

Application of pressure-swing absorption (PSA) and liquid nitrogen as methods for providing controlled atmospheres in grain terminals

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Abstract

The use of controlled atmospheres as a method for providing pest-free grain is becoming more popular with increasing market demand for grain free of residues. Two systems for providing low oxygen atmospheres were trialed at GrainCorp export terminals in New South Wales, Australia. The pressure-swing absorption system (PSA), at the Port Kembla terminal, and a liquid nitrogen-based supply at the Newcastle terminal. The test bins at both terminals were sealed to a level that gave greater than 5 minutes for a pressure half-life (full bin). They were originally equipped for fumigation with methyl bromide under recirculation.

The PSA unit, with a gas output of 1.47 m³/minute at 0.7% oxygen, purged two 13660 m³ bins in series down to an oxygen concentration of <1%. The first bin in the series was purged down to 1.5% oxygen in 7 days. The concentration in both bins was maintained for 19 days below 1.5% and typically below 1.0% by gas provided on a pressure demand system. Due to the good sealing of the bins, the maintenance flow rate corresponded to the calculated maintenance rate required to compensate for gas loss due to diurnal temperature changes and resulting pressure cycling.

Vaporised liquid nitrogen was used to purge three 1954 m³ bins to an oxygen concentration of <1% throughout. The efficiency of purge was increased by the use of a higher flow rate. A flow rate of 6.02 m³/minute purged a bin down to <1% oxygen in 3.5 hours. The concentrations in the bins were maintained below 2% with a flow rate of about 50 L/minute. Individual bins required slightly different maintenance rates and entry position of the maintenance gas at either the top or base of the bin to hold the concentration in them below 1.0%. The maintenance rates were higher than those required to compensate for gas loss due to diurnal temperature changes alone, suggesting other leakage factors such as wind were involved in gas loss.

Both the liquid nitrogen and PSA systems were successfully demonstrated as practical systems for CA generation in the grain terminals, though the PSA system was of insufficient output to provide the required atmosphere rapidly. On the basis of a 4-week exposure at <1% oxygen, the resulting cost, including hire charges of the two systems, was \$A0.34/t for the PSA system and \$A0.65/t for the liquid nitrogen system. Although the material cost of phosphine fumigations is lower (\$A0.20/t) the operational advantages of nitrogen in the terminals make it the preferred option. Liquid nitrogen is now being used routinely at the Newcastle terminal to provide controlled atmosphere disinfection for 29000 t of storage capacity.

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Introduction

Nitrogen, carbon dioxide or a mixture thereof can be added to grain storages in a controlled fashion to displace or dilute the storage atmosphere to give a gaseous composition which is insecticidal. Further gas may be added to maintain this composition. The technique is known as controlled atmosphere (CA) storage.

It is well established (Banks and Annis 1990) that atmospheres containing less than 2% oxygen are capable of disinfesting stored grain. The technology of application of such low oxygen (O₂) CAs, using liquid nitrogen as a gas source, was developed in Australia and Italy in the 1970s. Commercial field trials were successfully carried out (Banks 1979; Tranchino et al. 1980) but these did not lead to the adoption of the technique. The remarkable success of phosphine fumigation (recirculation, surface application and SIROFLO®) displaced the need for nitrogen atmospheres in Australia, with phosphine fumigations generally both cheaper and easier to apply than nitrogen treatments. The sealed technology and standards for grain stores developed for nitrogen-based CA technology proved useful in improving phosphine technology.

With the increasing difficulties and regulations faced by fumigants, for example the anticipated phasing out of methyl bromide due to it being an ozone-depleting material, it is appropriate that CA systems, including nitrogen-based atmospheres, be re-evaluated. Nitrogen-based CA has several operational advantages over fumigants that may make its use preferable in certain circumstances.

These include:

- no use of acutely toxic materials
- little or no airing required before grain can be moved
- reduced hazard area in comparison with fumigants
- compatible with 'organic' and truly residue-free grain
- no sorption problems
- no environmental restrictions or obligatory monitoring
- capable of being produced from air on-site.

The trials detailed here were carried out collaboratively between GrainCorp Operations Limited and the Stored Grain Research Laboratory at two grain export terminals in New South Wales, Australia. These trials aimed to demonstrate the application of nitrogen atmospheres, to gather local costing and performance data, and specifically:

- to confirm the quantity of nitrogen required to create and maintain storage atmospheres at <1% O₂ throughout the planned 28-day exposure; and
- to determine what modifications would be required to rig a silo block for routine commercial use with nitrogen-based CA.

Two methods of nitrogen generation were assessed: the pressure-swing absorption system (PSA) at the Port Kembla

terminal and the liquid nitrogen-based supply at the Newcastle terminal. The bins treated at both these terminals were sealed to specification for methyl bromide fumigation.

The general design of the process, flow rates and subsequent calculations adopted for the trials follow those of Banks and Annis (1977).

Materials and Methods

Pressure-swing absorption system trial — Port Kembla

The trial was conducted on two 10000 t capacity white-painted steel bins, Bins A1 and A2, at the Port Kembla terminal. The bins contained Australian Standard White wheat from the 1990–91 harvest season. The capacity of the bins, contents and pressure decay times are given in Table 1. These bins were equipped for methyl bromide fumigation by recirculation, fitted with a pressure relief valve and required no additional sealing.

Table 1. Dimensions, tonnages and pressure tests for PSA system trial, Bins A1 and A2, Port Kembla terminal.

Parameters	Bin A1	Bin A2
Bin volume (m ³)	13660	13660
Bin height (m)	35.9	35.9
Rated wheat capacity (t)	11576	11576
Wheat tonnage (t)	9392	9958
Actual wheat volume (m ³)	11083	11750
Headspace volume (m ³)	2577	1910
Total free air in bin (est. m ³)	6789	6375
Full bin pressure decay time, 2–1.5 kPa (minutes)	80	30
Grain moisture content (% w.b.)	9	9

The PSA unit, hired from The Commonwealth Industrial Gases Pty Ltd, consisted of an air compressor and dryer, an air receiver tank, two absorber beds, and a nitrogen receiver tank. In the absorber beds, under pressure, nitrogen is separated from the oxygen by a carbon molecular sieve. The rated output from this unit was 1.33 m³/minute at 0.8% O₂ concentration, but slightly increased performance was obtained in practice (1.47 m³/minute at 0.7% O₂).

The two bins were purged in series. The PSA outlet was connected to the annular diffuser, originally installed to distribute methyl bromide, in the base of Bin A2 using 2" spiral wound PVC hose. The outlet at the top of Bin A2 was connected to the top of Bin A1, with Bin A1 venting from the diffuser at the base of the bin. The flow from the outlet of the PSA unit was measured using Rotameters and a Datametrics Model 800-LM hot-wire flow meter. The calculated purge time for Bin A2, following Banks (1984), was 9.8 days, assuming free mixing in the headspace and plug flow in the bulk, and taking into account the 86% filling ratio of the bin. Bin A1 was expected to require a longer purge time as some dispersion of the purging front would probably occur during passage through Bin A2.

After the initial purge period, the outlet from Bin A1 was closed and the PSA system was fitted with a regulating system to add gas on demand to maintain a fixed minimum pressure in the bins. The outlet regulator of the unit was set at 1.9 kPa, allowing gas into the bins when the gas pressure dropped below this value. The pressure levels in the bins were measured by a micromanometer (P.P.F.A.—EC060) connected to an outlet in the base of Bin A1. The oxygen concentrations in the bins were monitored for a further 8 days after shutdown of the

PSA unit to determine the natural leakage rate of air into the bins.

Liquid nitrogen based trial — Newcastle

The trials were conducted in Bins W1, X1 and X3, situated on the south-eastern corner of the Newcastle terminal. The bins contained wheat from the 1991–92 harvest season. Capacities, tonnages and pressure tests for each bin are given in Table 2. The bins were constructed of reinforced concrete. They were part of a 4 × 8 nested cell block, with each trial bin comprising part of the exterior wall. These bins were designed to be of gastight construction, but they had not been tested or used as sealed bins since commissioning in 1972.

Table 2. Dimensions, tonnages and pressure tests for liquid nitrogen based trial, Bins W1, X1 and X3, Newcastle terminal.

Parameters	Bins W1	Bin X1	Bin X3
Bin volume (m ³)	1954	1954	1954
Bin height (+ conical section, m)	27.7	27.7	27.7
Rated wheat capacity (t)	1655	1655	1655
Wheat tonnage (t)	1506	1506	1526
Actual wheat volume (m ³)	1780	1780	1800
Headspace volume (m ³)	175	175	152
Total free air in bin (est. m ³)	850	850	836
Full bin pressure decay time, 1000–500 Pa (minutes)	20	5	11
Grain moisture content (% w.b.)	10.0	9.2	10.0 (est)

Bins W1 and X1, equipped for recirculation fumigation with methyl bromide, each had an exterior return duct and diffusers for the gas on the inside of the bin cone. These required no modification to accept the nitrogen gas, except for fitting an inlet to the duct work between the closed valve leading to the recirculation fan and the diffusers. Bin X3 had no internal duct work. Nitrogen was introduced directly into the grain via a 2" diameter inlet fitted above the slide on the outloading valve. A small shedder plate was welded to the valve body on the inside to prevent blockage of the inlet by grain. The wall-to-roof and the wall-to-floor joints in Bins W1 and X1 were sealed with acrylic sealer (Duraflex), but Bin X3 was untreated. All bins met or exceeded the specified 5 minute (full) pressure half-life test.

Each bin was purged by introducing nitrogen gas into the base of the bin. Liquid nitrogen was vaporised in an electrically driven forced draft heat exchanger (max. capacity 16 m³/minute gas) before being fed to the bins in 2" PVC pipe-work. Nitrogen flows were regulated by a gate valve downstream of the vaporiser.

Bins W1 and X1 were purged in parallel at a relatively slow purge rate (~3 m³/minute each). The input flows were monitored by a Rotameter (W1) or Datametrics 800-LM hot-wire flow meter (X1). Bin X3 was purged at about 6 m³/minute, with an Annubar pitot tube to measure flows. About 1.3 m³ of N₂/t of grain was expected to be required to purge the 90% full bins (following Banks and Annis 1977). The amount of liquid nitrogen required to purge a 91% full, 1954 m³ bin, at 15°C, was calculated to be 2.3 t.

During the purge, the valve at the top of the bin was left fully open until the headspace oxygen concentration had fallen below 15%. The valve was then partially closed so as to create a small back pressure of about 50 Pa. This prevented influx of air under the windy conditions experienced during the purge and consequently decreased purge times. The purge was dis-

continued and the bin sealed when the headspace concentration fell below 2%. At this point the atmosphere in the grain bulk was < 1% O₂ throughout.

Gas for concentration maintenance was taken directly from the boil off of the storage vessel. This vessel was equipped with a pressure-raising circuit to maintain a set pressure above the stored liquid. Initially, the maintenance gas for Bins X1 and W1 was piped via 15 mm diameter garden hose into the inloading valve at the top of each bin. Maintenance flow was measured by a variable cross-section flow meter (Gapmeter). During the later part of the maintenance on these bins the flow was switched to the base of the bins. Bin X3 maintenance was applied via the base of the bin. The oxygen concentrations in Bins W1 and X1 were monitored for a further 12 days after shutdown of maintenance gas to determine the natural leakage rate of air into the bins. Bin W1 was left sealed, but Bin X1 had the 2" diameter gate valve at the top bin opened.

Gas and temperature monitoring systems

The gas in the bins was sampled through 3 mm O.D. nylon tubing. The sampling points in the bins for the two trials are shown in Table 3. Each line was sampled sequentially through a series of solenoid valves. The lines were purged using a diaphragm pump for a 12-minute period to ensure complete flushing. The oxygen concentration was measured by a Neotronics Otox 90 meter and at the end of the sampling period the results were recorded on a computer and a chart recorder. As a calibration check, lines were run from a nitrogen cylinder (instrument grade) and from ambient air. A flow meter was placed in line to check for any blockages.

Temperatures were measured using type T copper-constantan thermocouples. A thermocouple was laid at the same point as each gas line. The ambient temperature was measured at a point under the eaves of the monitoring shed. The temperatures were recorded on a Data Electronics Datataker DT100 data logger.

Table 3. Gas and temperature sampling point positions in the bins at the Port Kembla and Newcastle terminals.

Port Kembla terminal	Newcastle terminal
Headspace, 2/3 down from bin top	Headspace, 2/3 down from bin top
3 m into grain	1/3 down from bin top
Bottom of bin	1/3 up from bin bottom
Bin A1 only, at bottom ring	Bottom of bin

Results

Pressure-swing absorption system trial

The PSA system purged the bins at a flow rate of 1.47 m³/minute and an oxygen concentration of 0.7%. The first bin in the series, Bin A2, was purged down to 1.5% oxygen in 7 days. The purging efficiency, calculated on basis of plug flow in the grain bulk and free mixing in the headspace (E₃, of Banks 1979), of Bin A2 to a 1.5% O₂ concentration was 64%. Bin A1 had been purged at all sample points to below 1.0%, with a single point in the bin base, furthest from the gas inlet, at <3% after 36 days of purging. The maintenance phase was then started and continued for 19 days. Over the maintenance phase the O₂ concentration of the PSA output gas decreased to 0.5% with a flow rate averaging 352 m³/day. The changing oxygen concentration in the bins is shown in Figure 1. The times taken for each position to reach an oxygen concentration

of 1.5% and the final equilibrium O₂ concentrations at the end of the maintenance phase are given in Table 4.

Table 4. Time taken for the PSA system to reduce each sampling position in Bins A1 and A2 to an oxygen concentration of 1.5% and equilibrium oxygen concentrations obtained at the end of the trial.

Sampling points	Time to 1.5% [O ₂] (days)	Equilibrium [O ₂] (%)
A2 – bottom	9.8	0.5
A2 – 3 m into grain	1.9	0.5
A2 – headspace	7.1	0.5
A1 – headspace	10.4	0.5
A1 – 3 m into grain	11.6	0.5
A1 – bottom	37.5	1.17

Figure 2 depicts the flow of gas into the bins during the maintenance phase and the corresponding changes of pressure within Bin A1. On most days the flow was cut off by the pressure-sensing system at about 9.00 a.m. and recommenced between 3.00 and 4.00 p.m. Over the maintenance phase the running hours were approximately 7.8 hours/day, as determined from monitors on the compressor and measurements of the compressor motor temperatures.

The average temperatures in the bins over the trial period at the various sampling points are shown in Table 5. Temperature swings in the headspace, the calculated ventilation rates and actual compensatory maintenance rates required to cover gas leakage associated with these temperature swings (Banks and Annis 1984) are given in Table 6.

The weighted average of the various sampling points in the bin was taken to give a bin average O₂ decay rate. The decay rates, calculated as described by Banks (1984), and the time it would take each position to increase from 1% to 5% oxygen concentration, are given in Table 7.

Liquid nitrogen trial

The flow rates and the resulting purge data and efficiency are given in Table 8. At the end of purge the concentration of oxygen in the headspace of Bins W1 and X3 was 1.1% and of Bin X1, 2.7%. The change in oxygen concentrations in Bin X3 during the purge is shown in Figure 3. The other bins gave similar purging profiles. The resulting O₂ concentrations during the maintenance phase and the changes in flow rates and entry position for the maintenance gas into the bins are shown in Figure 4.

The average temperatures over the trial period are given in Table 9. The headspace temperature swings and the calculated gas loss and maintenance rate to compensate for this in Bin W1 are given in Table 6.

The bin average oxygen decay rates and the time for the O₂ concentration to increase from 1 to 5% are given in Table 7.

Table 5. Average temperatures in the Port Kembla terminal Bins A1 and A2 over trial period.

Sampling points	Temperatures (°C)	
	Bin A1	Bin A2
Headspace	15.6	15.5
3 m into the grain	23.3	20.0
Bottom	15.9	16.2
Ring	16.5	–

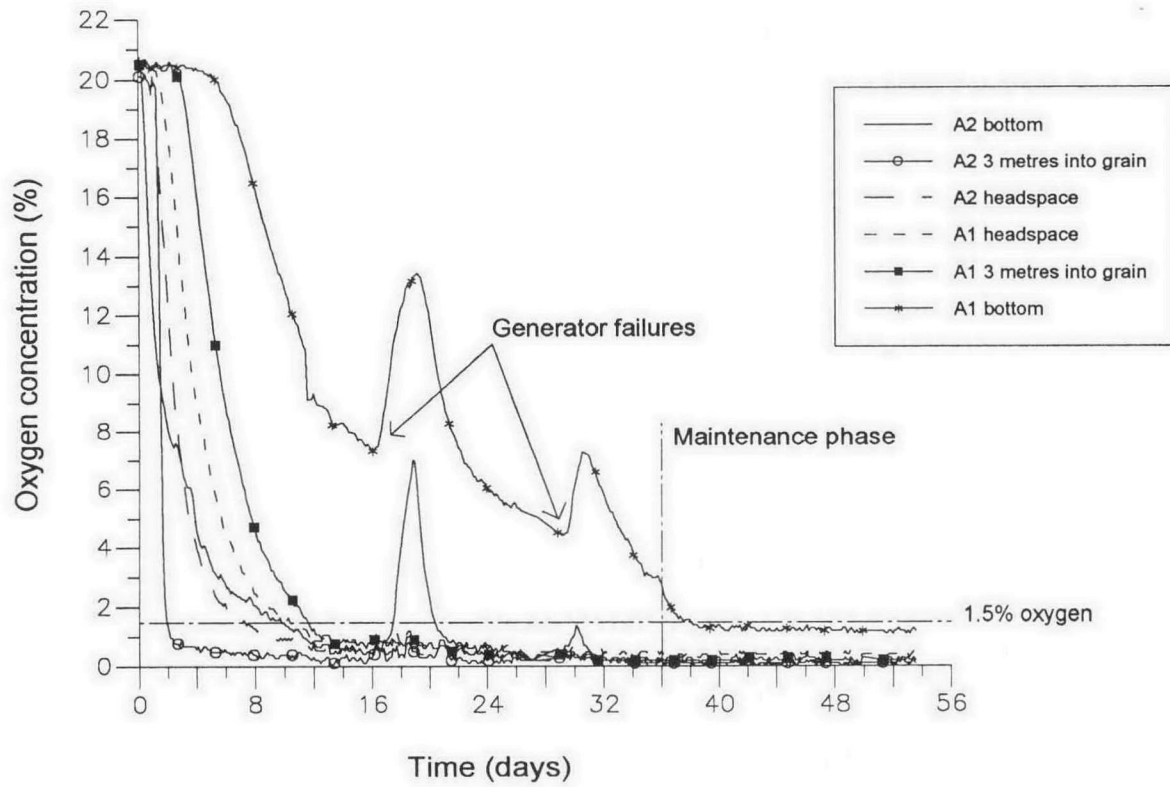


Fig. 1. Oxygen concentrations in Bins A1 and A2, Port Kembla terminal, during purge and maintenance.

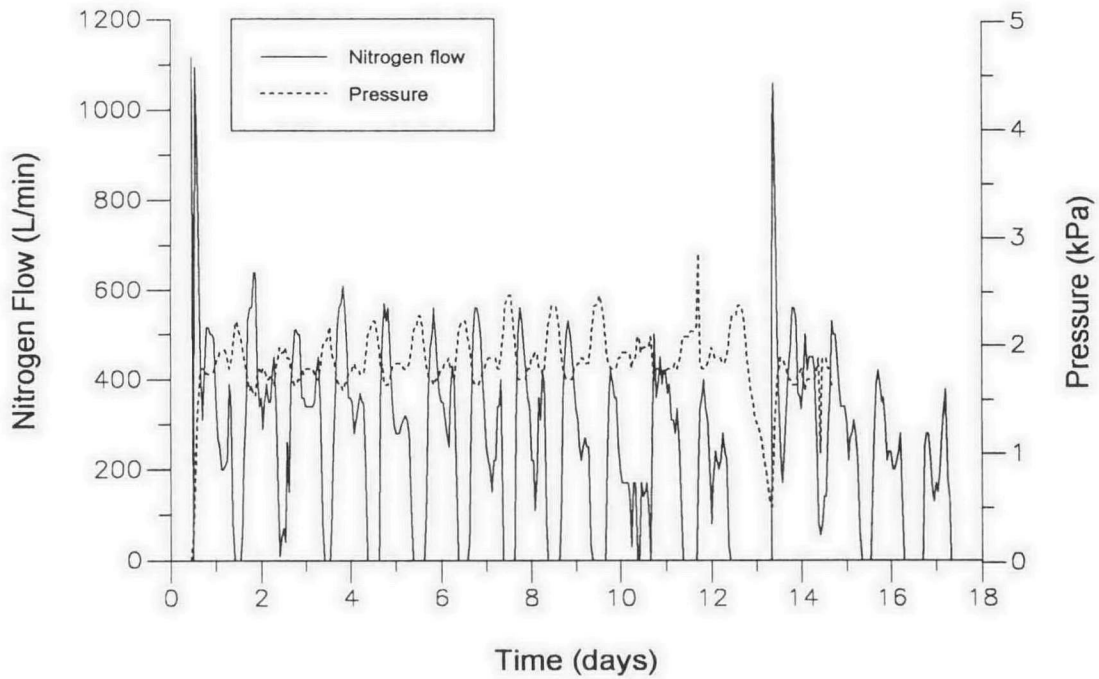


Fig. 2. Pressure in Bin A1 and flow rate from PSA system during the maintenance phase, Port Kembla terminal.

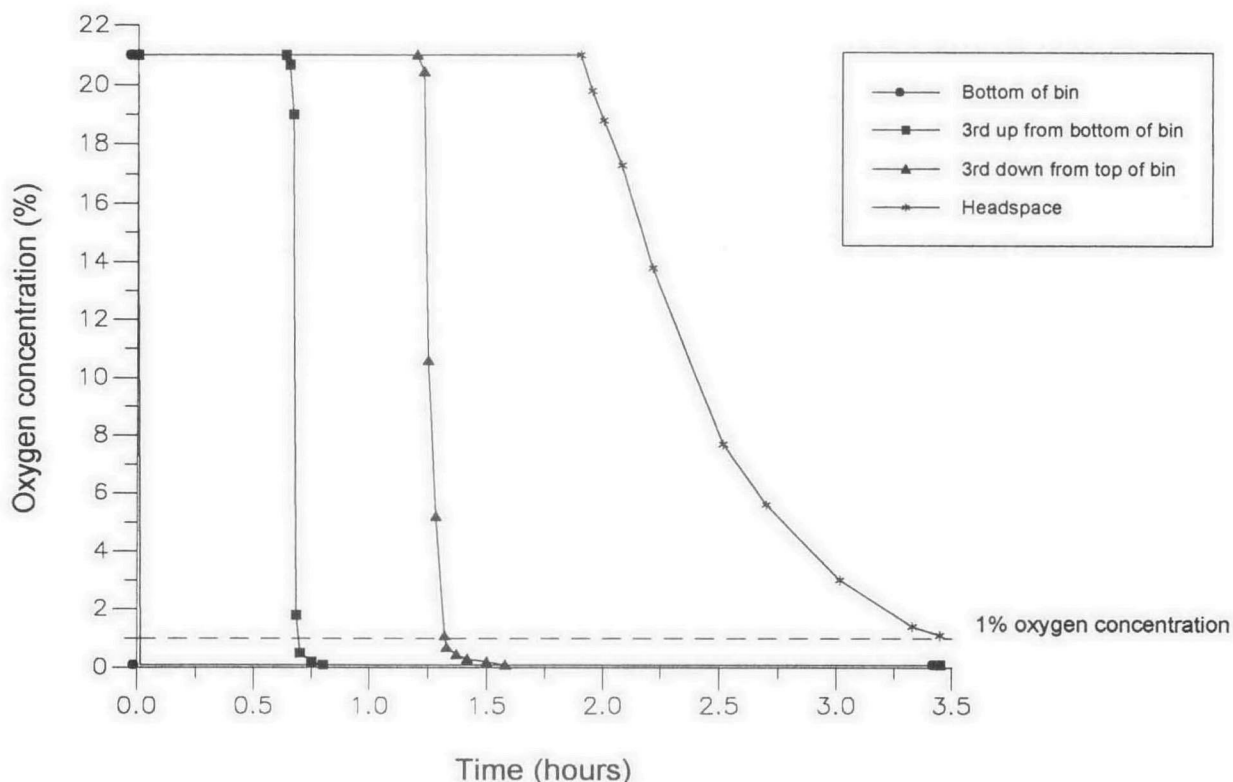


Fig. 3. Oxygen concentrations during purge of Bin X3, Newcastle terminal.

Table 6. Temperature factors involved in gas loss, gas loss rates expected, and observed and calculated maintenance rates.

Factors	Port Kembla Bin A1	Newcastle Bin X1
Average temperature decrease in headspace (°C)	11.3	8.3
Maximum temperature decrease in headspace (°C)	21.5	12.7
Equivalent ventilation rate (%/day)	1.5	0.59
Maximum ventilation rate (%/day)	2.9	0.90
Gas loss from temperature swings (av. m ³ /day)	102	5.0
Gas loss from temperature swings (max. m ³ /day)	195	7.7
Calculated maintenance rate (m ³ /day) ^a	306–585	15–23
Observed maintenance rate (m ³ /day)	352	65

^a Maintenance rate expected, calculated as 3x gas loss rate from temperature swings. This is equivalent approximately to that expected assuming free mixing in the headspace of air leaking in and no effects in the grain bulk.

Table 7. The bin average oxygen decay rates in the Port Kembla terminal Bins A1 and A2 and Newcastle terminal Bins W1 and X1, and the time taken for the oxygen concentration in the bins to increase from 1 to 5%.

Sampling position	O ₂ decay rate/day	Days to 5% [O ₂]
A1	0.006	37.2
A2	0.020	11.1
W1 (sealed)	0.021	10.7
X1 (valve at top open)	0.027	8.4

Table 9. Average temperatures in Newcastle terminal Bins W1, X1 and X3 over trial period.

Sampling points	Temperatures (°C)		
	Bin W1	Bin X1	Bin X3
Headspace	16.3	15.9	18.9
1/3 down from bin top	–	18.0	19.5
Bottom	15.2	15.3	17.4
Ambient	14.0	14.0	17.6

Table 8. Purge data for the liquid N₂ trial, Newcastle terminal Bins W1, X1 and X3

Bin No.	Purge rate (m ³ /minute)	Purge time (h:min)	Liquid nitrogen consumed (t)	Purging efficiency (E ₃ , %)
W1	2.71	10:28	2.02	70
X1	2.98	10:28	2.22	55
X3	6.02	3:27	1.48	91

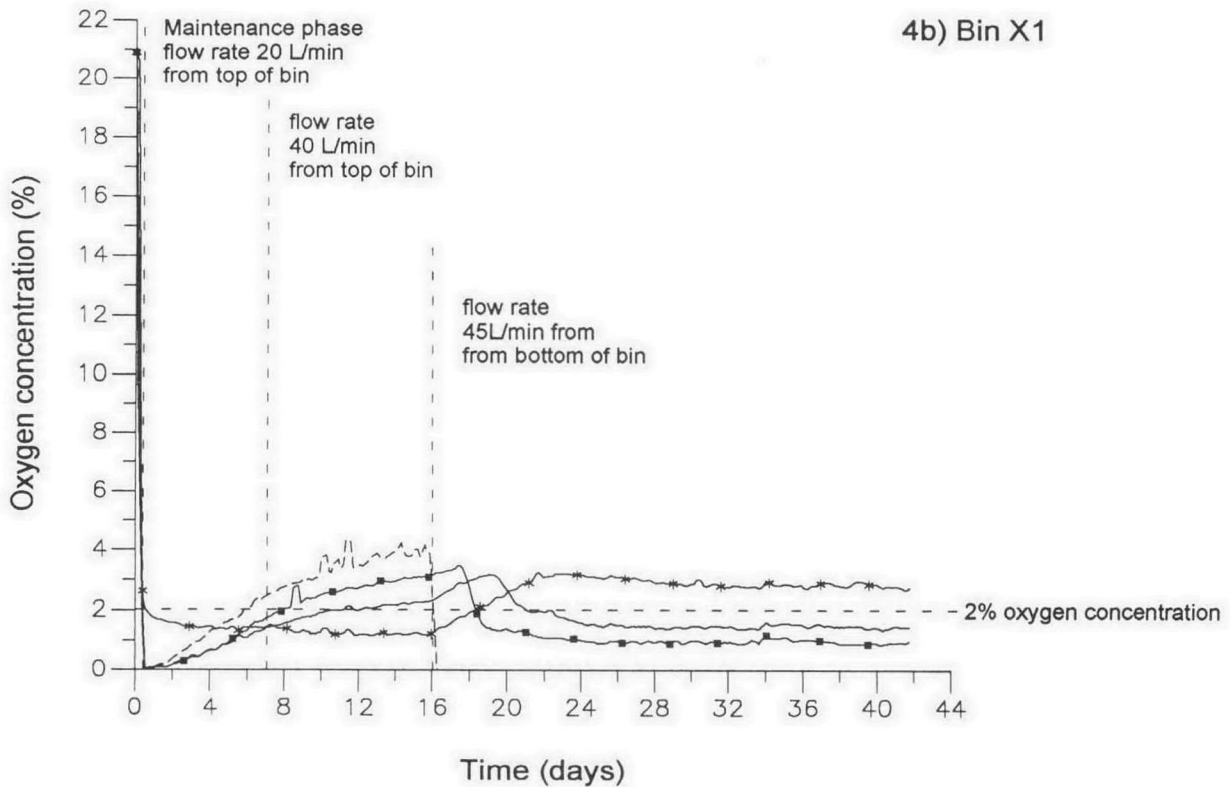
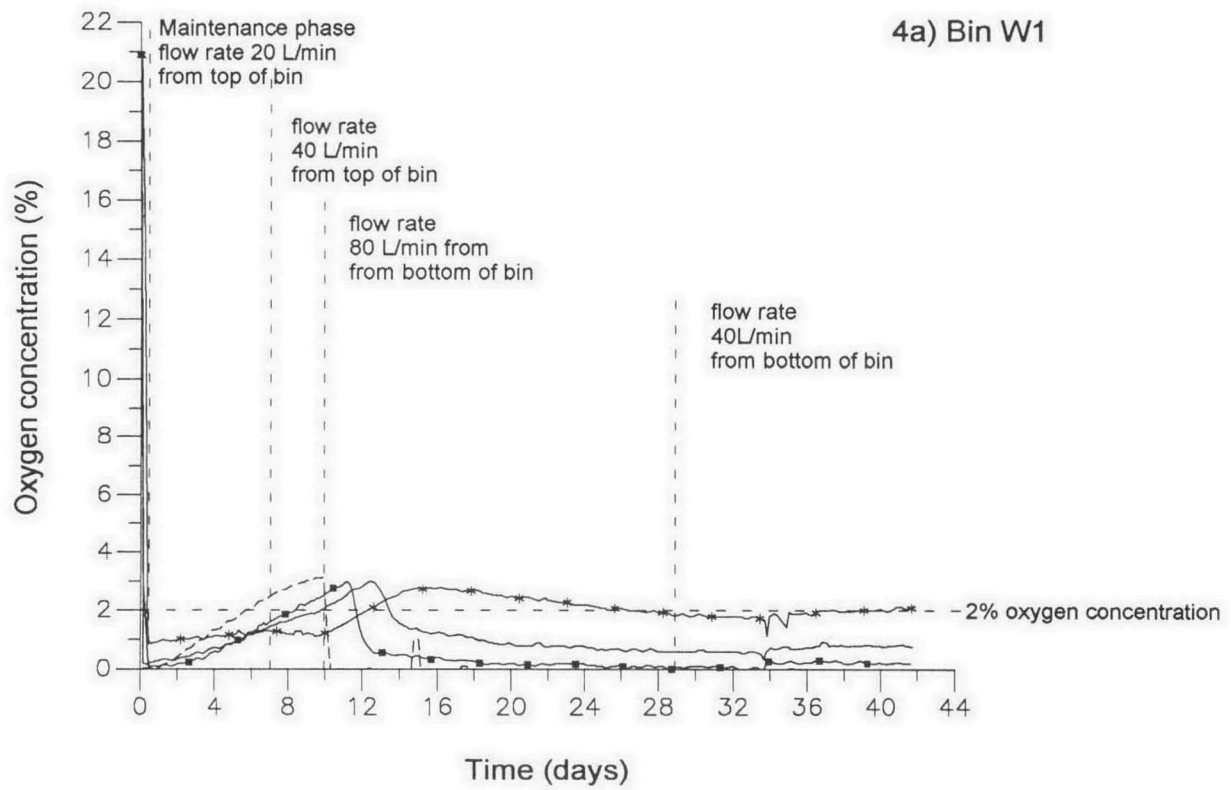


Fig. 4. Oxygen concentrations and position of maintenance gas line during maintenance phase in bins at the Newcastle terminal: (a) Bin W1; (b) Bin X1; and (c) Bin X3. --- Bottom of bin, —■— 1/3 up from bottom of bin, —●— 1/3 down from top of bin, —*— headspace.

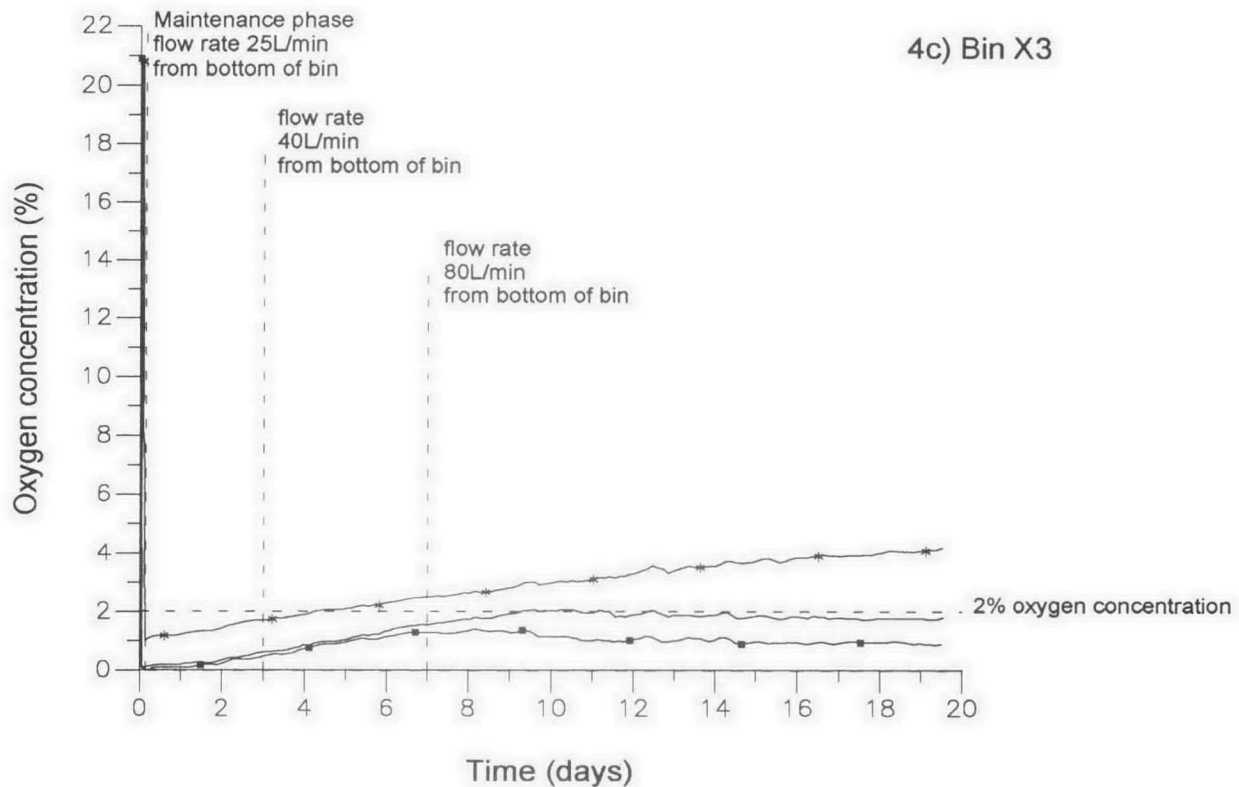


Fig. 4. – Cont'd

Discussion

The PSA unit purged the bulk of Bin A2 (the first bin to be purged) down to 0.7% O₂, close to the time expected (observed 11.5 days, calculated 9.8 days). However, in areas below the entry point of the gas, where mixing was slower or leakage occurred, the reduction of the O₂ concentration took longer. The low purging efficiency, 64%, indicates a substantial degree of mixing occurred in the grain bulk, presumably resulting from the low purge rate. Generator failures during the trial allowed the influx of air through the inlets and outlets raising the oxygen concentration and delaying the lowering of oxygen to specified levels in the bottom of Bin A1. Levels below 1.5% were eventually reached at all points in both bins (Fig. 1).

Maintenance using a pressure-demand system kept the O₂ concentration below 1% in all areas that had reached this level. The setting of the regulating system at 1.9 kPa was the lowest that could be obtained with the equipment available. A lower pressure setting (e.g. 100 Pa) may have been sufficient to maintain the low O₂ levels. As the bins were well sealed the maintenance rate was within the calculated rate (3× loss from temperature swings (calc.), i.e. assuming free mixing in headspace and no effect on the bulk concentration) to compensate for gas loss due to temperature swings in the headspace (Table 6).

The PSA unit output was insufficient to purge two large bins (10000 t capacity each) rapidly and was not sized optimally for the application. It is not known what effect a slow reduction of oxygen has on insect tolerance to low oxygen atmospheres. For simple logistic reasons more rapid purging

would have been preferred. Nevertheless, the trial showed, for the first time, that PSA systems could be used for generating N₂ CA atmospheres in grain storages if required.

The purge of the Newcastle bins using liquid nitrogen gave the expected pattern with dispersed plug flow in the bulk and some mixing in the headspace. The higher flow rate used in Bin X3 resulted in reduced mixing in the headspace and consequent increase in the efficiency of purging (Table 8). Increased efficiency with increased purging rate has been noted previously (Tranchino et al. 1980; Banks et al. 1980).

The initial input of the maintenance gas through the top of the bin was found in the case of Bins W1 and X1 to be unsatisfactory, with better results obtained from adding the N₂ at the base of the bin. A flow of >40 L/minute was required to keep the O₂ concentrations constant. Unlike Bins W1 and X1, even a higher maintenance rate of 80 L/minute from the base of Bin X3 still resulted in a rising headspace O₂ concentration. The mixing of the air leaking into the headspace with the low O₂ atmosphere resulted in a higher maintenance rate being required to flush out that extra volume of gas. Maintenance of this bin from the top may be more appropriate with the air leaking in being directly displaced by the maintenance gas. Leakage in Bins W1 and X1 may have been greater from the base of the bin than the headspace and also reduced by the sealing carried out on the wall to roof and wall to floor joints.

Although the concrete bins passed the pressure test of a minimum of 5 minutes they were less gastight than the steel bins (see Tables 1 and 2). Ventilation rates due to temperature swings in the headspace would be an important component in determining maintenance rates. The actual maintenance rates required were greater than the calculated rate, solely due to

temperature swings in the headspace. Other factors such as wind are expected to contribute to gas leakage and additional nitrogen input for maintenance (Banks and Annis 1984).

As oxygen levels of <18% become dangerous to humans, for safety reasons the bins were vented on completion of the trial. Although some (minimal) precautions are necessary to ensure adequate O₂ around CA treatment, the 'risk area' is usually small. The low oxygen decay rates, especially in the steel bins, would have allowed the grain to be protected for some time after shut down of maintenance. The venting of the grain would then occur naturally at outloading.

The setting up of the bins for CA required only a small capital outlay. Apart from the need initially for a well-sealed storage, the basic requirements were ducting, flow meters, a pressure sensor and an oxygen sensor to detect when the outlet from the bin has reached the required O₂ concentration to determine the end of purge. Up to a level where all leakage resulted from temperature and barometric cycling, expenditure in increasing the gastightness of the bin would be returned in reducing the maintenance rates.

The resulting costs of the two systems, taking into account hire charges and extrapolating running costs for a treatment time of 4 weeks [recommended dosage time at 23°C and 1% O₂ (Banks and Annis 1990)], were \$0.34/t for the PSA system and \$0.65/t for the liquid nitrogen system. In comparison with phosphine, at \$0.20/t for materials, the use of N₂ CAs is more expensive. However, this cost comparison does not take into account the cost of monitoring and logistic restrictions associated with phosphine fumigation. With these factors taken into account, use of nitrogen compares favourably with phosphine at the export terminals treated in these trials.

Conclusion

These trials were conducted at terminals where there is a particular requirement for a reliable supply of insect-free grain, to avoid the large costs associated with disruption of supply and demurrage incurred when insects are detected in a terminal on outloading. Both processes of atmosphere supply, liquid nitrogen and PSA systems, were demonstrated as feasible, though the more rapid pulldown achieved by the liquid-based system was clearly more convenient in the situations the two systems were used. Nitrogen CA treatments have been considered unsuitable in export terminals, but this is not so. Nitrogen treatments inherently require longer than phosphine or methyl bromide fumigations to achieve 100% kill of all stages of insect pests of stored products and do not allow quick throughput of grain through the terminal if applied only after infestation is detected. However, with correct management it is possible to apply such CA treatments even where timing can be critical. Treatments are applied immediately a particular parcel of grain is received, before loading to ship, thus maximizing chances of a successful outcome in insect-free condition.

The demand for residue-free grain, increasing regulations on the use of fumigants and availability of a method for fumigating commodities that are not recommended for treatments with phosphine or methyl bromide have all been factors in the consideration of N₂ treatments in situations where timing can be critical. The location of the terminals close to liquid nitrogen supplies, avoiding large transportation costs, and the availability of on-site nitrogen generators have made the use of CA more attractive. Although CAs are more expensive than phosphine fumigation in material costs, the changing market and environmental climate have resulted in low oxygen CA becoming a more acceptable alternative.

On the basis of this work, the Newcastle terminal management set aside 29000 t of storage which is regularly fumigated using controlled atmospheres. Over 60000 t of grain have been treated and exported from the terminal to date without stoppage for presence of insects on inspection of grain as it is loaded to ship.

Acknowledgments

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