# PURGE AND MAINTENANCE RATES OF CARBON DIOXIDE FOR WHEAT TREATMENT IN A REASONABLY SEALED SILO

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#### ABSTRACT

Two tests were conducted on wheat in a 100-tonne capacity silo using (1) carbon dioxide (CO<sub>2</sub>) from "Minitanks" and (2) burner-gas produced by propane combustion. To establish the efficacy of each treatment, gases were monitored using a HP 5880 gas chromatograph in a mobile laboratory stationed alongside the silo. Gas sampling lines of 2-mm bore special nylon tubing were placed at different depths in the grain, and were extended to and connected with the gas chromatograph. A clock-table programme was used to monitor gasses at specific intervals. After the initial purge from the bottom, it was noticed that the atmosphere in the silo could be maintained more efficiently by the addition of modified atmosphere (MA) to the head-space at the top of the silo. The economics of both gas supply systems was compared.

#### INTRODUCTION

Carbon dioxide (CO<sub>2</sub>), and effluent gas with a composition of about 13% CO<sub>2</sub>, less than 1% oxygen (O<sub>2</sub>) and the rest nitrogen (N<sub>2</sub>) from an efficient propane burning generator are considered environmentally acceptable for the treatment of food commodities, grain in particular. Over the past 20 years, several researchers in the US and Australia have achieved effective control using various methods of generating and applying modified atmospheres (MAs) (Banks 1979; Storey, 1980).

Work in the UK on cereals has progressed mainly along two fronts, firstly the use of CO<sub>2</sub> from cylinders or "minitanks" and secondly the development of a propane burner according to specifications provided by the Central Science Laboratory (CSL), Slough. The composition and behaviour of the gas produced were established using a Hewlett Packard 5880 gas chromatograph (GC) during a series of experimental trials in grain bins. In the first trial with the burner, it proved possible to bring down the

 $O_2$  level to 1% in a 200-tonne farm bin with 5 atmosphere changes (Bell *et al.*, 1991).

The trial described here was conducted to compare the performance of CO<sub>2</sub> and burner gas.

# METHODS AND MATERIALS The bins

The bin chosen was one of 30 similar bins in a food manufacturer's silo complex. Each bin was 13-metres in height and 4.5 metres in diameter, and was relatively gastight, being of welded steel construction. Although capable of holding 100 tonnes of grain, the experimental bin contained only 80 tonnes of wheat, leaving a headspace of  $28 \text{ m}^3$ . Considering that each tonne of grain occupies  $1.4 \text{ m}^3$ , the total volume of air to be replaced from the bin in one air change was approximately  $84 \text{ m}^3$  ( $28 \text{ m}^3 + 56 \text{ m}^3$ ). Entrance to the bin was possible through a sealable manhole on the bin-cap.

Positioning of gas-sampling lines and thermocouples

The grain surface was approximately 2.5 m from the bin top. Standing on the surface, 2 mm-bore nylon gas-sampling lines were inserted into the grain up to a 5-m depth at two different positions, one at the centre and the other 0.5 m away from the side wall under the hatch. These were spaced at 1-m intervals up to the surface, with an extra one at a 0.5-m depth at the centre. A sampling line was also placed in the headspace 1 m from the bin ceiling. Thermocouple wires and test insects (*Cryptolestes ferrugineus*, samples of 100 adults in nylon gauze cages) were placed alongside gas sampling positions. The lines and thermocouple wires were led out of the bin via a 10-cm diameter port at the bin top, dropped to the ground floor and extended outside the building to the mobile laboratory. Gas sampling lines were connected to ports of a GC, and the thermocouple wires to a chart recorder.

Bin sealing and gas-tightness assessment.

The small port for sampling lines, access hatch, and all other openings in the bin were sealed meticulously using plasticine, polythene sheeting, and masking tape. A valve was fitted to the bottom of the bin hopper which in turn, was connected to a compressed-air hose. A 3mm nylon tube from a small port on the bin-side of the valve was led to a digital manometer to assess gastightness. Pressure tests were performed initially on the empty bin and then on the bin containing 80 tonnes of grain. In each case, the bin was pressurised repeatedly to 1,000 Pascals and the average times recorded for the pressure to drop to 500 Pascals were 4 min and 2.1 min for empty and loaded bins, respectively. The difference was attributed to the surface-to-

volume ratio, the sources of leaks remaining the same but the volume of air being reduced by loading the bin.

**Preparation for dosing operations** 

A security cage was erected in the loading the silo complex to house the propane burner with fuel cylinders and the minitanks containing liquid C0<sub>2</sub>. A reinforced flexible hose having a 5 cm dia. was led outside the cage to the building and connected to a specially-welded spout just above the grain-retaining plate, the other end being connected to the minitank or burner. Another 9-mm nylon dosing line was placed in the headspace through the apical port and extended to the cage to either permit the dosing of gas to the headspace, or vent gases while dosing from the bottom.

Trial 1: Use of liquid carbon dioxide

The outlet from a minitank containing about 150 kg liquid CO<sub>2</sub> was connected to a 3-kW vaporiser that purged the bin at a rate of 17m<sup>3</sup> CO<sub>2</sub> /hr. When most of the interstitial atmosphere had been replaced and the CO<sub>2</sub> level was building steadily in the headspace, the vaporiser was disconnected from the system and the flow rate was reduced progressively until the headspace concentration of CO<sub>2</sub> started to drop below 60% (Fig. 1). After two weeks, the hatch was opened and the CO<sub>2</sub> in the bin was expelled using compressed air from the bottom. Some insects were replaced at this stage and the bin was flushed with air until the CO<sub>2</sub> concentration fell to atmospheric level so that the second purge could be started as soon as possible.

Initially the second purge proceeded by dosing from the base as before, until  $CO_2$  increased to 30% in the headspace and over 90% throughout the grain. Then dosing was switched from the base to the headspace and after the level of  $CO_2$  had increased to 60% in the headspace (Table 1), the flow rates were adjusted to find the minimum required to maintain this level (Fig. 2). The test was continued for two weeks and the bin was cleared of  $CO_2$  before removing all of the *C. ferrugineus* test insects.

Trial 2: Use of exhaust gas from a propane burner

The gas generator used was built by the Aerogen Company Ltd., Alton, Hants, UK. The exhaust gas produced by burning propane in closed combustion chamber was cooled to several degrees below ambient temperature before its introduction into the base of the bin (Table 2). The initial purge rate was 9 m<sup>3</sup>/hr and the flow rates were adjusted to reduce O<sub>2</sub> levels to 1% until the maintenance rates were established and then continued as in the previous trials (Figs. 3, 4, and 5).

Table 1: Rates of gas loss or gain during the trial maintaining a high CO<sub>2</sub> concentration by adding gas to the bin base or headspace.

Flow rate and location	Hours from last flow adjustment	Mean % CO <sub>2</sub> in grain	% loss or gain of atmosphere per day in grain	Mean % CO2 in headspace	% loss or gain of atmosphere per day in headspace
15 l/min	1	97.1	rade is cit man	72.2	Annyllin al
to base	5	96.8	-1.8	64.0	-51.5
	9	96.1	-4.3	58.1	-44.0
	13	95.6	-3.1	53.9	-36.3
	Mean % loss	per day:	3.1		44.0
25 1/min	0	72.9	200 no <u>1</u> 10 Jan	27.8	or all
to base	12	74.2	of an of the	30.5	
	24	76.7	5.2	33.7	21.2
	36	76.9	3.6	35.9	17.7
	48	76.9	0.2	39.1	16.0
	60	76.7	-0.2	41.0	14.2
	72	76.0	-1.2	43.2	10.5
	84	76.7	0.0	44.7	9.0
	96	76.6	0.8	47.3	9.5
	108	75.7	-1.3	48.9	9.4
	Mean % gain	per day:	0.9		13.4
10 l/min	17	83.7		52.4	1,
to head	41	66.2	-20.9	58.0	10.7
space	65	63.6	-3.3	59.1	1.9
7	89	62.9	-1.1	58.3	-1.4
	113	63.5	1.0	59.5	2.1
	137	63.6	0.2	61.8	3.9
	161	66.1	3.9	64.1	3.7
		(gas chromato	graph recalibrate	ed)	
	193	68.2	Faring engin leads	67.8	a sadin'ila ni
	217	68.9	1.0	67.4	-0.6
	241	68.0	-1.3	65.7	-2.5
	Mean % loss	per day:	2.6	Mean % gain per day:	2.2

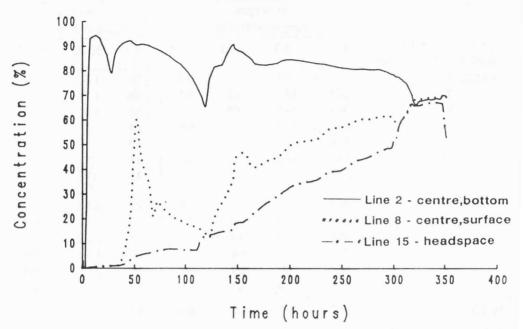


Fig.1: Carbon dioxide levels when applying CO2 to the hopper bottom.

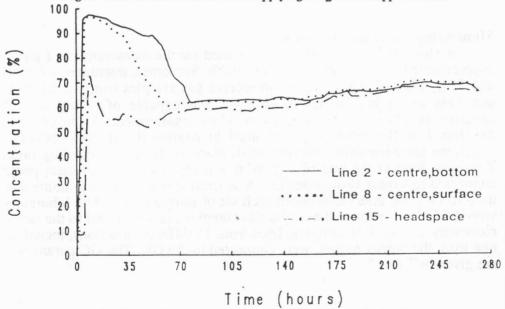


Fig. 2: Carbon dioxide levels when applying CO<sub>2</sub> to the headspace.

Table 2: Atmosphere replacement within the bin using burner gas at two different flow rates.

		% oxygen, purging at 9m³/hr			% oxygen, purging at 4.8m <sup>3</sup> /hr		
	Hours 0-3		8-11	12-15	14-17	30-33	33-39
Centre, 5	20.3	5.4	0.9	0.7	5.9	1.8	1.2
4	20.4	14.2	1.2	0.3	5.6	1.7	1.2
3	20.3	18.5	1.7	0.4	5.5	1.6	1.1
2	20.4	19.2	2.2	0.5	4.8	1.6	1.1
1	20.6	19.1	-	-	4.3	1.5	1.1
0.5	20.3	19.2	2.3	0.6	4.6	1.7	1.1
0	20.4	18.1	1.3	0.5	4.5	1.7	1.3
Side, 5	20.4	1.7	0.4	0.6	2.3	1.1	0.9
4	20.4	1.4	0.4	0.5	2.2	1.1	0.9
3	20.3	3 2.2	0.4	0.4	2.6	1.2	0.9
2	20.4	2.7	0.4	0.4	2.6	1.1	0.9
1	20.4	4.0	0.4	0.4	2.7	1.2	0.9
0	20.5	7.9	0.9	0.5	4.8	1.9	1.2
Headspace	20.7	18.5	10.1	0.9	4.2	1.8	1.2
Exhaust from bu	rner 0.2	0.3	0.3	0.3	0.8	0.9	0.7

Monitoring gas concentrations

A Hewlett Packard 5880 GC was used for the measurement of gases, supplemented occasionally by two portable Servomex instruments for O<sub>2</sub> and CO<sub>2</sub>. The GC was equipped to receive gas samples from 32 different positions and a built-in microprocessor was capable of taking sets of samples at chosen intervals, even when unattended. A programme developed in the laboratory was used to control the interval between injections allowing sufficient time to displace air from the sampling lines. This was done at the rate of 100 ml/min using a small diaphragm pump connected to a mass flow controller. A separate clock-table programme was used to vary the interval between each set of samples. A mobile laboratory with the GC was stationed near the silo complex and connected to the mains electricity supply. Gas sampling lines from 15 different positions, including one from the burner output, were connected to the GC. The GC parameters are given in Table 3.

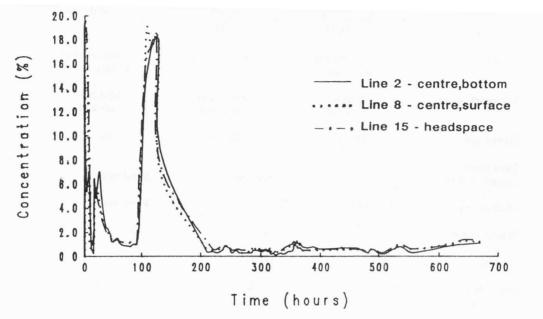


Fig. 3: Oxygen levels during the burner gas bin treatment.

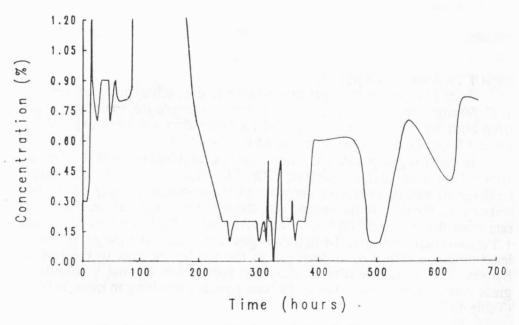


Fig. 4: Oxygen output of the burner during the bin treatment.

Table 3: The HP 5880 GC parameters for CO<sub>2</sub> and O<sub>2</sub> analysis.

	CO <sub>2</sub> only	O <sub>2</sub> only	O <sub>2</sub> and CO <sub>2</sub>	
Detector	TCD	TCD	TCD	
Column	Glass-lined ss 1m x 3 mm	Glass-lined ss 3m x 3 mm	Glass-lined ss 0.6m x 6 mm	
Stationary phase	Porapak Q 80-100 #	Spherocarb 80-100 #	Mol. sieve 60-80 #	
Carrier gas	Helium	Helium	Helium	
Flow rates column A & B	20 ml/min	20 ml/min.	30 ml/min.	
Modulator flow	45 ml/min	45 ml/min.	45 ml/min.	
Detector temp.	150°C	150°C	150°C	
Inj. temp.	150°C	150°C	150°C	
Oven temp. Temp. prog:	150°C	150°C	50°C	
Initial time Prog. rate Final value			5 min 20°C/min 150°C	
Ret. time	1.8 min	0.7 min	4.55 min. & 20 min.	

## **RESULTS AND DISCUSSION**

Both  $CO_2$  and burner gas proved effective in achieving complete kill of *C. ferrugineus*, the former within 2 weeks of exposure, maintaining gas from both the bottom and the top, and the latter after a 3-week exposure at about 1%  $O_2$ . Grain temperature ranged from 4 - 10°C.

In the first purge with CO<sub>2</sub>, more gas was required (over 200 m<sup>3</sup> or 2.5 atmosphere changes) to achieve 60% CO<sub>2</sub> than in the second purge. Leakage of gas or excessive sorption by the commodity may have been responsible for this. In the second run, the bin was purged at the maximum rate from the start until 60% CO<sub>2</sub> was achieved in the headspace after only 1.5 atmosphere changes. To maintain gas levels after the purge, it was at least twice as effective to dose gas to the headspace than to the hopper bottom. The higher density of CO<sub>2</sub> and application against a column of grain would increase pressure at the base possibly resulting in more leakage (Table 1).

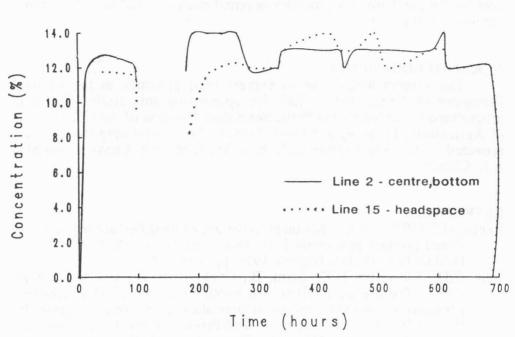


Fig. 5: Carbon dioxide levels during burner gas bin treatment.

The trials with burner gas confirmed the initial observations in earlier trials. In order to maintain the required atmosphere in the bin after the initial purge, it seemed to be three times more efficient to apply gas to the top than to the hopper-bottom. Other factors remaining unaltered, leakage of gas from the bottom or ingress of air in the headspace may be possible explanations.

The trials showed that both systems could be used effectively for the treatment of grain in reasonably-sealed silos. The total amount of CO<sub>2</sub> from minitanks (150-160 kg CO<sub>2</sub>) or exhaust gas from the burner necessary to purge and maintain the required atmosphere in this silo would be about 550 m<sup>3</sup>. This could be provided by seven minitanks of liquid CO<sub>2</sub> or by burning 50 kg of propane which would cost approximately £800 and £25, respectively, including monthly hire charges for the cylinders. In addition, electrical costs of about 11p/hr are required for the burner. However, there is a limitation regarding the burner in that below a minimum output of the exhaust of 2.5 m<sup>3</sup>/hr, a flame failure may occur. At this minimum output after the purge, over the 4 weeks of the trial, about 200 kg of propane would

be required to run this particular burner, at a cost of about £100. This means a new burner design capable of maintaining lower exhaust flows or the use of the same burner with larger silos would be more economical. The capital cost for the purchase of a generator or rental charges could be offset against the low running cost.

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### REFERENCES

Banks, H.J. (1979) Recent advances in the use of modified atmospheres for stored product pest control. In: Proc. 2nd Int. Work. Conf. Stored-

Product Ent., Ibadan, Nigeria, 1978. pp. 198-217.

Bell, C.H., Llewellin, B.E., Sami, B., Chakrabarti, B., and Mills, K.A. (1991) The use of a self-cooled exothermic controlled atmosphere generator to provide a means of controlling insect pests in grain. In: Proc. 5th Int. Work. Conf. Stored-Product Protection, Bordeaux, France, Sept. 1990. (Edited by Fleurat-Lessard, F. and Ducom, P.), pp. 1769-1776.

Storey, C.L. (1980) Mortality of various stored product insects in low oxygen atmospheres produced by an exothermic insect atmosphere generator. In: Controlled Atmosphere Storage of Grains, (Edited by Shejbal, J.), pp. 85-92 Elsevier Sci. Publ. Co., Amsterdam, Holland.

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