

## **FLOW RATES OF CONTROLLED ATMOSPHERES REQUIRED FOR MAINTENANCE OF GAS LEVELS IN BOLTED METAL FARM BINS**

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### **ABSTRACT**

Tests were conducted on bolted metal bins of different size using units generating nitrogen-based atmospheres by combustion of propane or separation of oxygen (O<sub>2</sub>) from air by pressurised membrane filtration. All bins tested were loaded with wheat. Having purged a structure with an appropriate, low-O<sub>2</sub> atmosphere, the flow rates of gas required to maintain the atmosphere were assessed under typical weather conditions in the UK. The premise that larger structures of a particular shape and design would require fewer atmosphere changes per day to maintain a lethal atmosphere was investigated. The flow rates required for maintenance were controlled to some extent by wind-speed. Data are presented that relate maintenance flow rate to structure size and the prevailing weather conditions.

### **INTRODUCTION**

In the search for alternatives to using toxic chemicals to protect or treat stored grain, research in the field of controlled atmospheres has gathered momentum over the last two decades. The stability of atmospheres based on either carbon dioxide (CO<sub>2</sub>) (Jay and Pearman, 1973) or replacement of oxygen (O<sub>2</sub>) with nitrogen (N<sub>2</sub>) (Shejbal *et al.*, 1973; Storey, 1973) for treating grain is well established, provided that gas levels can be maintained. This can be accomplished most easily by a continuous flow of gas to the storage structure (Jay and d'Orazio, 1984).

Over the past six years, work in the UK has concentrated on the use of gas produced by propane combustion, and, more recently, the investigation of N<sub>2</sub>-generating systems. In welded steel bins sealed to a pressure test half-life of 7 mins, it was discovered that wheat at over 14% moisture content (m.c.) at 18°C, or over 15% m.c. at 13°C, had the effect of maintaining O<sub>2</sub> levels below the 1% output level of the apparatus used to supply a simulated burner gas atmosphere (Bell, 1987). Thus, after the initial 3-day purge at

0.3 m<sup>3</sup> of gas per hr, no further top-up of gas was required. Microbiological activity was thought to be responsible for this effect. Further work demonstrated that low O<sub>2</sub> levels persist in 16% m.c. grain in these bins with the seal reduced to a pressure test half life of 2 min or less (Bell *et al.*, 1990). With grain at a lower m.c., only one atmosphere replacement every two weeks was needed to maintain O<sub>2</sub> levels near 1%. It was recognised, however, that these standards of sealing were much higher than those obtainable in bolted corrugated metal bins, which account for over 90% of UK farm or cooperative grain silo storage facilities.

Recently, a self-cooled propane combustion unit, developed by Aerogen Co. Ltd. according to specifications defined at the Ministry of Agriculture, Slough, was used to purge a typical farm bin (Bell *et al.*, 1991). It proved possible to bring O<sub>2</sub> levels down to 1% in 180 tonnes of wheat with just over 5 atmosphere changes, purging at a gas-flow rate of 10-11 m<sup>3</sup>/hr, with sealing restricted to sheeting the grain surface inside the bin and blanketing off auger and aeration inlet points with polythene sheeting.

Two further trials were conducted, one with the propane burner to treat 600 tonnes of grain in a larger bin, and the other with a low output membrane filter-based N<sub>2</sub> generator to lower O<sub>2</sub> levels in a 250 tonne bin of grain.

## **MATERIALS AND METHODS**

### **Bins used for the treatments**

For the trial with the burner, the bin chosen was one of a set of six 780-tonne capacity bins of 11 m diameter and 9.9 m height (to eaves). It was loaded with approximately 600 tonnes of wheat that formed a shallow cone at the top to fill the bin to within 2 m of the eaves at the sides. To receive a 5 cm-diameter flexible, reinforced dosing hose from the burner outlet, a plate incorporating an appropriate fitment was engineered to replace the cover for the aeration ducting at the base of the bin. The bin used in the trial with the N<sub>2</sub> generator was one of an adjoining set of eight 250-tonne capacity bins at the same site as the bin described above. These bins were of a 6.4 m diameter and 10.6 m height and were mounted each on a concrete base that accommodated an auger tube and housed the aeration ducting. The auger was sealed by inserting a metal sheet in the pipe connecting it to the main conveying system, and by packing and wrapping with polythene sheeting. A plate was fashioned to fit over the 20-cm diameter opening of the basal aeration duct to receive the 9-mm diameter nylon dosing line. This was pushed well into the duct so that gas would be introduced close to the silo centre. An additional dosing line was run to the bin headspace to permit introduction of gas to the grain surface at a later date.

To obtain a record of wind conditions throughout each trial period, an anemometer was installed on the gantry near the apex of the test bin and connected to a data logger in a mobile laboratory stationed below that housed

the analytical equipment needed for the trial.

### **Preparation of test insects**

Grain stored in the UK is often cooled to temperatures below 10°C. Under such conditions only adult stages of stored-product beetles are likely to be present, these often tending to aggregate in the warmer parts of a store from where breeding may start. Thus cooled bulks may contain wandering adults with immature stages occurring only where there has been some local warming. The test insect chosen for the burner gas trial was *Cryptolestes ferrugineus*, the rust-red flat grain beetle, because this was known to be tolerant of low-O<sub>2</sub> atmospheres in the adult stage.

To obtain experimental samples, cultures of *C. ferrugineus* were set up at monthly or fortnightly intervals and reared at 30°C for greater productivity on a 2:1 mixture of rolled oats and wheat flour with an added 5% portion of dried yeast powder. Adults emerging in these cultures were separated and placed on food prior to transport to the site, and were acclimatised at 20°C for at least 5 days in preparation for low temperature exposure. A laboratory control sample was held at 20°C throughout each trial, and a site control sample was inserted to a 1-m depth in an adjacent bin filled with wheat.

### **Positioning of gas sampling lines, thermocouples, and test insects**

Working from inside the bin, nylon sampling lines of 2 mm bore were inserted into the grain using metre lengths of threaded rod, the first one being fitted with a slip attachment holder at one end. After reaching the required depth, the column of rods were drawn up leaving the sample line in position. Lines were inserted at the centre and at one side of the bin at metre intervals from a minimum of 5 m depth to the surface. Extra lines were placed at a 0.5-m depth at the centre and side positions and also near the bin sides at the other three compass points. All lines were fed out of the bin via the access hatch in the roof and tied in a bundle down the side of the bin to reach the mobile laboratory stationed below.

Test insects placed in nylon gauze sachets were inserted into the grain in threaded cage holders. Rod spacers were used to bring cage holders to lie as close as possible beside the sampling lines, i.e., at 1 m intervals from a 5-m depth to the surface. Copper constantan thermocouples were also inserted by attachment to the rods, at a 5-m and 3-m depth and near the surface at both centre and side positions. These, too, were fed out of the bin via the hatch.

After insertion of samples and equipment, the bin surface was sheeted with a lightweight laminate material having low permeability to O<sub>2</sub>.

### **Use of the propane-fuelled exhaust gas generator - Trial 1**

The generator incorporated an open flame burner unit in a closed combustion chamber with a self-regulating fuel and air supply. Fan assistance

was provided in the outlet line to counteract back pressure and the product gas was cooled to several degrees below ambient temperature by an ammonia solution refrigeration circuit powered by the combustion chamber and linked to a water glycol heat exchanger. Firing was accomplished by an ignition and safety cut-out cycle which provided protection against the risk of explosion caused by faulty gas supply or mounting back pressure caused by freezing of the heat exchanger.

The burner was fired on several occasions before direct application of gas to the bin to check output rates and O<sub>2</sub> levels. After these preparations were complete, the initial purge of the bin with burner gas was started by introducing gas to the base at a rate of 12.5 m<sup>3</sup>/hr. Gas concentrations were monitored to find the number of atmosphere replacements required to bring O<sub>2</sub> levels to 1%, and the output was adjusted to establish the maintenance flow rate required to hold the low O<sub>2</sub> atmosphere under the prevailing weather conditions.

### **Use of the nitrogen generating system - Trial 2**

The N<sub>2</sub> generator comprised a small air separation unit and a 6.35 kW air compressor unit supplied by Calor Gas Ltd. The air separator, based on "Permea" hollow fibre gas selective membranes, was capable of producing an atmosphere containing less than 1% O<sub>2</sub> at a flow rate of 3 m<sup>3</sup>/hr and an operating temperature of 50°C. At 4.8 m<sup>3</sup>/hr, the maximum flow rate obtainable from the compressor and the rate chosen to start the purge, the O<sub>2</sub> level increased to approximately 1.5%. After obtaining an equilibrium in O<sub>2</sub> levels within the bin at this flow level, the flow was progressively reduced to approximately half, dosing to the bin base or headspace, and was then increased to 6 m<sup>3</sup> per hour for the last week of the trial when a larger compressor was available.

### **Collation and assessment of results**

Most gas concentration measurements were taken with a Hewlett Packard microprocessor-controlled 5880 gas chromatograph installed in the mobile laboratory, using a thermal conductivity detector. Supplementary readings were taken with three instruments manufactured by "Servomex": a digital paramagnetic O<sub>2</sub> analyser 574, and, for the trial with burner gas, a PA404SVA carbon dioxide infra red analyser and a 1400 series infra red carbon monoxide analyser. Frequent readings were taken during the purging of the bin and the gas chromatograph was then programmed to take a complete set of readings from each position within the bin every four hours. To measure CO<sub>2</sub>, the gc column was packed with Porapak Q which at an oven temperature of 150°C gave a very short gas retention time (<1 min), making it possible to reduce the interval between injections to about 6 min. To measure both CO<sub>2</sub> and O<sub>2</sub> this interval was extended to 22 min because of the need to lower the oven temperature to 50°C and the column packing to

"Spherocarb" for O<sub>2</sub> to be separated from N<sub>2</sub>. Measuring O<sub>2</sub> alone reduced the interval to about 12 min. For those occasions when both gases were monitored, the interval between sets of samples was extended to 8 hr.

Windspeed and temperature were logged automatically throughout the trial. At the end of each trial generators were shut down and fresh air was blown through the grain before unsheeting and removing equipment from the grain the following day. Test insects were returned to the laboratory for assessment of survival after 2 months' incubation at 25°C.

Data on O<sub>2</sub> levels within the bin were collated, graphed, and related to gas flow rates, windspeed, and the quantity of propane or electric power used.

## RESULTS

### Trial 1

In the trial on the 600-tonne bulk of grain using the propane burner, a gas input rate of 11.0 - 12.5 m<sup>3</sup>/hr (fuel input to burner of 8-9 l/min) took 4 days (2.8 atmosphere changes) to bring O<sub>2</sub> levels down to less than 2% at all points (Figs. 1 and 2). Two days later, sustained winds of greater than 5 m/sec caused O<sub>2</sub> levels to rise above 3% at several positions after a lag of about a day. O<sub>2</sub> levels fell rapidly to below 2% within 12 hr of the wind dropping. During the second week, windspeeds again increased but on this occasion were less constant, although reaching a mean of 8.4 m/sec on the 9th day of the purge. In addition to being more gusty, the wind direction shifted from south to southwest. On this occasion O<sub>2</sub> levels in the bin did not rise above 2% (Figs. 1 and 2). Reducing the gas flow to 9.5 m<sup>3</sup>/hr (7 l/min fuel to burner) revealed an increased sensitivity to windspeed, but under calm conditions at the end of the trial, O<sub>2</sub> levels in the bin held down well. Throughout the purging of the bin, the burner produced an O<sub>2</sub> level of 0.4-0.9% in the exhaust gas, with less than 150 ppm carbon monoxide and 12.5-13% CO<sub>2</sub>.

The results for the test insects are presented in Table 1, together with grain temperatures. All adult *C. ferrugineus* died when held below 1.5% O<sub>2</sub> for a total of 8 days at temperatures below 8°C. As can be seen from Figs. 1 and 2 at each point O<sub>2</sub> levels rose to over 2% in response to wind and other factors at various times during the experimental treatment, but after the initial purge rarely reached 3%. At each point, the periods at less than 1.5% O<sub>2</sub> all occurred during the last ten days of the purge. However, at most points in the upper 3 m of grain, survivals were high enough to give rise to a large population increase, approaching that observed in the control. At these positions, a shorter time was experienced at below 1.5% O<sub>2</sub>, and at the bin centre temperatures were slightly higher and could have increased survival (Table 1).

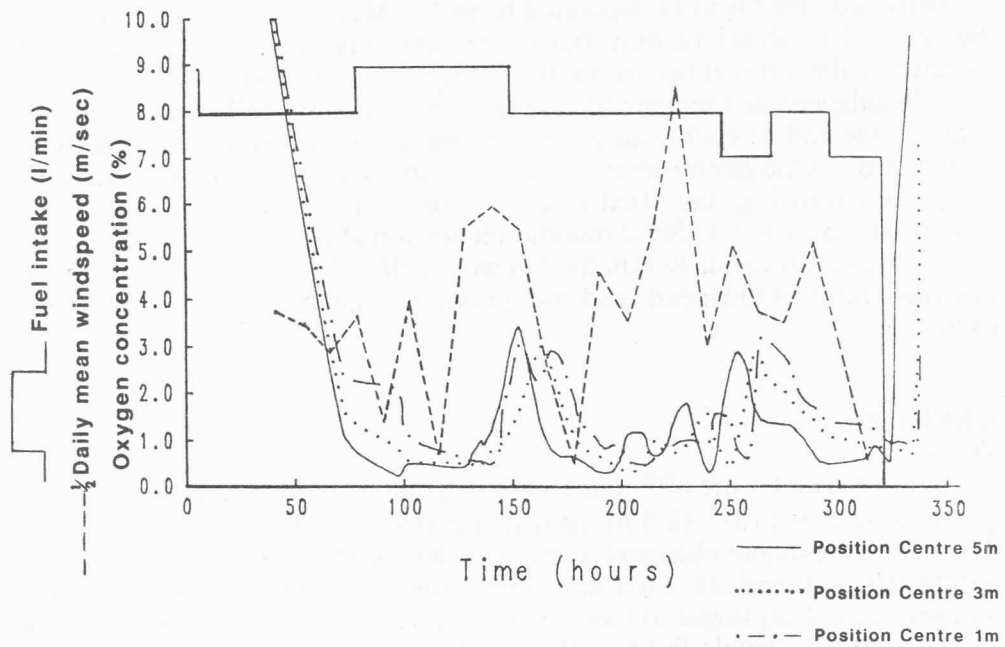


Fig. 1: Oxygen levels in the centre position during the burner gas bin treatment.

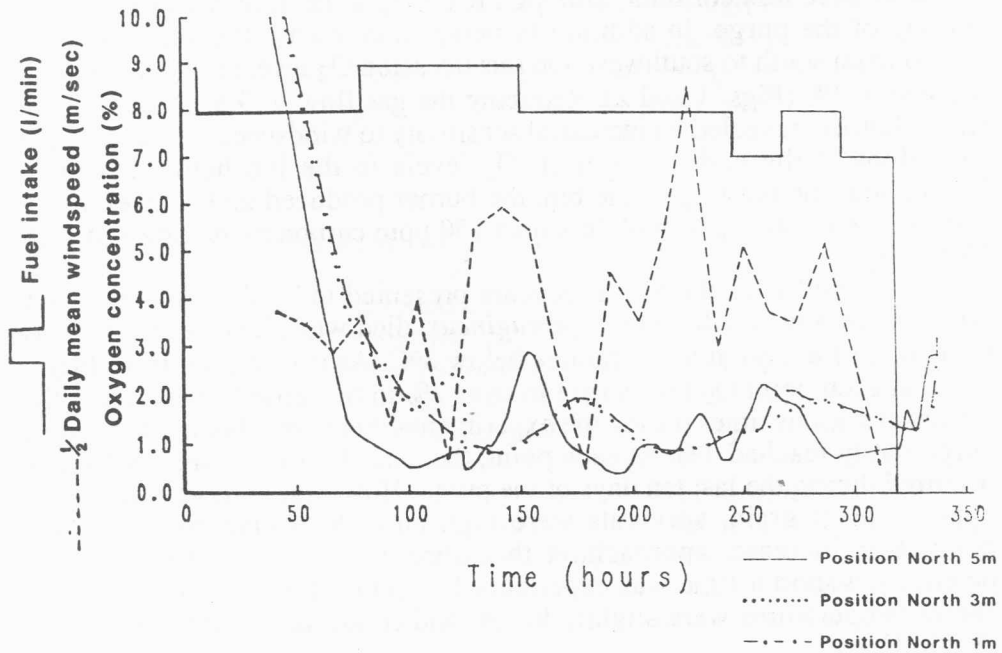


Fig. 2: Oxygen levels in the north position during the burner gas bin treatment.

Table 1: The survival of adult *C. ferrugineus* at various positions within a bin of cooled wheat held under low O<sub>2</sub> by application of burner gas.

Depth (m)	Bin Centre			North Side		
	Days at <1.5% O <sub>2</sub>	Grain temp (°C)	Effect on insects	Days at <1.5% O <sub>2</sub>	Grain temp. (°C)	Effect on insects
5	8	8.4	None alive	8	7.4	None alive
4	8		1 survivor	8		None alive
3	8	12.8	Large F1	6		5 survivors
2	7		Large F1	6		Large F1
1	7		Large F1	6		Large F1
Surface	6	9.9	Large F1	5	6.6	Large F1
Control	0	12.8	Large F1			

Over the 14 days of the experimental purge, the burner consumed approximately 285 kg propane (just over six 47 kg cylinders at a cost of approximately £20 each). The cost of holding the 600 tonnes of grain at 1-2% O<sub>2</sub> for a 4-week period would thus be approximately 43p per tonne, plus a modest electrical cost for fans and air supply.

## Trial 2

The initial purge of the 250-tonne bin using the N<sub>2</sub> generator, applying gas at a rate of 4.8 m<sup>3</sup>/hr reached equilibrium after 70 hr, after approximately 2 atmosphere changes. Half-daily means for windspeed ranged from 0.2-15.8 m/sec with weekly means of 2.4-4.3 m/sec for the first weeks of the trial, and under these conditions the mean O<sub>2</sub> level in the bin was 6.1% with an input gas O<sub>2</sub> content of approximately 1.6% (Table 2). Increase in windspeed (Fig. 3) or decreases in temperatures (Fig. 4) resulted generally in an increase in O<sub>2</sub> levels.

Reductions in flow rate resulted in an increase in O<sub>2</sub> levels but flows were not maintained long enough for reliable end point O<sub>2</sub> levels to be attained. However it did appear that dosing to the headspace was less efficient in holding down mean O<sub>2</sub> levels than dosing from the base. Increasing the flow rate to the bin base to 6 m<sup>3</sup>/hr caused a reduction in mean O<sub>2</sub> levels in the bin to 4.8%, even though the O<sub>2</sub> content of the product gas rose to 2.3% (Table 2). Furthermore, O<sub>2</sub> levels were still falling when the trial was disbanded. A simple linear extrapolation of the data indicated that the flow rate that would have been required to hold the bin below 1% O<sub>2</sub>, assuming a machine O<sub>2</sub> output of 0.5%, was approximately 7 m<sup>3</sup>/hr.

## DISCUSSION

In both experiments, the importance of windspeed was demonstrated in

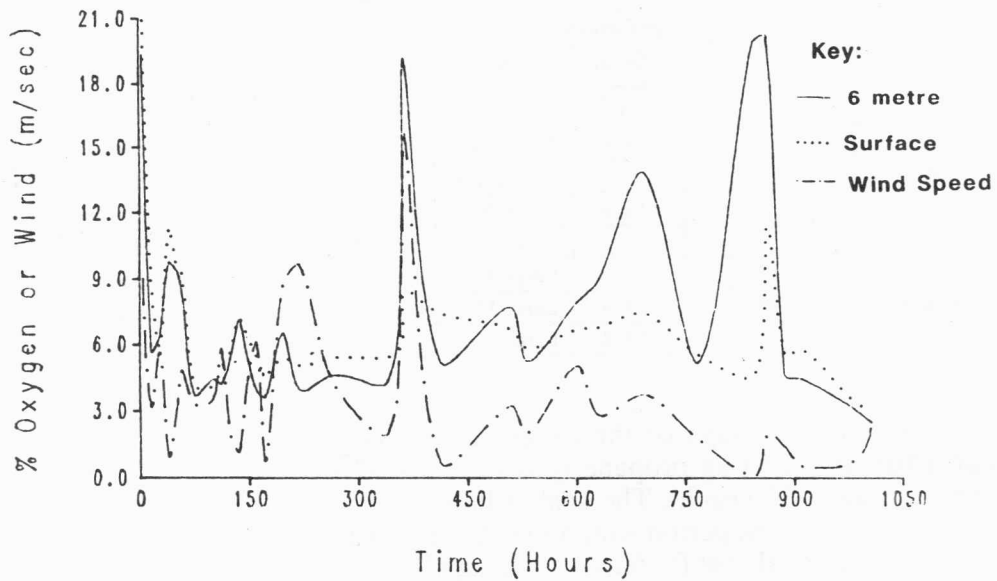


Fig. 3: Effect of the wind on the % oxygen levels in the centre of the silo.

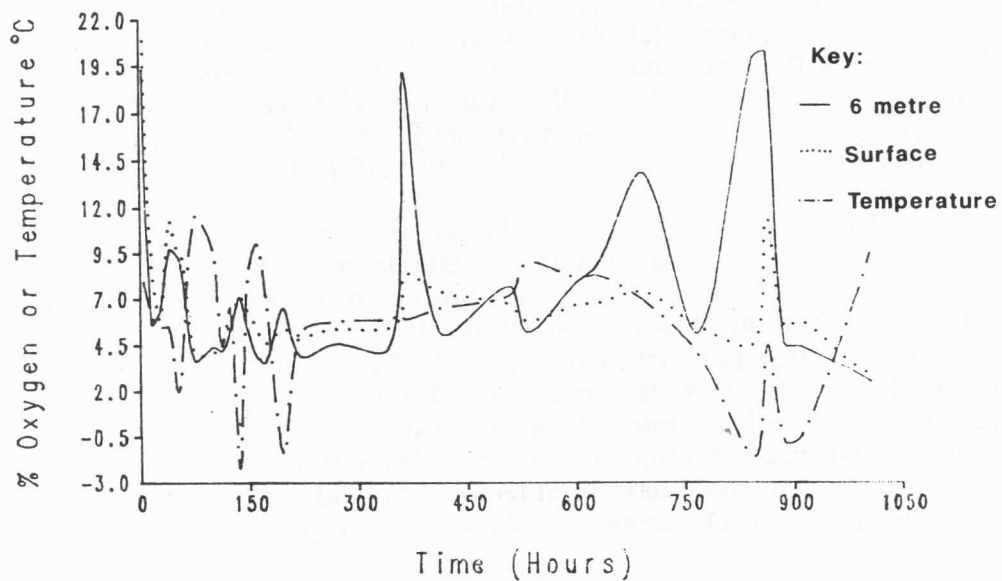


Fig. 4: Effect of the ambient temperature on the % oxygen levels in the centre of the silo.



Table 2: Application of a N<sub>2</sub>-rich gas to 250 tonnes of wheat in a silo.

Flow rate from N <sub>2</sub> generator (l/min)	Duration of input (days)	Total gas used		O <sub>2</sub> contents (%)		Adjusted O <sub>2</sub> level in bin (B-A)(%)
		Vol. N <sub>2</sub> (m <sup>3</sup> )	Bin atmos. per day	Input gas to bin (A) (mean)	Mean level in bin (B) (19 lines)	
80	17	1,958	0.71	1.6	6.1	4.5
50	5	360	0.44	1.5	7.4	5.9
35	10	504	0.31	1.5	7.7	6.2
65*	4	374	0.58	1.4	12.7	11.3
100	7	1,008	0.89	2.3	4.8	2.5

\* Gas applied to grain surface.

determining the flow rate necessary to maintain a low O<sub>2</sub> atmosphere. With the 600-tonne bin, a sustained period at 5 to 6 m/sec caused a greater rise in O<sub>2</sub> levels than a period of gusty weather with a resultant higher 12 hr mean of 8 m/sec (Figs. 1 and 2). A slight change in wind direction may have contributed to this effect. Under the weather conditions prevailing during the trial, a maintenance flow rate of 8 l/min fuel (11 m<sup>3</sup>/hr product gas) appeared adequate to hold the low O<sub>2</sub> atmosphere. Under calm conditions, a flow rate of 7 l/min fuel (9.5 m<sup>3</sup>/hr product gas) may have sufficed, but O<sub>2</sub> levels rose with windspeeds of 3 to 4 m/sec (Figs. 1 and 2).

In a previously described trial on bolted metal bins (Bell *et al.*, 1991), a 180-tonne bulk of grain in a smaller but similarly shaped bin required a maintenance flow rate of approximately 5 m<sup>3</sup>/hr of gas in the absence of wind, increasing to 8 m<sup>3</sup>/hour under windy conditions. Theoretically, a doubling of bin size would result in a 1.59 fold increase in flow rate to maintain an atmosphere because of the likely relationship between leakage rates and the surface area to volume ratio. A trebling of size as between the two structures considered here would result in an approximately two-fold increase in maintenance flow rate, which is in fact the difference observed under calm conditions. Of considerable interest is the possibility that the importance of windspeed may diminish as structure size is increased, because a relatively small increase in flow to the 600-tonne bin appeared able to compensate for the effect of quite windy weather.

It was demonstrated once again that the efficiency of purge is poor when conducted at flow rates only slightly higher than the maintenance rate. It required a total of 96 hr at a mean flow rate of just over 11 m<sup>3</sup>/hr (approximately 2.8 atmosphere changes) to bring the interstitial atmosphere of the 600-tonne wheat bulk to within 1% of the burner O<sub>2</sub> output and a further day (total of 3.5 atmosphere changes) for all points to come to 1% or less O<sub>2</sub>. The test insects used, selected for their likely presence in cooled

grain, demonstrated that the low-O<sub>2</sub> atmosphere was effective in achieving control, even though there were periods when the prevailing wind increased O<sub>2</sub> levels.

Laboratory experiments currently in progress also indicate that considerable fluctuation in the O<sub>2</sub> level can occur without disrupting the efficacy of the exposure, provided that the upper level is not too high and that the periods at such levels are of short duration in relation to the overall exposure. Following the recommendation of Howe (1974), the measure of success in the present trial was taken as the appearance of the F1 generation of the exposed adults. An F1 appeared in only one sample at a depth of 3-m or more in the grain, indicating the likelihood of obtaining complete control by prolonging the exposure. Normally a 4-week exposure would be recommended for treatment of grain at temperatures around 10°C.

Data have now been obtained for three different bins of corrugated metal sheet construction containing different quantities of grain. Maintenance rates of approximately 5, 7, and 9.5 m<sup>3</sup>/hr have been estimated for 180, 250, and 600 tonnes of grain, respectively, under calm conditions. For the first two bins, these flow rates represent approximately one atmosphere change per day, but for the larger bin only 0.6 atmosphere changes per day were required. Clearly, more data are required to establish whether maintenance flow rates may be expected to decrease in proportion to bin size as predicted by the surface area to volume ratio, but the results obtained so far give grounds for optimism. For the propane burner running costs for a four-week treatment of bins upward of 1,000-tonnes capacity could work out as less than 50 p/tonne. Costs for the N<sub>2</sub> generating system would be controlled by the power consumption of the air compressor.

#### ACKNOWLEDGEMENTS

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