon dioxide and can be used for Nitrogen, and I think the industry is very carefully not committing itself to one theme. One might say all gases are the same, but they're really not. It could be with these pesticides that we will find cross-resistance, but we may not. We would certainly like to be and I think it's our job to get with finding alternative classes. The obvious one is to develop something of an insect growth regulator and an ecdysone inhibitor. We must remember that it's a big field and sometimes small changes can make a complete difference to these insects bio-chemistry.

Mr G Corbett: Could I take up another point made in the report, and this was on the number of field trials, necessary before you can say something with certainty. I think the figure of 20 was mentioned. I'd very much like to hear of any other comments on this. Because when anything new is being introduced in an FAO project, for instance, if we are trying a new improved farm bin, or a new procedure of any kind, it matters a lot how many times we have to replicate that in a one or a two year project, to be sure that we've got reasonable results. You said it might be controversial, I would like to hear any other comments on the number of field trials of that type that need to be under-taken to arrive at a definite result.

Dr J Desmarchelier: Perhaps I can call on the gentleman that might just like to respond.

Dr M Bengston: Look Mr Chairman, my head is clearly on the chopping block, we've given an estimate when clearly it depends upon the variation in your system. Now that is a Biologist's estimate of the variation among field occurrences of infestation, I guess within Australia. With a precision engineering process, presumably if you did it once you would be there. We of course in this estimate of 20, would pick up the frequency with which things will go wrong. You may have heard the statement 'if they can go wrong they will go wrong' and that is the other thing that we pick up. Given the diversity of things, even within Australia, I would hate to come less than 20.

Dr J Desmarchelier: Now would anyone else like to take up an answer or make a comment on this?

Mr G Corbett: Nice simple answer.

Dr J Desmarchelier: Well I will make a comment then, I'll reiterate what Peter Annis said. You remember that the first work on sheeted stacks done in Indonesia, and I think the first six stacks were completely successful. So there you had six out of six, terrific, and then they did it three more times and there was one failure, or something like that. There is a lot of work I know, but I think it is a terribly important point to cover. I would tend to support Dr Bengston that you need 20.

Dr M Bengston: Could I, Mr Chairman, just make a more general comment about grain protectants. My impression from talking to chemical companies gives mention to growth regulators, the possibilities of new types. It seems to us at the moment, that we have chemicals in the pipeline as grain protectants, which will take us to the year 2000.

Certainly they are eminently practicable things to use. It's my guess that their future depends upon consumer attitude towards residues. We can win the resistance battle given the technology and the input of resources. Market residues are, I find, impossible to predict. Within Australia we are currently adopting the philosophy, that there are genuine concerns about residues. It would be though, putting it the other way around, irresponsible of us to stop the development of a very useful alternative.

Dr S Navarro: We were talking here about Controlled Atmosphere, which is one of the long well known chemical control measures, and I was working on chemical pesticides used, specifically toxic chemicals. I would feel some depression because everybody was most successful with sealant application of this method in this wonderful country. But my general comment is related to a number of situations where we need the chemicals - as in many South American countries - if we go with the application of this controlled atmospheres into practice, and the full physical control method is in the silo with the gastight sealing. It is in this country invasion of insects coming around the field - and physical control would not work in this case, - then we must use chemicals in order to provide a surface treatment to eliminate the invasion or the entry of these insects into the silo. Even the simplest one, aeration, which is used in other parts of the world, is not practical. Then the use of chemicals in this instance, I think, has a very great value, as a supplement to controlled atmosphere techniques.

Dr Evans: If I could make a brief comment on Dr Navarro's comment - one of the things that we find in developing sealing, is that it has a degree of insect proofing. And we obviously come back to this business of, how many times do you do an experiment before you actually know that you've got insect proofness. In fact, how do you measure insect proofness? Because whenever you put something out in the field, you can't really measure the invasion pressure. You've got statistics involved with whether they find the hole, you've got statistics involved with how many find the hole and the assessment of this sort of thing, is very difficult. We have done experiments under high invasion pressure and the Indonesian experiment that Peter Annis talked about was one of these. Where the whole store was filled with insects, and we had a sealed system, which for six months protected that grain against invasion of unknown tropical humid invasion pressure. Now, I believe that this is something towards a physical process which doesn't require chemicals, but on the other side of the coin, I'm sure it would be much assisted by the judicious use of chemicals, to provide less invasion pressure, and so increase the chances of always being successful. Here we have to make the balance of how much of one thing you use, and how much of the other, and obviously you use both.

Mr Ripp: I think I'd just like to make a point on the chemical thing that the sociological pressure Jim talks about is real to a degree, and our view is, if we can avoid using residual chemicals, we should do so. If we move into controlled atmosphere, as an example, aeration or refrigeration or other physical means, and cut out the chemical usage in grain storage, the influence on total chemical usage throughout the world, I believe, would hardly be noticed. Because the total amount of chemicals that's used on grain is very very small. I don't think there is any alternative answer to the question, although we'd only use chemicals as Jim indicates, there is just no doubt about it, they are a valuable commodity.

RAPPORTEUR'S REPORT

SESSION 12

INTEGRATION

Dr. D. E. Evans

The feasibility of using very low dosages of phosphine (0.0303 mg/l) in hermetic pit-storages was indicated by C P F de Lima. Storage periods of 3 - 4 years, typical for emergency or famine reserves, allow adequate CT products to accumulate and, thereby, complete kills to be obtained. Combining hermetic storage and the use of low concentrations of phosphine may be attractive when facilities for supplying N_2 or CO_2 enriched atmospheres are lacking.

H. G. Greening described experiments to evaluate the influence of "PVC-sheeting" on the efficacy of carbon disulphide and phosphine applied in unsealed circa 60 t farm bins. Carbon disulphide was effective only in "sheeted" bins. Although insects caged within both sheeted and unsheeted bins did not survive exposure to phosphine at rates of 2, 6 and 12 g t^{-1}, live, free-living adult *Tribolium castaneum* were found, after treatment, near the grain outlets and auger chutes of "unsheeted" bins. In comparisons of phosphine formulations applied at 6 g t^{-1}, live *Tribolium castaneum* and *Sitophilus oryzae* were again found after treatment. The author claimed that such insects had found their way into the bins after treatment. Others, however, argued strongly that such insects had survived the treatment and further that failure to sheet and to seal farm bins adequately would result in sub-lethal dosing and, hence, selection for resistance.

The methods used to promote the concept of silo sealing on farms in Western Australia were outlined by D. A. Chantler. Several species of grain pest had been "declared" illegal and farmers were now required to take remedial action, when such pests were found by inspectors of the Agriculture Protection Board. Sealing was mostly undertaken by specialist contractors working to high standards. As a result of the campaign, most new bins were designed and built to high standards of gastightness. In spite of conservatism and the costs involved, about $10 per tonne which is, however, partially offset by reduced treatment costs, there had been an excellent response to the campaign, because of the combined efforts and good will of the growers, the contractors, the Protection Board and bodies such as CBH.

J. A. Conway and G. Mohiuddin described trials to evaluate the efficacy of fumigation in silos of the Ministry of Food, Bangladesh, whose suitability for fumigation was suspect because of structural faults. Of several methods of application, only conventional admixing of phosphine (at 2 g m^{-3}) during inloading of the grain, combined with exposure periods of at least 7 days, provided adequate *CT* products. Although the presence of phosphine-resistant insects in 'go-downs' in Bangladesh was mentioned, no evidence was produced to link less-than-adequate *CT* products in silo fumigation with resistance. However, the potential for inadequate fumigation in leaky silos to provide sub-lethal dosing and hence selection for resistance was clear.

M. Rahmin, S. P. Tee and B. L. Parker drew attention to work in Malaysia on the potential of slow-release pesticide strips for protecting milled rice within small packages. PVC formulations impregnated with malathion or chlorpyriphos gave protection against *Sitophilus oryzae* for up to 6 months. Further work is needed to determine, through more rigorous experimentation, the pesticide dosage required, and to consider factors such as the influence of the PVC carrier on grain, and sorption of the pesticide by the commodity. An important point not mentioned by the authors is the possibility of selecting for resistance unless the lethal dose is maintained for somewhat longer than the expected shelf life of the packaged commodity.

E. J. Bond, in considering the place of fumigants in integrated pest management, emphasised the very limited number of fumigants available and, consequently, the need to use such scarce resources wisely. The erection of increasingly stringent health criteria for registration, lack of knowledge of the mammalian toxicity of some of the older materials, and the ever increasing sensitivity of analytical methods may lead to some of the older fumigants being withdrawn. The development of resistance to fumigants poses a further threat, the reality of which is evidenced by the occurrence of high levels of resistance to phosphine in Bangladesh. There is an urgent need to know more about the nature of resistance to fumigants, and to ensure that fumigants may continue to be used within a framework of integrated pest management. No indication was given, however, either as to what sort of research could be undertaken to facilitate this - perhaps modelling and simulation as is being done with insecticides - or to consider likely technical and operation interactions between fumigation and other methods of control.

D. A. Chantler and J. A. Ritchie outlined the composition and activities of the Grain Weevil Liaison Committee in Western Australia. Inspection of farm storages and remedial action on farms, i.e. at the beginning of the grain pipe-line, has reduced the number of insects reaching the Bulk Handling Authority and successfully dealt with specific problems such as the occurrence of multiple resistant *Rhyzopertha* and outbreaks of *Trogoderma variabile*. The sealing of farm bins, and the more effective fumigation achieved thereby, has provided a means of dealing with pests resistant to grain protectants. The authors stressed that close co-operation and excellent rapport between growers, bulk handling authority, industry and government bodies, in a situation where grower representatives had the greatest voting power, had been central to the Committee's effectiveness. I believe that such co-operation and rapport in the work of the Committee serves as a valuable lesson to all of us involved in attempting to integrate pest control measures.

From a practical viewpoint, R. Grant reviewed the factors needed to ensure effective integrated pest management. Success depends on the activities and expertise of growers, Agriculture Protection Boards, Bulk Handling Authorities and Transportation and Marketing Authorities. At the level of the Bulk Handling Authorities, adequate sampling for pests when the grain is received from growers, the choice of an appropriate control measure, and regular inspection during storage are of immediate importance. Underpinning these factors, are the proper design of storages and handling facilities with regard to hygiene, maintenance of appropriate temperature and moisture levels within grain bulks, and ease of pest control both on receival and during storage.

Although the papers presented all had merit, I feel that this Section of the Symposium did not achieve its aim, that is, to discuss the integration of controlled atmosphere storage and fumigation with other control measures to provide systems of pest and commodity management. I say this because some papers were concerned only with given methods of control (and should have been placed elsewhere) and others, while mentioning integrated pest management (IPM), listed ingredients rather than provided recipes for integration. The number of processes available for use as agents for pest control and quality maintenance is limited and, unless there are sudden breakthroughs, unlikely to change in the near future. It is therefore essential that both 'research' and 'industry' face reality and devote thought and resources to devising ways in which given methods of control, each having constraints, may be integrated into rational systems of pest and quality management sympathetic to but not dominated by current storage and handling procedures.

DISCUSSION

Moderator: (Mr B E Ripp)

Mr Woodcock: I've heard a few comments on the cost per tonne of sealing a grain storage on a farm. Perhaps Don Chantler can clarify the cost savings to the farmer, Because his costs of $10 per tonne are against C.B.H.'s much, much lower figure.

Don Chantler: Yes, certainly. I gave the figure of $10 per tonne based on bins of about 56 tonnes capacity.

When we actually promoted the concept of silo sealing on the farms we did a little bit of a sum for the farmers. What we showed him was that in an unsealed silo he was required to use up to 10 tablets per tonne of grain which in an average 56 tonne silo was of course, at 10 cents per tablet, $56.00. That was the cost of one fumigation, and we pointed out that over a normal year he would have to refumigate that silo three times - $168 total cost per year. We then told him that when he had sealed his silo he only needed, not 10 but 1 tablet per tonne to do the job. Same 56 tonne silo, instead of $56 - $5.60. We then said you won't have to do that three times, once will be sufficient, because the fumigation will have been totally effective and there will be no reinfestation because of purely the physical barrier of sealing the silo, which is very easy to prove. Then we have a saving of $160 per year. At the cost of around $500, if he carried out the sealing himself, in three years approximately he'd be saving £448, he would have recovered the cost of his sealing. The other thing was, it was pointed out, that silo sealing was a tax deduction, there was a tax incentive and again the big thing was with a sealed silo he was getting total control of all stages of all insects. In an unsealed silo, if he attempted to fumigate it, we felt he was just about wasting his money. That is the way we put it to the farmer.

Moderator: Would Graham Love like to comment on that. No, he's not here.

B Elder: I'd just like to ask if there is no tax concession on the use of the tablets?

Don Chântler: Yes, it would be the same.

B Elder: When the Inspectors go on to the farm with this one shot fumigation how do they ensure that reinfestation does not occur?

Don Chantler: We do not carry out fumigations as Inspectors of the Board. Fumigation is left up to the farmer, subject to our inspections.

B Elder: Well, how do you really inspect the silo?

Don Chantler: In a sealed silo, we take it that the silo is sealed, we do not break the seal to carry out an inspection. If it's one of the newer types that have got a self sealing hatch, we will obviously take a sample directly from the outlet - either the bagging off chute or the boot, which as you've heard is the area most prone to stored grain insect attack. I think it would be farily justified to say that if that particular area was clear of insects then the rest of the silo would be in a satisfactory state.

Moderator: There was a reference to 'Dryacide' throughout these proceedings. Dryacide will be discussed tomorrow on the bus to country facilities but has anybody got any questions left they would like to cover.

Nothing further on space sprays, residual sprays, weedicide, grain protectants, the role of hygiene, anything?

No questions, left in your minds?

Dr Evans: Well, if thats ample sufficiency perhaps I can take the opportunity to say a few brief words of thanks to

Mick Gayfer the Chairman and his fellow directors on the Board of Co-operative Bulk Handling Limited, to the sponsors of this Symposium, the Honourable members of the International Steering Committee. I think its often very difficult to measure the success or failure of such a Symposium, very difficult to measure intangibles such as how effective the information exhcange was, etc. I am sure that all the delegates to this Symposium will join with me in saying that this ten days have been extremely valuable. We have all learned a great deal. There has been something for everybody. We have gone from research to technology, to management. We've gone through the whole thing. I think particularly valuable has been the opportunity to meet with people of different countries, different backgrounds. To meet with people from different industries, from different parts of our own industry here in Australia. I certainly, and I'm sure everybody else have found it a very stimulating exercise and I think most important, we will be encouraged to continue our enthusiasm and to look forward to the third Symposium on this subject in a few years time. A very great compliment is that whoever thinks of sponsoring the next Symposium is going to have an extremely hard act to follow, because there is no doubt about it C.B.H. have done us proud, their hospitality has been tremendous, their organisation has been tremendous and they've certainly done a first class job in all respects. Please join me in thanking the Board of Directors of C.B.H., our Steering Committee and the sponsors of this Symposium.

Mr B E Ripp: Thank you David. To really wind up this Symposium now we need to look at the conclusions that can be drawn from this particular period of this week. Last week was specific Controlled Atmosphere and Fumigation and it looked after itself. To briefly review the first week, we agreed that research should continue into the production of effective atmospheres, above ground hermetic storage, biology of insects and the correlation of lab and field results, sealing methods and materials as well as the use of controlled atmosphere storage of grains with high moisture content.

This week has been more variety, although we couldn't escape from going a little more into controlled atmosphere and fumigation. The type of conclusions that we can bring out of this week strike me in two particular aspects, firstly that the industry as a whole must applaud and support the efforts of the researchers and their organisations in to all possible methods of grain quality preservation, whether it be used immediately or not. Their research must go on and we must be aware of all the possible methods that we can turn to given perhaps a different set of circumstances.

The second one that stands out and pointed out in detail by Geoff Corbett is the need for integration of sealing for controlled atmosphere and fumigation into grain storage systems. It has also been clear that further research is necessary into fumigant resistance, the integration of Pest Management processes, thermal disinfestation and refrigerated aeration. International co-operation is necessary on detailed investigation into data on carcinogens, toxicity and toxicology of fumigants as well as monitoring of very low concentrations and the effects of low residues on foodstuffs.

Have I missed any conclusions that should be noted from our time together?

No? Thank you.

O.K. - then to allow some time for the barley discussion to follow it behoves me to wind this up now, and from our point of view, from Co-operative Bulk Handling and the people involved in Co-operative Bulk Handling I would like to express their appreciation on behalf of our Chairman, and General Manager who are out this week with the shareholders in various parts of the country, and send their apologies for not being here for this particular part of the proceedings, but we are represented, or they are represented by Bill Huxley our Deputy Chairman of the Board. Would you show our appreciation on behalf of everybody concerned - Bill - to the Board, and to the staff of C.B.H. - the people of C.B.H. who have done all the work, the organisation of the set up over quite a long period of time and quite alot of effort. I can assure you the delegates, that each one of them enjoyed their participation, no matter how hard they worked and some of them did work - they enjoyed it and to see that you've enjoyed it during the proceedings is their reward and that applies to each and every one of us. To the sponsors for sharing the load of the Symposium, they helped tremendously. The authors, who actually make a Symposium possible by their participations and one of the tangibles that David mentioned, is the value of inter-action by seeing these people together, and this can't be measured.

To the Chairman of sessions and rapporteurs for their input of efficiency and knowledge seeing all the way through that the conduct and the continuity of the presentation and the windup sessions were presented in one hundred percent fashion. And lastly, but certainly not leastly to the Steering Committee, the people that through their efforts and their presence have added immeasureable dignity and validity to the contents of this Symposium, and their work is not finished yet, they will go on until such times as the proceedings are published. They will all be involved with that. I would express our deepest thanks to each and every one of those persons that I mentioned and I would ask you to join with me and thank each other.

Mr Huxley: It is quite unconventional to stand up but I feel more comfortable. I feel that I must pay tribute to the quality, the intellectual quality, the practical qualities that I have around me now and I believe that what we have done here over the last couple of weeks has been something in the nature of an olympic gathering on a higher scale. I think its even greater than olympics, because we are not looking just for personal glory, we are looking for the development of practical approaches to something which we believe is of great value to mankind and therefore I really believe that you all here and the others that have been with us, over the last week or so, working so hard, not only in the business sessions, but the strain that has been put on our constitutions at the weekends. You've done a tremendous job. We, the Board of Directors, I think probably had to be talked into the prospect of having an International Symposium here. We didn't know whether we could cope with it. But then again we didn't know the quality of the staff we had working for us. This is one of the most important things that has come out of this Symposium as far as I'm concerned. We knew we had alot of good men amongst our staff, we knew that they had alot of initiative, that they were very able, but the heights to which they could rise, I personally and I'm sure the Board, didn't realise until we saw the culmination of their efforts at the Symposium. So to you Eric, on behalf of your Committee, and also for the wonderful assistance that Jonathon gave and also to the International Committee that has organised this, we feel very proud of the work that has been done and I don't think if there was an occasion for another International Symposium in say, twenty or thirty years time, that we'd take alot of talking into it. (Laughter) Thank you all for being here and particularly Eric - thank your staff for the tremendous job that, we the Directors, feel that they have done.

Concludes.

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