

EXPERIMENTAL AND COMMERCIAL MODIFIED ATMOSPHERE TREATMENTS OF STORED GRAIN IN AUSTRALIA.

BANKS, H.J., ANNIS, P.C.,

CSIRO, Division of Entomology, P.O. Box 1700, Canberra City, A.C.T., Australia.

HENNING, R.C.,

State Wheat Board, P.O. Box 136, Toowoomba, Queensland, Australia.

WILSON, A.D.,

Grain Elevators Board of Victoria, G.P.O. Box 2289U, Melbourne, Vic., Australia.

ABSTRACT

Modified atmosphere techniques for grain storage have been under study in Australia for over 60 years but until recently have been widely used only during one brief period (1917-1919). Full scale testing of nitrogen-based atmospheres, started in 1972, and has led to development of a process, based on a tanker-delivered liquid nitrogen, similar to that developed by Snamprogetti in Italy. Since, under dry Australian conditions, the quality of grains such as wheat is maintained adequately in storage, the emphasis in the use of nitrogen-based systems has been on insect rather than quality control. More recently the use of CO₂ has been investigated. CO₂, at present, is the preferred atmosphere for insect control as it is easy to apply and, unlike nitrogen atmospheres, does not require a continuous input of gas after the initial purge if the storage is well sealed. CO₂-based atmospheres are now in use in Australia for insect control in stored grain. A summary of the unpublished experimental and commercial trials carried out with modified atmospheres in Australia is given. If insect control only is required, it should only be necessary to maintain the modified atmosphere until elimination of insects is achieved. The sealed fabric of the storage should then provide a barrier against reinfestation. This proposition is discussed and examples of storage free of insects for more than four months after brief modified atmosphere treatment are given.

INTRODUCTION

The technique of modified or controlled atmosphere storage of grain involves alteration of the concentrations of the normal atmospheric gases present in a storage so as to give an artificial atmosphere that is insecticidal and prevents mould growth and quality deterioration of the stored product. Two classes of externally generated modified atmosphere are available: low oxygen atmospheres, (generated by adding nitrogen, or the gas mixture resulting from burning hydrocarbons, to the store) or high CO₂ atmospheres (made by adding carbon dioxide). It should be noted that the technology of use of these atmospheres differs substantially from that of modified atmospheres generated by biological processes within a sealed structure, i.e. hermetic storage. Only externally generated systems are considered in this paper.

There has been a continuing interest in various forms of modified or controlled atmosphere storage of grains in Australia over many years: the first recorded large scale treatments were carried out in 1917. Although these were successful the process was not again used widely until 1979 when two grain storage organisations carried out commercial treatments with carbon dioxide. This use was based on experience gained during the 1970's from various full scale experimental treatments with either carbon dioxide or nitrogen as sources of the modified atmosphere. This paper summarises the details of the unpublished recent commercial and experimental applications of modified atmospheres to large bulks of stored grain (> 300 tonnes) in Australia. Evaluation of such treatments is an important step in the development of the technique into a routine system. The number of sites used in the basic experimental work was limited and application of the technique over a significant number of storages may reasonably expected to reveal problems not encountered previously.

The exposures to modified atmospheres currently believed to give complete insect mortality under Australian conditions are given in Table 1 (Banks and Annis, 1977; Banks, 1979).

TABLE 1

Exposure periods proposed for modified atmosphere disinfestation of grain (< 12% moisture content).

Atmosphere source	Initial target concentration in storage	Final target concentration in storage	Period of exposure within these limits
Carbon dioxide	> 70% CO ₂ in air	> 35% CO ₂ in air	> 10 days at 20°C
Nitrogen	< 1% O ₂ in N ₂	< 1% O ₂ in N ₂	> 6 weeks at 20°C ^α

α = Temperature dependent. For other temperatures see Banks and Annis (1977).

The CO₂ dosage regime given in Table 1 is referred to below as the 'target' regime and, if further gas is not added after the initial purging, implies a semilogarithmic gas loss rate of < 6.9% day⁻¹.

In Australia, most grains are harvested in a condition regarded as dry by world standards. The receipt of wheat, the major grain crop, into the central bulk handling system is not permitted if the moisture content is > 12%. Though the grain may often be warm in storage (> 30°C) the qualities required for most of its end uses (e.g. baking, animal feed, noodle-making) are adequately retained in air over long periods of storage in the absence of insect pests.

In commercial terms, germination may fall significantly for some commodities, notably malting barley, but most of the grain produced does not need to be viable. Since modified atmosphere storage is generally not required for quality control in Australia, it appears unnecessary to maintain the atmosphere for longer than needed to ensure complete mortality of all insect pests in the storage. A perfectly sealed storage should then, in theory, act as an insect-proof enclosure and prevent re-invasion of the stored grain by insects. However, the standard of sealing currently accepted (Banks and Annis, 1977, 1980), though high does allow for some imperfections in the enclosure. The degree of sealing is assessed by a pressure decay test. This test cannot differentiate between residual leakage resulting from a few large holes in the fabric or an equivalent leak resulting from the combined effect of many small ones. On a 2000 tonne-capacity storage cell, a typical storage unit in Australia, the imperfections may amount to about 1 cm^2 in total. It is thus possible that there may be a number of imperfections present of a size through which an insect can enter a store. The degree of protection which a store sealed to the required standard affords in practice results from a combination of a number of factors which, because of their random nature, cannot be assessed in the laboratory. These factors include the possible presence of a suitable hole through which an insect may enter, the presence of invading insects and the ability of the insects to locate the hole (see Barrer and Jay, 1980). Some data from commercial practice relevant to the assessment of the protection which can be expected is given below.

USE OF MODIFIED ATMOSPHERE TREATMENT OF GRAIN IN AUSTRALIA, 1917-1919

Externally generated atmospheres were used for disinfesting stored bagged grain in Australia during the period 1917-1919. This was possibly the first extensive use of such a system in the world (Winterbottom, 1922). Disruption of world trade by war had led to a substantial holding of grain in bag stacks around Australia. About 60,000 t wheat were treated with a low oxygen atmosphere generated from burning coke in a modified producer gas generator. At that time, carbon dioxide was thought to be the toxic agent to the insects and the generator was tuned to give maximum CO_2 output, although originally designed to produce carbon monoxide. Fig. 1 shows the arrangement for treatment of a bag stack. We know now (Bailey, 1965) that the lack of oxygen was probably the cause of death, although its action would have been assisted by the CO_2 present (see Bailey and Banks, 1980). After this highly successful operation, no further large scale use of controlled atmosphere disinfestation has been made for stored grain in Australia until the current experimental and commercial applications.

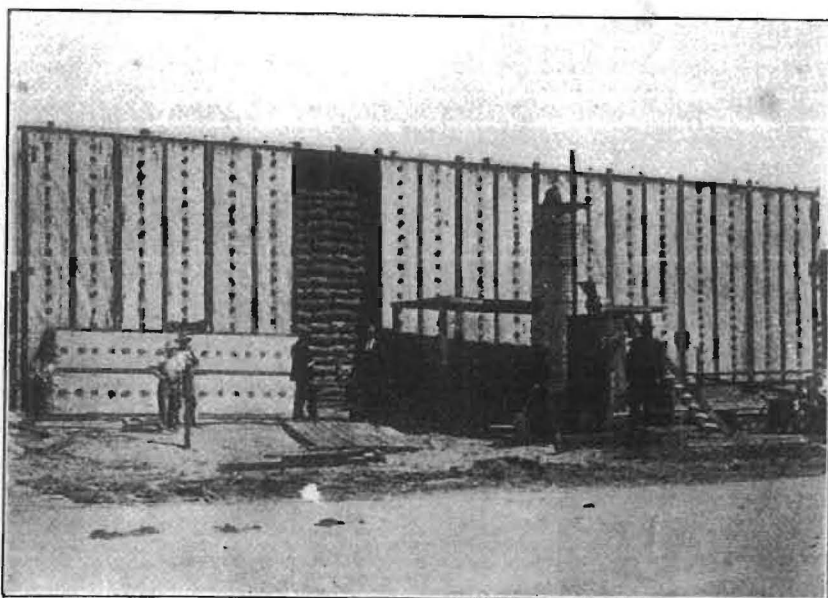


Fig. 1. A bag stack of wheat enclosed in wooden sheeting treated with bituminous materials for modified atmosphere treatment. In Australia during the period 1917-1919. Note the gas generator to the right of the picture (From Winterbottom, 1922).

PILOT COMMERCIAL USE OF MODIFIED ATMOSPHERES BASED ON LIQUID NITROGEN

In Australia, experimental trials under full scale storage conditions using tanker-delivered liquid nitrogen as a gas source were begun in 1972. The results of these trials, summarised by Bailey and Banks (1975) and Banks (1979), formed the basis for a publication (Banks and Annis, 1977) setting out how nitrogen atmospheres might be used commercially for treatment of bulk stored grain. The process is similar to that developed by Snamprogetti (Shejbal, 1979) in Italy. Several pilot commercial trials have been carried out using liquid nitrogen. These trials, summarised in Table 2 and 3, were conducted in general according to the procedures set out by Banks and Annis (1977): minor modifications were made as required by the equipment available. In two of these trials, the additional nitrogen required to maintain the low oxygen atmosphere after purging was initially added to the storage using a demand system set to keep a small positive pressure (120 Pa) within the storage. This system was later discarded in favour of a simple continuous gas input system, (Banks and Annis, 1977). The latter used less nitrogen than the former system to maintain the same oxygen level.

The efficiency of generation of the low oxygen atmosphere, can be assessed by comparison against a theoretical gas requirement where the interstitial atmosphere in the bulk is displaced by plug flow and the oxygen content in the headspace is reduced by a mixing process ('E₃' of Banks (1979)). The efficiencies of purging

TABLE 2.

Pilot commercial usage of modified atmospheres generated in stored bulk grain from liquid nitrogen. Details of the storage and initial addition of nitrogen.

Site, bin no.	Date of trial	Tonnage treated and commodity	Filling ratio	Purge rate ($\text{m}^3 \text{min}^{-1}$)	% O_2 at end of purge	Purging efficiency, E_3 (%)	Nitrogen used ^c ($\text{m}^3 \text{t}^{-1}$)	Carried out by
Tara, 2	1977	1870 wheat	0.96	0.7-0.9	2.0	72	0.81	State Wheat Board, Queensland
Rennie ^b , 2	1977	1890 wheat	0.97	1.0	1.0	55	1.11	Grain Elevators Board, Victoria
Tara, 2	1978	1880 wheat	0.95	0.8-1.5	1.5	65	0.93	State Wheat Board, Queensland
Tara, 3	1978	1870 wheat	0.96	0.8-1.5	2.8	61	0.92	State Wheat Board, Queensland
Macalister, 3	1978	1650 wheat	0.85	1.0	1.0	55	1.11	State Wheat Board, Queensland
Toowoomba, 2	1980	930 barley	0.99	1.2	0.4	65	1.26	Barley Marketing Board, Queensland
Toowoomba, 3	1980	930 barley	0.99	4.8	0.6	79	1.01	Barley Marketing Board, Queensland

^a = Efficiency, E_3 , as defined in Banks (1979).

^b = A second bin was also treated at this site and gave similar results.

^c = Gas usage giving the observed headspace oxygen levels. These figures are not directly comparable with those in Banks and Annis (1977) which are for 1% O_2 in the headspace.

TABLE 3.

Pilot commercial usage of modified atmospheres generated in stored bulk grain from liquid nitrogen. Details of sealing level of the storage and gas requirement to maintain the low oxygen atmosphere.

Site, bin no.	Date of trial	Pressure decay test, 1500-750 Pa (mins)	Filling ratio	Maximum % O ₂ maintained	Observed maintenance rate (m ³ day ⁻¹)	Predicted ^d maintenance rate (m ³ day ⁻¹)
Tara, 2	1977	102 ^a	0.96	1.1	30-35	26
Rennie, 2 ^b	1977	6.0	0.97	1.8	27	25
Tara, 2	1978	78 ^a	0.95	2.0	40 ^c	27
Tara, 3	1978	22	0.96	2.0	40 ^c	27
Macalister, 2	1978	19	0.85	> 2.0	> 36	47
Toowoomba, 2	1980	7.8	0.99	1.0	17	20
Toowoomba, 3	1980	7.8	0.99	1.0	17	20

^a = These values may be too high as they were taken during a period when the headspace was gaining heat.

^b = A second bin treated at this site gave similar results.

^c = This rate was used when the oxygen level was already > 2.0% in the bin and gave a slow reduction in oxygen level.

^d = Derived from Banks and Annis (1977, Table 3) by linear interpolation.

in these trials are given in Table 2 and are, in general, slightly less than obtained under experimental conditions (73-92%; Banks, 1979). It will be noted that even in the case where the purging was least efficient (55%, Rennie, 2), the saving in gas usage, and thus cost of treatment, to be achieved by raising the efficiency to the level found in experimental trials would be small. In this example, an improvement in efficiency from 55 to 75% represents a saving of only $0.2 \text{ m}^3 \text{ tonne}^{-1}$. The values of expected nitrogen usage for various filling ratios given by Banks and Annis (1977) were based on results obtained from trials using filling ratios of < 0.92 , and in practice, as the observed efficiencies given in Table 2 suggest, may be slightly low. After purging, the low oxygen atmospheres within the storages were maintained by addition of nitrogen to the headspace. The rates of input required are given in Table 3, together with pressure test values on the filled storages. The rates of gas input required are consistent with those given in Banks and Annis (1977).

At present only one series of treatments using liquid nitrogen as an atmosphere source is being carried out in Australia. These are still in progress. Shejbal (1979) observed that the germination of malting barley could be preserved for a longer period under nitrogen than in air storage. The current trial is designed to verify this observation under Australian conditions. In general, there is little current interest in Australia in use of nitrogen because, under the prevailing storage conditions, carbon dioxide-based systems are at present more attractive, both practically and economically.

EXPERIMENTAL AND COMMERCIAL APPLICATION OF CO_2 -BASED MODIFIED ATMOSPHERES

Experimental treatments using carbon-dioxide based systems

Banks (1979) provided details of the experimental trials carried out to that date by CSIRO with carbon dioxide in various types of bulk grain storage and transport vehicles. Wilson et al. (1980) described two pilot commercial treatments in welded steel bins and, in particular, demonstrated the need for gas recirculation during CO_2 use in sealed structures in order to avoid regions of inadequate CO_2 concentration in the upper parts of the storage. The Barley Marketing Board, Queensland (unpublished data) treated 950 tonnes of barley with 80% CO_2 using a purge rate of about 1 t h^{-1} in a sealed metal bin (pressure decay time for 1500-750 Pa, 16 mins). An initial CO_2 level of 79% was achieved with a subsequent decay rate of $1.8\% \text{ day}^{-1}$ and an overall gas usage of 1.08 tonnes CO_2 per 1000 tonnes barley. No detrimental effect of the CO_2 on the barley was observed.

While CO_2 is now being applied commercially in welded steel storage bins (see below) it is not yet used in other structures in Australia. Until recently, the main limitation to the more widespread use has been the lack of methods for

sealing other structures to an adequate level. This problem has now been overcome (Banks and Annis, 1980). The main constraints will now be financial, as the cost of sealing some structures can be high.

As a demonstration of the feasibility of using CO_2 in a large shed-type storage, a type commonly found in many grain handling systems, a 16,400 tonne-capacity shed filled with wheat was treated with this gas, after the store had been sealed as described in Banks et al. (1979). The gas was added through a perforated aeration duct which ran longitudinally along the centre of the grain bulk. After purging, the gas was recirculated to maintain a uniform atmosphere within the shed. No further CO_2 was added. Table 4 gives the general details of two treatments in successive storage seasons.

TABLE 4.

Details of CO_2 treatment of a large, sealed grain shed (Harden, N.S.W.).

Nominal shed capacity	16,400 t	
Total enclosed volume	33,100 m ³	
Season	1977-78	1978-79
Load	13,606 t wheat	16,144 t wheat
Pressure decay test (100-50 Pa)	5 mins	5 mins
CO_2 added	33 t	32 t
Rate of CO_2 applied	2.4 t CO_2 per 1000 t wheat	2.0 t CO_2 per 1000 t wheat
Maximum average CO_2	59% v/v	73 % v/v
Average CO_2 at 10 days	38% v/v	43 % v/v
Gas loss rate	4.5% day ⁻¹	5.2 % day ⁻¹
Purging efficiency (E_1) ^a	73%	92%
Gas addition rate	0.6 t h ⁻¹	2.8 t h ⁻¹

^a Calculated as in Banks (1979), assuming direct displacement of enclosed atmosphere by CO_2 .

The increased purging efficiency in the second year is largely attributable to slight alterations in the design of the ductwork and increased speed of purging. In the first season the maximum concentration of CO_2 achieved was lower than the target values (70%). However, an acceptable decay rate (i.e. < 6.9% day⁻¹), was attained showing that the shed was adequately sealed. In the second season, the target CO_2 regime was met. The average CO_2 levels in the storage over the first 15 days of the trial are shown in Fig. 2. The form of this curve is typical of the decay of CO_2 concentration which we have observed in various trials. There is an initial rapid decay shortly after purging and then a slower phase in which $\log [\text{CO}_2]$ is proportional to time. This semi-logarithmic relationship is used below to estimate the initial $[\text{CO}_2]$ obtained

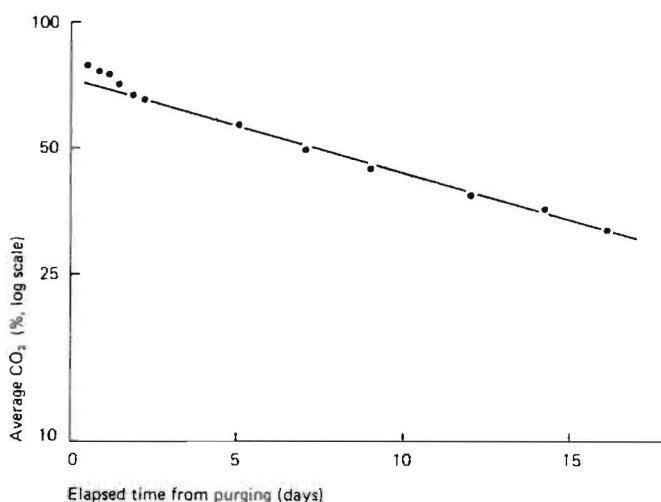


Fig. 2. Decay of average CO₂ concentration with time observed during the treatment of a sealed 16,400-tonne capacity shed as Table 4, 1978-79 season.

but the value calculated is slightly less than the true value because of the initial deviation from the relationship.

Commercial treatments with CO₂ in welded steel bins

In an adequately sealed storage, i.e. meeting the gastightness specification given in Banks and Annis (1977, 1980), it has been shown experimentally (Banks, 1979; Wilson et al., 1980 and above) that CO₂ may be applied in a 'one-shot' operation. That is, a rapid initial purge is used to give a high-CO₂ atmosphere within the storage. No further gas need be added. The target regime for insecticidal action (Table 1) can still be met, as gas loss is sufficiently restricted by the sealed enclosure. In contrast, nitrogen-based low oxygen atmospheres require addition of further gas to maintain oxygen levels below the target concentration during the required exposure period. The cost of the additional gas and its storage on site is significant in the overall economics of the process using nitrogen. The lack of need for such additional gas is the main commercial advantage which CO₂ has over nitrogen in the modes of use currently proposed in Australia. The details of commercial usage of CO₂ as a 'one-shot' system are given below. They confirm that the CO₂ application technique demonstrated experimentally (Banks, 1979; Wilson et al., 1980) can be duplicated in routine practice.

Two authorities in Australia concerned with bulk grain storage, the State Wheat Board, Queensland and the Grain Elevators Board, Victoria, have carried out commercial treatments of bulk wheat using CO₂. The carbon dioxide was supplied to the storage sites as a liquid and applied at the rate of $0.5-2 \text{ t h}^{-1}$ after vaporisation as described by Wilson et al. (1980). In Victoria, a single

TABLE 5.

Details of commercial treatment of bulk wheat, stored in sealed 2400 m³ capacity welded steel cells, with CO₂ for the 1979-80 storage season by State Wheat Board, Queensland.

Site, bin no.	Tonnage treated <i>a</i>	Pressure decay test 1500-750 Pa (mins)	CO ₂ used (t per 1000 t wheat)	Initial ^b % CO ₂	Decay ^b rate (% day ⁻¹)	Efficiency of purging (E ₁ , %) ^c	Period of storage after CO ₂ treatment (months)
Bell, 1	1811	11	1.04	74	5.1	76	12 ^e
Bell, 3	1831	8	1.03	77	8.3	78	11 ^{ef}
Brigalow, 3	1796	10	1.06	75	4.1	76	3
Brookstead, 2	1809	20	1.10	75	6.3	73	4.5
Cambooya, 3	1870	24	0.98	79	3.7	80	0.7
Macalister, 1 ^d	1800	12	1.04	66	3.4	68	8 ^f
Macalister, 2 ^d	1751	19	1.17	70	3.5	69	7
Macalister, 3 ^d	1831	-	1.07	78	4.5	76	5
Macalister, 4 ^d	1757	7	1.16	82	3.7	87	5
Macalister, 5 ^d	1797	10	1.16	74	2.8	71	5
Meandarra, 1	1644	11	1.18	84	4.1	94	^g
Meandarra, 2	1866	8	1.05	80	3.4	76	2
Meandarra, 3	1861	7	1.00	62	4.9	62	^g
Norwin, 1	1861	10	1.20	87	6.0	72	1
Norwin, 2	1874	9	0.96	86	6.9	70	6
Norwin, 3	1888	6	0.98	85	7.4	84	2
Tara, 1	1907	21	0.93	60	1.0	61	6
Ulimaroa, 1	1864	7	1.08	91	9.6	84	5
Ulimaroa, 2	1920	5.5	0.98	68	4.5	64	0.7
Ulimaroa, 3	1842	7	1.05	67	4.6	66	6
Ulimaroa, 4	1874	20	1.06	85	7.7	79	5
Warra, 1	1854	17	1.04	71	3.0	69	1
Warra, 3	1772	34	1.08	62	0.5	65	3.5
Warra, 5	1900	-	0.99	71	5.6	69	4
Warra, 6	1764	25	1.10	66	3.1	68	4 ^f
Warra, 8	1900	24	1.03	38	1.2	35	5 ^f
Yandilla, 1	1786	9	1.04	84	6.1	89	2

a = The bulk density of the grain stored varied widely. All bins were full, filling ratio > 0.96.

b = Estimated from two readings only taken between 1 and 4 days and 4 and 17 days after purging.

c = Efficiency, E₁, as defined by Banks (1979).

d = Purge gas composition variable from 50-100% CO₂ in air.

e = Retreated with CO₂ after 7 months as a precautionary measure.

f = Insects present on outloading (see text).

g = Still in storage.

TABLE 6.

Details of CO₂ usage and tonnage of wheat treated with pure CO₂ (1979) or 85% CO₂ (1980) at two sites by Grain Elevators Board, Victoria.

Site, bin no. (date)	Pressure test ^a for 750-375 Pa (min)	Initial ^b % CO ₂	Decay ^b rate (% day ⁻¹)	Tonnage treated	Total CO ₂ used (t)	CO ₂ used (per 1000 t wheat)
Yarrowonga, (1979)	1	69	4.6	11,800	19.73	1.04
	3	89	4.3			
	5	102	6.7			
	7	89	4.7			
	9	97	5.6			
	11	85	4.5			
Rennie, (1979)	1	86	7.9	7,200		
	3	75	5.4			
	4	75	5.6			
	5	85	6.8			
Yarrowonga, (1980)	1	-	-	1,750	14.7	1.13
	3	53	1.5	1,850		
	5	48	0.3	1,790		
	7	65	3.1	1,820		
	9	65	3.2	2,080		
	11	67	3.4	1,870		
	13	59	2.7	1,840		
Rennie, (1980)	1	61	2.6	1,500	7.5	1.06
	3	62	3.7	1,600		
	4	64	3.3	2,000		
	5	56	2.2	2,000		

^a Interpolated semilogarithmically from observations of the decay rate between 750 and 250 Pa.

^b Estimated from readings for between 5 and 12 days after purging.

shielded inlet in the wall of the bin, as described by Banks and Annis (1977), was used. The Queensland bins were already equipped with a perforated distribution duct running radially from the floor centre to the bin wall and this was used for gas introduction. In most cases, pure CO_2 was introduced. Exceptions are given below. The atmosphere within the bin was recirculated as described by Wilson et al. (1980) after CO_2 introduction.

Tables 5 and 6 give details of the commercial treatments of bulk wheat with CO_2 carried out for insect control since the beginning of 1979. The State Wheat Board carried out a further 13 treatments, in addition to the 27 summarised in Table 5. The details of these treatments are incomplete, but those available are consistent with the data in Table 5. The treatments given in Table 5 used an average of 1.06 t CO_2 per 1000 t grain at an average purging efficiency of 74%.

In most of the treatments detailed in Tables 5 and 6, the target CO_2 regime (Table 1) was exceeded. In a few instances the decay rate exceeded $6.9\% \text{ day}^{-1}$ but in these cases, e.g. Ulimaroa, 1 (Table 5), the initial CO_2 level was higher than the minimum acceptable (70%) and the decay rate was such as still to leave $> 35\% \text{ CO}_2$ after 10 days. In some bins an inadequate initial CO_2 level was achieved. On some occasions this could be a result of adding an inadequate quantity of CO_2 , e.g. Meandarra, 3 (Table 5), but appears, on others, to be associated with unusually low loss rates, e.g. Tara, 1 (Table 5). The latter effect also is apparent in the 1980 results shown in Table 6. It should be noted that there is a significant correlation ($P < 0.01$) between calculated initial CO_2 level and loss rate, suggesting that the very low and high initial CO_2 levels calculated for some trials may not represent the true values achieved but result from sampling and measurement errors which inevitably occur under the practical constraints of normal commercial practice.

Though the low values for the initial CO_2 concentrations given in Table 6 may in part be due to inaccuracies in the extrapolation, they may also result from the use of 85% CO_2 not 100% CO_2 as a purge gas in the 1980 treatments. If purging is continued, as in these cases, until at least 80% CO_2 is issuing from the top vent, a considerable quantity of CO_2 is lost from the bin, resulting in a reduced purging efficiency and thus lower initial CO_2 levels for an equivalent quantity of 100% CO_2 . Which of these two effects caused the low initial CO_2 values cannot be distinguished on the basis of the available data for the treatments.

The results given in Tables 5 and 6 show that the target regime for CO_2 can be met under routine commercial conditions in full, welded steel bins using about 1.05 tonnes CO_2 per 1000 tonnes wheat, meeting the current standard for sealing (pressure decay of 1500–750 Pa in > 5 mins in a full bin, see Banks and

Annis, 1980). Further work appears necessary to assess the advantages and disadvantages of purging bins with air-CO₂ mixtures (e.g. 85% CO₂) rather than with pure CO₂.

Application of CO₂-based modified atmospheres in concrete cells

Concrete cells are routinely constructed by the State Wheat Board, Queensland to a gastightness standard exceeding that currently considered suitable for modified atmosphere use (see Banks and Annis, 1980). Five of these cells were treated with CO₂, in the same manner as the welded steel bins discussed above. After the purge was complete the bins were sealed as usual. A substantial negative pressure differential relative to the external atmosphere rapidly formed in the bin, which had to be relieved to prevent structural damage to the bin. In one case a -1500 Pa differential developed over 25 mins. The process continued over several days and is attributed to sorption of CO₂ on the concrete, i.e. carbonation as discussed by Hamada (1968). The details of the treatments in concrete cells are summarised in Table 7. It is notable that, despite a high level of sealing as assessed by the pressure test and a similar usage of gas and efficiency of purging to that obtained in welded steel cells (see Table 5 and 6), the decay rates are much higher and, indeed, are too high to allow the target CO₂ regime to be met unless additional gas is introduced.

Concrete grain storage cells are the only common type of large bulk storage in Australia that has not been treated successfully with 'one-shot' CO₂ atmospheres. The use of CO₂ in concrete cells has been discontinued commercially until the possible effects of the gas on the structure have been fully assessed. It is also necessary to determine if part or all of the sorption process is reversible as this will influence the gas usage in cells that have been treated with CO₂ more than once.

It is notable that CO₂ has been used with some success in the U.S.A. in concrete cells on both maize and peanuts (Jay and Pearman, 1973; Jay et al., 1970). However, further CO₂ was required to maintain adequate CO₂ levels after the initial purge and the rate of addition required was high.

INSECTICIDAL EFFICACY OF CO₂ TREATMENTS AND THE DEGREE OF PROTECTION AFFORDED BY A PARTIALLY SEALED STRUCTURE

In the mode of use proposed for CO₂ in Australia, the gas is added rapidly to the sealed enclosure and held there for a period exceeding 10 days. No further gas is added. The laboratory data on the action of air-CO₂ mixtures is incomplete and limited by its very nature to results from treatment of small numbers of insects that must be presumed to be of restricted genetic variability. Though laboratory observations in general support the target regime used, it requires

TABLE 7.

Details of treatment of wheat with CO₂ stored in 3400 m³ capacity concrete cells by State Wheat Board, Queensland, during 1979.

Site, bin no.	Tonnage treated	Pressure decay test, 1500-750 Pa (mins)	CO ₂ used (t per 1000 t wheat)	Initial ^a % CO ₂	Decay ^a rate (% day ⁻¹)	Efficiency ^b of purging (E ₁ , %)
Oakey, 2	2880	6.5	1.05	101	17.3	78
Oakey, 2	2880	6.5	0.93	74	10.0	64
Brookstead, 2	2880	9	1.00	75	19.5	62
Millmerran, 2	3032	20	1.00	98	13.1	68
Meandarra, 1	2787	22	1.15	55	8.9	42
Meandarra, 2	2689	22	1.30	107	31.1	79

^a = Estimates of initial concentrations of CO₂ and the semilogarithmic decay rate made from two readings only taken from 1 to 2 days and 3 and 17 days after purging.

^b = Efficiency, E₁, as defined in Banks (1979).

verification in practice where the numbers and variability of the insects to be controlled are much greater and where the insects are free to move in the grain bulk.

Assessment of the effectiveness of CO₂ treatment under field conditions is a complex problem. In general, adult stored product pest insects are more susceptible than most immature stages to air-CO₂ mixtures. If the latter survive, they may escape detection if inspection is carried out soon after treatment. If adult insects are discovered some time later, it is not possible to distinguish between a control failure in CO₂ use and an infestation derived from insects entering the storage after the end of the treatment. The two problems, the effectiveness of the current target regime and the degree of insect proofing afforded by an enclosure sealed to the current standard for use with modified atmospheres, must thus be considered together.

In the cases discussed below, the grain treated was in a condition (25-30°C, 10-12% m.c.) favourable for rapid multiplication of Rhyzopertha dominica and Tribolium castaneum, the prevalent pest species. Thus, if after a period of some months subsequent to CO₂ treatment insects are still not detectable. It is reasonable to assume that the treatment used gave a very high or complete kill of insects present. Furthermore, since in an unsealed structure, unprotected grain soon becomes infested, absence of infestation after treatment demonstrates that the sealed structure gives significant protection against reinfestation.

The period of storage of the grain in steel bins treated with CO₂ by the State Wheat Board is given in Table 5. The period was determined by operational requirements and not by insect infestation. In most cases, the grain was known to be infested prior to treatment and was stored for more than 3 months after treatment. Except as noted below, no live insects were found when the grain was sampled at a rate of about 0.1 kg t⁻¹ from the grain stream on outloading. In Victoria, during the 1979 season, with the exceptions given below, no live insects were detected on outloading using a similar sampling technique, although the grain had been held for 7 months after CO₂-treatment (Table 6) and infestation was present before treatment. (Note the grain treated in 1980 as detailed in Table 6 has not yet been outloaded and no inspections have been carried out). These observations show that, in general, the CO₂ treatment regime used was highly effective and a substantial period of storage (several months) was achieved with grain being free of insects at outloading as judged by Australian commercial standards. Furthermore, reinfestation from external sources was not significant.

Observations of insect infestation subsequent to CO₂ treatment

Instances of apparent control failures with CO₂ must be considered against this evidence of successful use. There are 6 cases involving 11 lots of grain, summarised below, where infestation has been detected after CO₂ treatment. In those cases where the target regime was met, infestation was detected only after some months of subsequent storage and thus survival of the treatment cannot be distinguished from reinfestation as its source.

Case 1. Bell, 3, Macalister, 1 and Warra, 6. (Kaimkillenbun, 1 (not given in Table 5, data incomplete) apparently similar). Grain harvested at the end of 1978 and treated then with fenitrothion and bioresmethrin (12 and 1 ppm respectively). Dosed with CO₂ as Table 5 one year later. Target CO₂ regime met or almost met (see Table 4). Very few R. dominica detected at outloading when about one-fifth of each bin had been discharged, but not before or subsequently, suggesting that the infestation was at the surface and very localised. Source of infestation: not known. The localised nature of the infestation suggests that it came from external sources shortly before outloading and was not a survival of treatment.

Case 2. Harden. 1977-78 storage season, treated as described in Table 4. Heavy infestation of T. castaneum and R. dominica prior to treatment. Apparently insect-free (probe sampled) 6 weeks after beginning of treatment, but R. dominica with some T. castaneum found after 13 weeks. Suggested source infestation: insect survival from treatment which did not attain the target initial CO₂ level.

Case 3. Harden. 1978-79 storage season, treated as described in Table 4. No insects found by probe sampling prior to treatment. Apparently adequate CO₂ regime. Store unsealed and small quantities of grain removed from time to time from 2 months after CO₂ dosing. Localised but rapidly developing infestation of T. castaneum found 6 months after treatment. Very few R. dominica and C. ferrugineus also present. Suggested source of infestation: insects introduced into store on machinery or personnel or insects flying in after breaking the seal. A very low level of survival from treatment cannot be excluded as source.

Case 4. Rennie, 3 and 4 in 1978-79 storage season. Treated with CO₂ as per Table 6 after 3 months storage. Condensation damage present in bin 3 prior to treatment. After 7 months from treatment, severe condensation on grain surface with many T. castaneum present. Source of infestation: not known. Recirculation duct may have been blocked resulting in low CO₂ levels in upper part of bin.

Case 5. Warra, 8. Grain harvested and treated as Case 6. CO₂ treatment as Table 5. CO₂ level apparently lower after 4 days after treatment (41%) than after 10 days (43%) suggesting poor mixing or recirculation. Many R. dominica present at outloading 5 months after treatment. Suggested source of infestation: insect survival of treatment because of inadequate CO₂ levels in parts of bin.

Case 6. Yarrawonga, 9 and 13. 1978-79 season. 1870 t wheat in each bin treated 2 months after loading with about 1 t CO₂ per 1000 t wheat. CO₂ regime in bin 13 apparently adequate, but in bin 9 inadequate because of leakages. Bin 9 retreated 1 month later after sealing leak, as Table 6, with an adequate CO₂ regime. Inspected by turning the grain through the elevator and sampling after 10 weeks storage after treatment. No insects found. At outloading 7 months after treatment, light crusting present on grain surface and a few *T. castaneum* found. Suggested source of infestation: insects introduced during inspection or inadequate CO₂ levels in upper parts of the bin resulting from blocked recirculation ducts.

In view of the uncertainties inherent in this kind of assessment, cases such as these cannot be taken as firm evidence that the target regime is inadequate, but should be considered in the light of further commercial experience. This should give evidence of whether such apparent control failures are to be expected or whether they result only from incorrect application of the system.

CONCLUSION

The experimental and commercial trials and treatments reviewed above are part of the general development of various modified atmosphere techniques of grain storage and disinfection for Australian conditions. Techniques are now available for the modification and treatment of most large types of storage with some form of modified atmospheres, although CO₂-treatment of concrete cells is still not commercially feasible and treatment of a large horizontal shed with nitrogen has not been attempted. In the future, it seems profitable to research on-site generation of various modified atmospheres, giving methods of avoiding the expense of transporting liquified gases to the storage site. A further assessment is required of the period of storage after an inert gas treatment that can be expected before insects reach commercially detectable levels. The technique will also present managerial problems that will need to be resolved. These include the need for a system of transportation of the insecticide-free grain from the storage to its destination which does not allow the grain to become sensibly infested en route, development of suitable remote sampling procedures to detect infestation if it occurs and management of the modified atmosphere treated stocks to ensure that any infestation is promptly eradicated before damage results.

The modified atmosphere systems described above are now at a stage in Australia where they can be regarded as one of the strategies available commercially against insect infestation. Their use will depend on the economics of the systems, which are already competitive with current practices in many situations, and the requirement, if it arises, for treatments that do not leave chemical residues on the grain.

ACKNOWLEDGEMENTS

Financial assistance from the Australian Wheat Board for several of the trials reported here is gratefully acknowledged. The gas supply companies, Carba Australia and The Commonwealth Industrial Gases Pty. Ltd., have contributed substantially to the commercial development of the system.

REFERENCES

- Bailey, S.W., 1965. Air-tight storage of grain; its effect on insect pests - IV *Rhyzopertha dominica* (F.) and some other Coleoptera that infest stored grain. *J. stored Prod. Res.*, 1: 25-33.
- Banks, H.J., 1979. Recent advances in the use of modified atmospheres for stored product pest control. *Proc. 2nd Internat. Working Conf. Stored-Prod. Entomol.* Ibadan, Nigeria (1978), pp. 198-217.
- Banks, H.J. and Annis, P.C., 1977. Suggested procedures for controlled atmosphere storage of dry grain. CSIRO Aust Div. Entomol. Tech. Pap. No. 13, 23 pp.
- Banks, H.J. and Annis, P.C., 1980. Conversion of existing grain storage structures for modified atmosphere use. In: International symposium on controlled atmosphere storage of grains. Elsevier, Amsterdam (in press).
- Banks, H.J., Annis, P.C. and Wiseman, J.R., 1979. Permanent sealing of 16,000-tonne capacity shed for fumigation or modified atmosphere storage of grain. CSIRO Aust. Div. Entomol. Rep. No. 12. 16 pp. + plates.
- Barrer, P.M. and Jay, E.G., 1980. Laboratory observations on the ability of *Ephestia cautella* (Walker) (Lepidoptera, Phycitidae) to locate, and to oviposit in response to a source of grain odour. *J. stored Prod. Res.*, 16: 1-7.
- Hamada, M., 1968. Neutralisation (carbonation) of concrete and corrosion of reinforcing steel. *Proc. 5th Internat. Symp. on Chem. of Cement*, Tokyo, Japan (1968), pp. 343-384.
- Jay, E.G. and Pearman, G.C., Jr., 1973. Carbon dioxide for control of an insect infestation in stored corn (Maize). *J. stored Prod. Res.* 9: 25-29.
- Jay, E.G., Redlinger, L.M. and Laudani, H., 1970. The application and distribution of carbon dioxide in a peanut (groundnut) silo for insect control. *J. stored Prod. Res.* 6: 247-254.
- Shejbal, J. 1979. La tecnologia Snamprogetti per la conservazione di cereali in azoto. *Tech. Molitoria*, 30: 512-520.
- Wilson, A.D., Banks, H.J., Annis, P.C. and Guiffre, V., 1980. Pilot commercial treatment of bulk wheat with CO₂ for insect control: the need for gas recirculation. *Aust. J. exp. Agr. Anim. Husb.*, (in press).
- Winterbottom, D.C., 1922. Weevil in wheat and storage of grain in bags: a record of Australian experience during the war period (1915 to 1919). Government Printer, Adelaide, 122 pp.