

**Cooperative Research Centre
for National Plant Biosecurity**

Final Report

CRC50060

Phosphine fumigation of cool grain

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1. Executive Summary

This is the final report for CRC50060 'Phosphine fumigation of cool grain'. The project began as GRDC project DAQ00098 and was incorporated into the CRC National Plant Biosecurity on 1 July 2007 as part of the supplementary bid to include stored grain.

Aims and objectives

The biosecurity problem addressed was the need to understand and evaluate phosphine fumigation of cool grain (i.e. 20°C or less) as a means of controlling resistant biotypes of insect pests of stored grain which are major emergency plant pests (EPPs) threatening the grain industry.

The benefits of cooling and phosphine fumigation are that cooling preserves grain quality and reduces insect population growth, and phosphine kills insects and has a residue free status in all major markets.

The research objectives were to:

- conduct laboratory experiments on phosphine efficacy against resistant insects in cool grain, and determine times to population extinction.
- conduct laboratory experiments on phosphine sorption in cool grain and quantify.
- complete fumigation trials in three states (Queensland, Western Australia and New South Wales) on cool grain stored in sealed farm silos.
- make recommendations for industry on effective phosphine fumigation of cool grain.

Phosphine is used by growers and other stakeholders in the grain industry to meet domestic and international demands for insect-free grain. The project aim was to generate new information on the performance of phosphine fumigation of cool grain relevant to resistant biotypes. Effective control of resistant biotypes using phosphine to fumigate cool grain will benefit growers and other sectors of the grain industry, needing to fumigate grain in the cooler months of the year, or grain that has been cooled using aeration.

Key findings and implications

Successful fumigation of resistant insects requires sufficient phosphine concentrations for long enough to control all life stages of the insects. Lower temperatures maintain grain quality and reduce insect population growth, but phosphine is generally less effective at lower temperatures.

The relative importance of phosphine resistant strains of key pests changes with temperature. The most resistant Australian strains of two pests are known to respond similarly to phosphine but the project showed that one species became much harder to control in cool grain.

Sorption will be the major cause of loss of phosphine in a well-sealed silo. Rate of sorption was lower at lower temperatures, meaning that higher concentrations will be achieved for longer. Older grain tended to be less sorptive, so delaying fumigation may result in higher concentrations for longer. Sorghum was more sorptive than wheat so the margin for error is smaller for this grain.

In farm silos, phosphine gas was liberated from the aluminium phosphide formulations used despite the low grain and ambient temperatures (e.g. 10°C).

The results for the silo trials varied but three general observations were made. Lower concentrations tended to be measured deeper in grain mass. Lower concentrations tended to be measured on the northern side. Concentrations measured higher in the grain mass tended to peak earlier.

The silo trials showed that control of resistant insect populations is possible subject to good gas-tightness and adequate exposure periods. Exposure to cool temperature alone was not the cause of insect mortality.

Recommendations

Phosphine fumigation of cool grain to control resistant insect populations is possible subject to good gas-tightness and adequate exposure periods.

Growers and others planning to fumigate cool grain in sealable silos should aim for the current silo pressure test standard of a three minute halving time for a full silo or a five minute halving time for a partially full silo.

Delaying fumigation may result in more effective fumigation because the sorptive capacity grain decreases with age.

Growers and others planning to fumigate grain that has been aerated or has been harvested and stored in the cooler months of the year measure grain temperature before fumigation to ensure that an appropriate exposure period is used.

The use of recirculation methods, either forced or passive (e.g. thermosiphon), should be investigated to promote rapid and even distribution of phosphine from the headspace throughout the grain bulk.

1.1. Aims and objectives

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The benefits of cooling and phosphine fumigation are that cooling preserves grain quality and reduces insect population growth, and phosphine kills insects and has a residue free status in all major markets.

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- complete fumigation trials in three states (Qld, WA and NSW) on cool grain stored in sealed farm silos.
- make recommendations for industry on effective phosphine fumigation of cool grain.



Phosphine is used by growers and other stakeholders in the grain industry to meet domestic and international demands for insect-free grain. The project aim was to generate new information on the performance of phosphine fumigation of cool grain relevant to resistant biotypes. Effective control of resistant biotypes using phosphine to fumigate cool grain will benefit growers and other sectors of the grain industry, needing to fumigate grain that has been harvested and stored in the cooler months of the year, or grain that has been cooled using aeration.

1.2. Key findings

During the project research was completed in three interrelated areas:

- laboratory experiments on phosphine efficacy against resistant insect strains
- laboratory experiments on phosphine sorption in grain
- farm silo trials of phosphine fumigation of grain in three states

1.2.1 Laboratory experiments on phosphine efficacy

Data were generated on phosphine efficacy against resistant insects at cool temperature to fill data gaps in a large data set developed in earlier projects funded by the Grains Research and Development Corporation and the Australian Centre for International Agricultural Research. This large data set has been used to develop label recommendations, and much of it has been published in peer-reviewed journals (Daglish et al., 2002; Collins et al., 2005). The new results when combined with the existing data set will allow insights into the effects of temperature, exposure time and phosphine concentration on insect populations.

Efficacy experiments were conducted based on published methods (Daglish et al., 2002; Collins et al., 2005). Wheat containing mixed-age cultures (i.e. eggs, larvae, pupae and adults) were exposed to constant PH_3 concentrations at 15°C. This was the lowest temperature achievable in the controlled environment rooms used for this type of fumigation during the project. Samples were taken at intervals during each fumigation, any live adults were recorded, and if no live adults were found the samples were incubated at 25°C for 10 weeks to allow any surviving eggs, larvae or pupae to complete development. The two resistant strains chosen were a strong resistant strain of *Rhyzopertha dominica* and a weak resistant strain of *Sitophilus oryzae*. The resistance factors based on adult mortality were about 600 times at 48 h for *R. dominica* (Collins et al., 2002), and about 10 times at 24 h for *S. oryzae* (Daglish et al., 2002). In addition, a phosphine-susceptible strain of *Cryptolestes ferrugineus* was fumigated for comparison, because this species is a common pest in North America where grain tends to be cooler.

We believe that the strong resistant *R. dominica* strain and the weak resistant *S. oryzae* strain used in this study reflect the strongest PH_3 resistances present in these species in Australia. Published data show that there are minimal differences between these two strains when they are fumigated at 25°C (Daglish et al., 2002; Collins et al., 2005), but we found that the weak resistant strain of *S. oryzae* was much harder to control at 15°C (Table 1). This shows that the relative importance of resistant strains from different species depends on temperature. Complete control of strong resistant *R. dominica* and susceptible *C. ferrugineus* was achieved in less than 14 days at either 240 or 720 ppm. The trends for *S. oryzae* show that 99-100% reduction would be expected by 14 days under the same conditions (Table 2).

Table 1. Results of phosphine fumigation of mixed-age populations of *Rhyzopertha dominica*, *Sitophilus oryzae* and *Cryptolestes ferrugineus* in wheat at 15°C.

Days elapsed	Live adults (Mean ± SD, N =2) after 8 wk incubation at 25°C.		
	<i>R. dominica</i> (Strong resistant)	<i>S. oryzae</i> (Weak resistant)	<i>C. ferrugineus</i> (Susceptible)
Concentration = 0.3 mg L ⁻¹ (240 ppm)			
0	783.0 ± 335.2a	4016.0 ± 340.8a	280.5 ± 87.0a
6	6.0 ± 1.4b	652.0 ± 5.7b	5.0 ± 0.0b
7	0.5 ± 0.7c	351.5 ± 2.1b	1.0 ± 1.4c
8	0.0 ± 0.0c	311.5 ± 94.0b	0.0 ± 0.0c
9	0.0 ± 0.0c	96.0 ± 87.7c	0.0 ± 0.0c
10	0.0 ± 0.0c	97.5 ± 41.7c	0.0 ± 0.0c
11	0.0 ± 0.0c	7.5 ± 3.5d	0.0 ± 0.0c
Concentration = 1 mg L ⁻¹ (720 ppm)			
0	1048.0 ± 306.9a	4325.0 ± 312.5a	317.0 ± 142.8
8	82.0 ± 5.7b	618.0 ± 281.4a	0.0 ± 0.0
9	1.0 ± 1.4c	456.0 ± 176.8ab	0.0 ± 0.0
10	0.0 ± 0.0c	14.5 ± 2.1bc	0.0 ± 0.0
11	0.0 ± 0.0c	70.0 ± 97.6bc	0.0 ± 0.0
12	0.0 ± 0.0c	47.0 ± 63.6bc	0.0 ± 0.0
13	0.0 ± 0.0c	0.5 ± 0.7c	0.0 ± 0.0

Within each dose and species, means in columns followed by different letters are significantly different ($p < 0.05$) based on analysis of transformed data.

Table 2. Response of mixed age cultures of phosphine-resistant *Sitophilus oryzae* to phosphine at 15°C.

Phosphine (ppm)	LT _{99.9} (95% fiducial limits) (days)
240	13 (12-17)
720	13 (11-25)

1.2.2 Laboratory experiments on phosphine sorption in grain

Successful fumigation of grain depends on achieving high enough concentrations for long enough to control the most tolerant developmental stages which are usually the eggs or pupae. Loss of phosphine gas from silos will occur through leakage and sorption by grain. While leakage can be minimised by using well maintained sealable silos, it is difficult to reduce sorption. Limited practical information is available in the literature so experiments were undertaken to generate data to help interpret the results of trials in sealable farm silos.

The general methods used in the sorption experiments have been published (Daglish and Pavic, 2008). Glass flasks (2 L capacity) filled to 95% of volumetric capacity were injected with PH₃ at a dose of 1.5 mg L⁻¹ based on the volume of the empty flasks. This dose equates to an application rate of 1.5 tablets of aluminium phosphide per cubic metre of empty silo volume. Phosphine concentration was measured at intervals during storage beginning 2 h after injection and ending 7-11 days after injection. Grain was collected either at harvest or after a period of storage in farm silos and generally stored frozen until shortly before the experiments began.

Sorption by grain can reduce the amount of PH₃ to which insects are exposed. The experiments showed that from 1-2 h after injection loss of gaseous phosphine could be described by simple exponential decay equations (i.e. $C_t = C_0 e^{-kt}$) with the implication that phosphine is lost at a constant rate per day. Percentage daily loss is calculated as $100 \cdot (1 - e^{-k})$.

Initial experiments were conducted on wheat at 25°C (Table 3). Percentage sorption per day was directly proportional to filling ratio, and was negatively correlated with dose for any given filling ratio. Based on the results, a ten-fold increase in dose would result in a halving of the percentage daily loss. Wheat was less sorptive if it was fumigated for a second time. Two reasons for the higher sorption in the recently harvested wheat could be the age of the wheat or the variety used.

Table 3. Effects of dose and filling ratio on daily phosphine sorption (%) in wheat (25°C, 55% RH). (Phosphine measured at intervals beginning 1 h after injection.)

Dose (mg L ⁻¹) ^a	Filling ratio			
	0.25	0.5	0.75	0.95
Commercially supplied wheat				
0.1	1.7	4.6	7.1	9.8
0.3	1.9	3.2	4.9	7.1
0.6	2.2	4.2	5.2	6.9
0.6 ^b	0.0	1.2	2.8	4.2
1	1.5	2.0	4.1	5.5
1.5	0.7	1.9	3.1	4.6
Recently harvested wheat				
1	1.4	4.8	6.0	8.4

^aBased on volume of empty flask. ^bRepeat fumigations of the wheat previously fumigated at this dose.

The effects of dose and temperature on sorption were determined in experiments on recently-harvested sorghum (Table 4). Temperature had a pronounced effect on sorption with rate of sorption at 15°C being about half the rate at 25°C. As was shown for wheat, rate of sorption was negatively correlated with dose.

Table 4. Effects of dose and temperature on daily phosphine sorption (%) in recently harvested sorghum (13.7% m.c.). (Phosphine measured at intervals beginning 2 h after injection.)

Dose (mg L ⁻¹) ^a	Temperature (°C)	
	15	25
0.3	20.5	38.0
0.6	17.8	32.7
1	14.9	29.2
1.5	12.8	23.8

^aBased on volume of empty flask.

Phosphine sorption was investigated in four samples of sorghum which had been stored in farm silos for several months after harvest. The samples came from four different sorghum bulks which were to be fumigated in silo trials. Each bulk had been stored in farm silos for 3.5 months from harvest (Table 5). Sorptive capacity varied among the four samples. Sorghum samples from 2006 were less sorptive than the samples from 2008 despite both lots of sorghum having been stored for similar periods of time. Moisture content did not appear to be

the reason for the differences. For each sample phosphine was sorbed more slowly at the lower test temperature than at the reference temperature of 25°C.

Table 5. Effect of temperature on daily phosphine sorption (%) in sorghum sampled from farm silos and fumigated at 1.5 mg L⁻¹ of flask volume. (Phosphine measured at intervals beginning 2 h after injection.)

Sample	Year	Moisture content (%)	Temperature (°)	Sorption (%)
Trial 1	2006	12.2	15	6.9
			25	11.9
Trial 2	2006	13.2	15	7.0
			25	13.2
Trial 3	2008	13.2	10	5.1
			25	18.3
Trial 4	2008	13.6	16	10.6
			25	22.1

The possibility was investigated that time in storage before fumigation affected the rate at which grain sorbs phosphine. Data from fumigations of sorghum after various periods of storage at 25°C show that rate of sorption was lower the longer the sorghum had been stored before being fumigated for the first time (Table 6). There was only a slight decline in moisture content during storage indicating that time in storage rather than change in moisture content was important.

Table 6. Effect of storage at 25°C on daily phosphine sorption in sorghum fumigated at 1.5 mg L⁻¹ of flask volume. (Phosphine measured at intervals beginning 2 h after injection.)

Age of grain (days)	Moisture content (%)	Daily sorption (%)
5	14.4	34.6
33	14.3	22.9
111	13.6	13.6

Another experiment was conducted in which sorghum was stored at either 15 or 25°C and rate of sorption was determined for samples taken during storage (Table 7). The fumigations were conducted at the same temperature at which the sorghum had been stored. Rate of sorption tended to decrease with time spent in storage. Rate of sorption was lower when a batch of sorghum was refumigated, but this could be explained by time in storage rather than refumigation per se. Rate of sorption was always lower at 15°C compared with 25°C.

After establishing that time in storage was an important factor affecting rate of sorption in sorghum, a similar experiment was done on wheat (Table 8). The same general trends were observed with wheat as had been observed with sorghum.

In the experiments above phosphine concentration declined exponentially from 1-2 hours after injection of phosphine. Fumigations lasting 24 h were undertaken on wheat and sorghum at 25°C to estimate the free-air-space in grain bulks and to determine the pattern of phosphine loss in the initial stages of sorption (Table 9). Flasks half-filled with grain were injected with phosphine and inverted twice to ensure that the phosphine gas was distributed evenly in the headspace and throughout the free airspace in the grain bulk. Concentration declined during the 24 h but was most rapid in the first hour after injection. This rapid initial phase of sorption lasting about 1 h does not fit the exponential pattern of decline observed from 2 h onwards in

the earlier fumigations which lasted up to 11 days. The concentration shortly after injection was about 1.5 times the dose, suggesting that the intergranular airspace occupied about a third of wheat and sorghum bulks because these flasks were half filled with wheat.

Table 7. Effect of storage at two temperatures on daily phosphine sorption in sorghum fumigated at 1.5 mg L⁻¹ of flask volume. (Phosphine measured at intervals beginning 2 h after injection.)

Age of grain (days) ^a	15°C		25°C	
	Moisture content (%)	Daily sorption (%)	Moisture content (%)	Daily sorption (%)
6	15.4	22.4	15.1	37.5
20	15.0	15.6	14.8	27.9
27 ^b	15.3	12.7	15.1	21.6
48	14.7	13.3	14.3	20.7
69 ^b	14.4	10.0	14.1	15.1
97	14.3	11.9	14.1	16.1

^aIgnoring time stored at -15°C. ^bWheat fumigated after 27 and 69 days storage had been fumigated previously after 6 and 48 days storage respectively.

Table 8. Effect of storage at two temperatures on daily phosphine sorption in wheat fumigated at 1.5 mg L⁻¹ of flask volume. (Phosphine measured at intervals beginning 2 h after injection.)

Age of grain (days) ^a	15°C		25°C	
	Moisture content (%)	Daily sorption (%)	Moisture content (%)	Daily sorption (%)
5	12.2	4.2	12.3	9.4
19	12.1	3.5	12.3	6.9
26 ^b	12.2	3.4	12.3	7.6
47	12.2	3.9	12.3	6.4
68 ^b	12.2	3.2	12.3	4.8
103	12.4	4.4	12.3	5.2
124 ^b	12.3	2.8	12.1	4.1
159	12.3	2.1	12.3	3.7

^aIgnoring time stored at -15°C. ^bWheat fumigated after 26, 68 and 124 days storage had been fumigated previously after 5, 47 and 103 days storage respectively.

Table 9. Phosphine concentrations (Mean ± SD, N = 4) in flasks containing wheat or sorghum (0.5 filling ratio) fumigated for 24 h (25°C, 55% RH).

Grain	Time elapsed (h)	Phosphine (mg L ⁻¹)
Wheat (12.4% mc)	0.05 (3 min)	2.2097 ± 0.0387a
	1	2.0158 ± 0.0122b
	2	1.9777 ± 0.0401b
	24	1.8902 ± 0.0203c
Sorghum (13.4% mc)	0.033 (2 min)	2.1851 ± 0.0405a
	1	2.0456 ± 0.0582b
	2	1.9506 ± 0.0628c
	24	1.7188 ± 0.0182d

Within grain type means followed by different letters are significantly different ($P < 0.05$).

The key findings from the sorption experiments are:

- sorghum is more sorptive than wheat so the margin for error is smaller when fumigating this grain.
- the rate of sorption is lower at lower temperature meaning that fumigations of cool grain will achieve higher phosphine concentrations for longer.
- older grain tends to be less sorptive so delaying fumigation will result in higher concentrations for longer.

1.2.3 Phosphine fumigation of grain in sealable farm silos

Phosphine fumigation of cool grain was undertaken in sealable silos in three states (Queensland, Western Australia and New South Wales) because of the need to collect data under practical conditions. All silos used were cylindrical with conical tops and bases.

The general approach used in the fumigation trials has been described (Newman et al. (2004)). Essentially, silos containing grain were pressure-tested and then fumigated using aluminium phosphide as the source of phosphine. Aluminium phosphide was placed into the headspace to avoid potential contamination of the grain with unreacted residues, and phosphine concentrations were measured within the grain bulk during the fumigations. In all fumigations, concentrations were measured at approximately 3-5 points along the vertical axis of the silo, and in some cases concentrations were measured at other locations around silo. Concentrations were measured by attaching nylon sample tubing to electronic phosphine monitors. Grain and ambient temperatures were also measured. Percentage phosphine loss per day (sorption + leakage) was estimated using regression. The mean concentration for all measurement points was determined for each day and an exponential equation fitted to the means for the part of the exposure period where mean concentration was declining. Efficacy was estimated by comparing concentrations achieved with known dosage requirements for resistant strains or by placing cages containing mixed-age populations of insects into the grain bulks before fumigation.

1.2.3.1 Trials in Queensland

Four trials were completed in Queensland on sorghum stored in a 158 m^3 silo which was 8 m high and 6 m wide. All four trials had the following basic set up. The dose of phosphine applied was 1.5 tablets per cubic metre of silo capacity yielding 1.5 g of phosphine per cubic metre. Phosphine was measured at 14 points within the silo. There were six points along the central vertical axis i.e. 0.5 (headspace), 2, 3.5, 5, 6.5 and 7 m. Measurements were measured also at eight points around the silo referred to as the upper and lower compass points. There were four points 2.7 m from the top of the silo and 0.5 m from the silo wall corresponding to north, south, east and west. There were four points 4.9 m from the top of the silo and 0.15 m from the silo wall corresponding to north, south, east and west. Temperature was measured at 0.5, 2, 3.5, 6.5 m points along the central vertical axis, and ambient temperature was monitored with an electronic weather station nearby. Grain temperature was calculated as the mean of the temperatures measured at 2, 3.5 and 6.5 m.

Extra data were obtained in Trials 3 and 4. Cages containing mixed-age populations of strong resistant *R. dominica* and weak resistant *S. oryzae* were placed 2.4 and 4.9 m from the

top of the silo and 1 m from the silo wall. Phosphine concentration and temperature were measured at these upper and lower cage points. Temperature was measured also at the upper compass points. As controls, identical cages of insects were stored in a controlled environment cabinet for the same period and approximate temperature as with the fumigation. The mean temperatures for the controls were 10.1 and 13.6 for Trials 3 and 4 respectively. The sorghum used in Trial 4 had a natural infestation of the maize weevil, *Sitophilus zeamais*, so pre- and post fumigation samples of sorghum were collected to determine fumigation success.

In Trial 1, the phosphine concentration x time profiles were similar at all measuring points, with maximum uniformity achieved by 10 days (Figure 1). The lowest mean concentration achieved over 21 days was 709 ppm at the lower north point. Loss of phosphine was estimated to be 7% per day based on analysis of the mean concentrations for days 10-21. Laboratory testing of this sorghum showed that rate of sorption at 15°C was 7% per day so sorption at the trial grain temperature of 11.5°C would have been low (Table 5).

In Trial 2, the phosphine concentration x time profiles varied greatly depending on where the measurements were made, with maximum uniformity achieved by about 10 days (Figure 2). Higher concentrations were achieved earlier in the upper parts of the silo (Figure 2a). Concentration peaks were observed in the first 5 days for the 0.5, 2 and 3.5 m measurement points and the second 5 days for the 5, 6.5 and 7 m measurement points. In relation to the compass point measurements, the upper south point had the highest mean concentration and lower north point had the lowest mean concentrations (Figure 2b). The highest mean concentrations achieved on the upper level were at the south point followed by the west, east and north points. Similarly, the highest mean concentrations achieved on the lower level were at the south point followed by the west, east and north points. The lowest mean concentration achieved over 14 days was 271 ppm at the lower north point. Loss of phosphine was estimated to be 10% per day based on analysis of the mean concentrations for days 6-14. Based on laboratory testing of this sorghum at 15 and 25°C, the rate of sorption at the trial temperature of 21.7°C would have been between 7 and 13% (Table 5).

Trial 3 was similar to Trial 2 in that the phosphine concentration x time profiles varied greatly depending on where the measurements were made (Figure 3). More than 10 days was required to achieve maximum uniformity. Higher concentrations were achieved earlier in the upper parts of the silo (Figure 3a). In relation to the compass point measurements, the upper south point had the highest mean concentration and lower north point had the lowest mean concentrations (Figure 3b). The highest mean concentrations achieved on the upper level were at the south point followed by the east, west and north points. Similarly, the highest mean concentrations achieved on the lower level were at the south point followed by the east, west and north points. The lowest mean concentration achieved over 14 days was 351 ppm at the lower north point. Loss of phosphine was estimated to be 5% per day based on analysis of the mean concentrations for days 10-14. This sorghum sorbed phosphine at the rate of 5% per day at 10°C in the laboratory, compared with the total loss of 5% per day observed in the trial at a grain temperature of 10.6°C (Table 5).

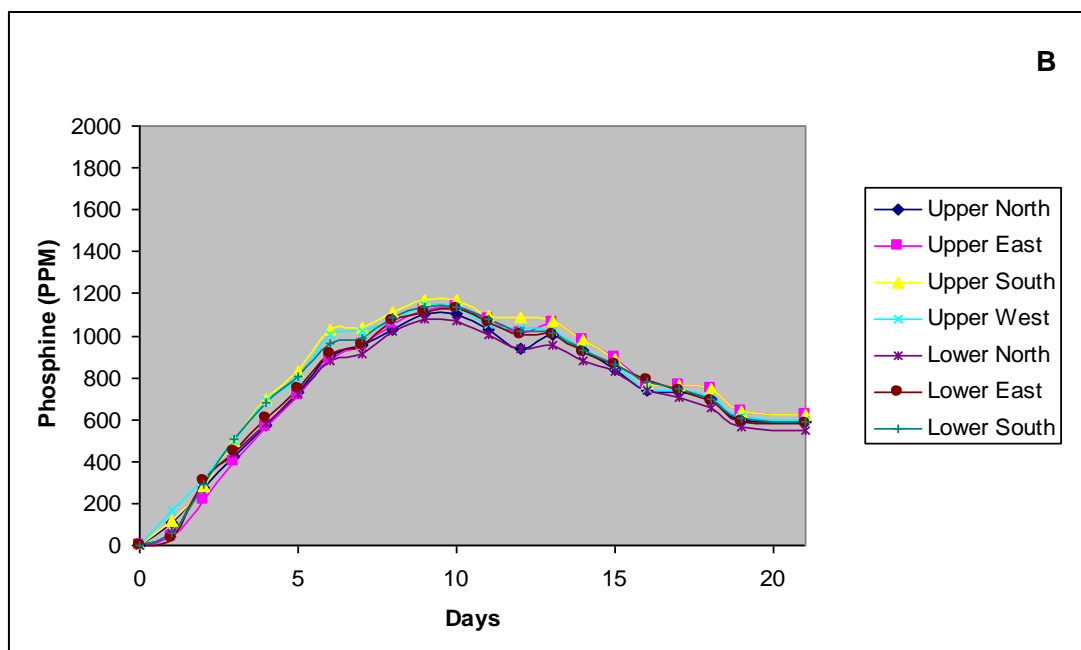
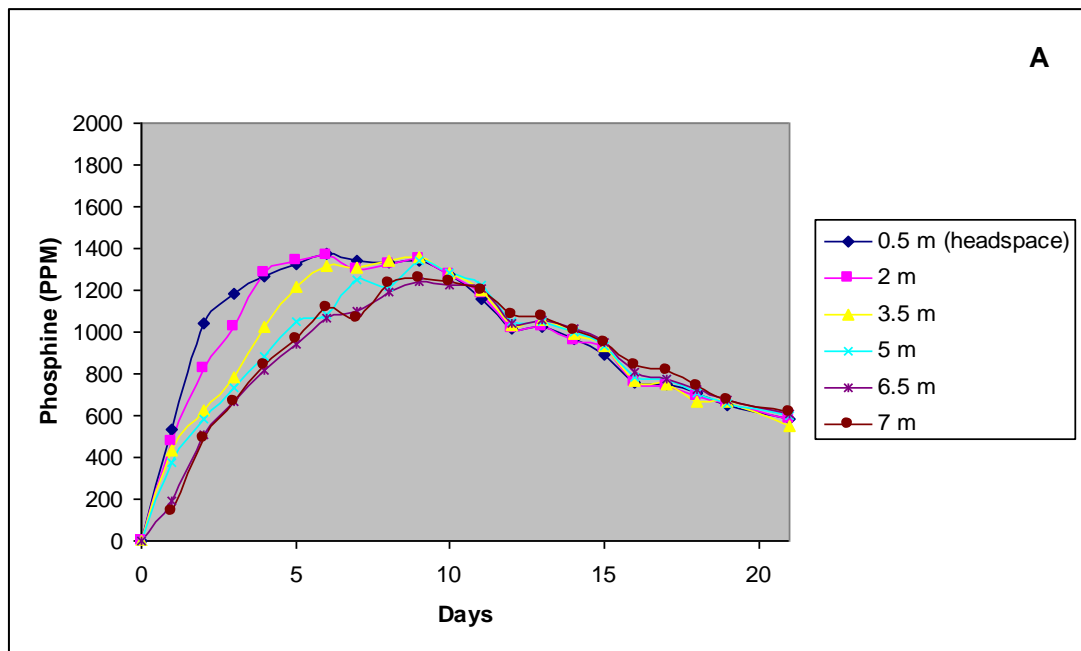


Figure 1. Phosphine concentrations measured at different locations in a sealed silo containing sorghum (12.0% mc). (A) - sampling points along central vertical axis, (B) - peripheral sampling points. Grain temperature was 11.5°C and mean minimum and maximum air temperatures were 7.6 and 22.6°C (mean 15.1°). The silo pressure halving time was 165 seconds.

Trial 4 was similar to Trials 2 and 3 in that the phosphine concentration x time profiles varied greatly depending on where the measurements were made (Figure 4). Maximum uniformity was not achieved by the end of the trial (10 days). Higher concentrations were achieved earlier in the upper parts of the silo (Figure 4a). In relation to the compass point measurements, the upper and lower south points had the highest mean concentrations and lower north point had the lowest mean concentration (Figure 4b). The highest mean concentrations achieved on the upper level were at the south point followed by the east, west and north points. Similarly, the highest mean concentrations achieved on the lower level were at the south point followed by the east, west and north points. The lowest mean concentration achieved over 14 days was 150 ppm at the lower north point. Loss of phosphine could not be estimated because mean concentration peaked too late, but this sorghum sorbed phosphine at the rate of 11% per day at 16°C in the laboratory, and the grain temperature during the trial was 15.8°C (Table 5).

Fumigation at grain temperatures of less than 15°C is not recommended but in practice would be occurring because growers are unlikely to measure grain temperature before fumigating. Figure 5 shows the concentration x time profiles measured at the location of the upper and lower cages in Trials 3 and 4, and Tables 10 and 11 show the results of exposure of mixed-age populations of resistant *R. dominica* and *S. oryzae* to phosphine in Trials 3 and 4. Table 12 show the effect of phosphine fumigation on a natural infestation of *S. zeamais* in Trial 4. This population was confirmed by laboratory bioassays to be susceptible to phosphine (Table 13). These results show that high levels of control are achievable at grain temperatures as low as 10°C. The lowest mean concentration achieved over 14 days in Trial 3 was 351 ppm at the lower north point. This is lower than the mean of 409 ppm measured at the lower cages, suggesting that parts of the sorghum bulk would have needed longer than 14 days to achieve high levels of control. The lowest mean concentration achieved over 10 days in Trial 4 was 150 ppm at the lower north point. This is lower than the mean of 281 ppm measured at the lower cages, suggesting that parts of the sorghum bulk would have needed longer than 10 days to achieve high levels of control.

Exposure of caged insects was not possible in Trials 1 or 2 but these two fumigations were likely to have achieved high levels of control. The lowest mean concentration achieved over 21 days in Trial 1 was 709 ppm at the lower north point, and the grain temperature in this trial was 11.5°C. The lowest mean concentration achieved over 14 days in Trial 2 was 271 ppm at the lower north point, and the grain temperature in this trial was 21.7°C. Based on the results achieved in Trials 3 and 4 (Tables 10 and 11) high levels of control would have been expected in Trials 1 and 2 as well.

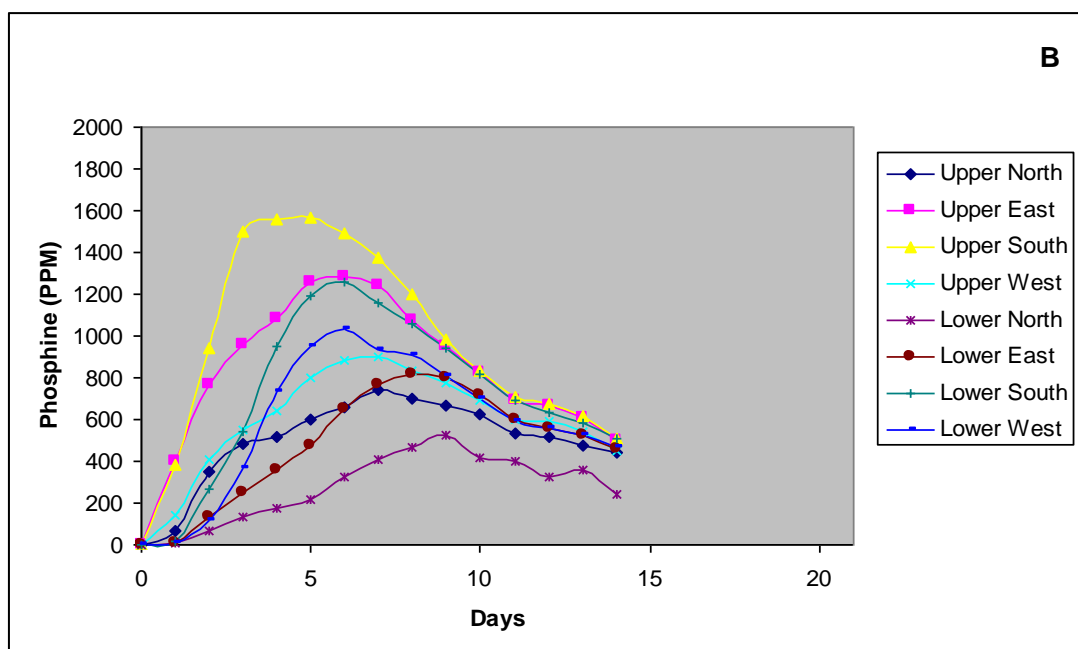
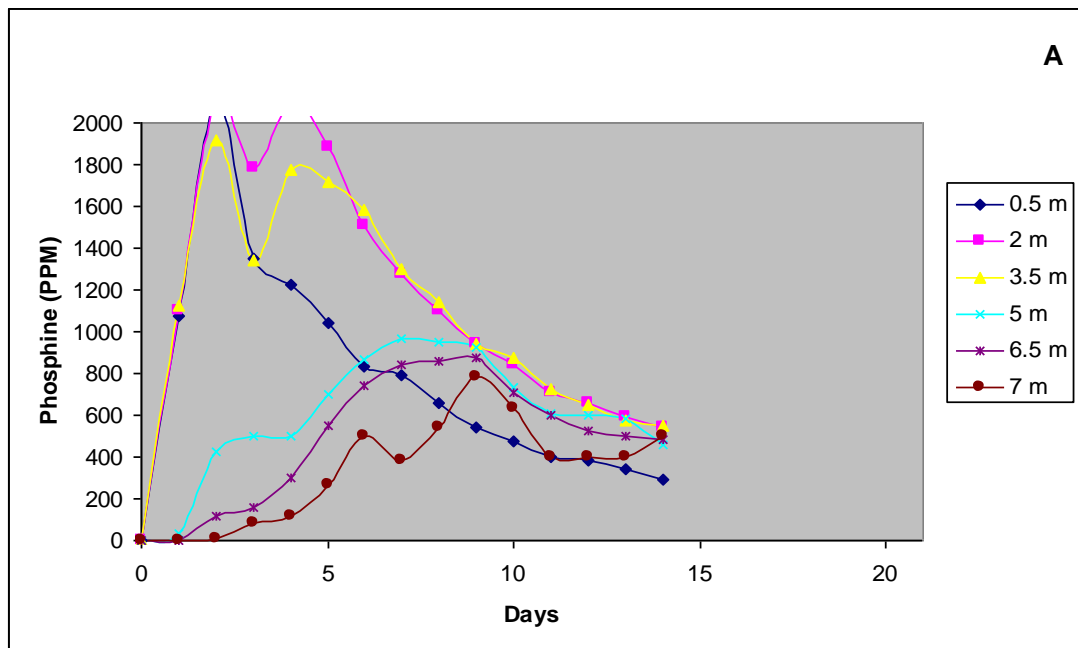


Figure 2. Phosphine concentrations measured at different locations in a sealed silo containing sorghum (12.7% mc). (A) - sampling points along central vertical axis, (B) - peripheral sampling points. Grain temperature was 21.7°C and mean minimum and maximum air temperatures were 11.3 and 26.7°C (mean 19.0°). The silo pressure halving time was 170 seconds.

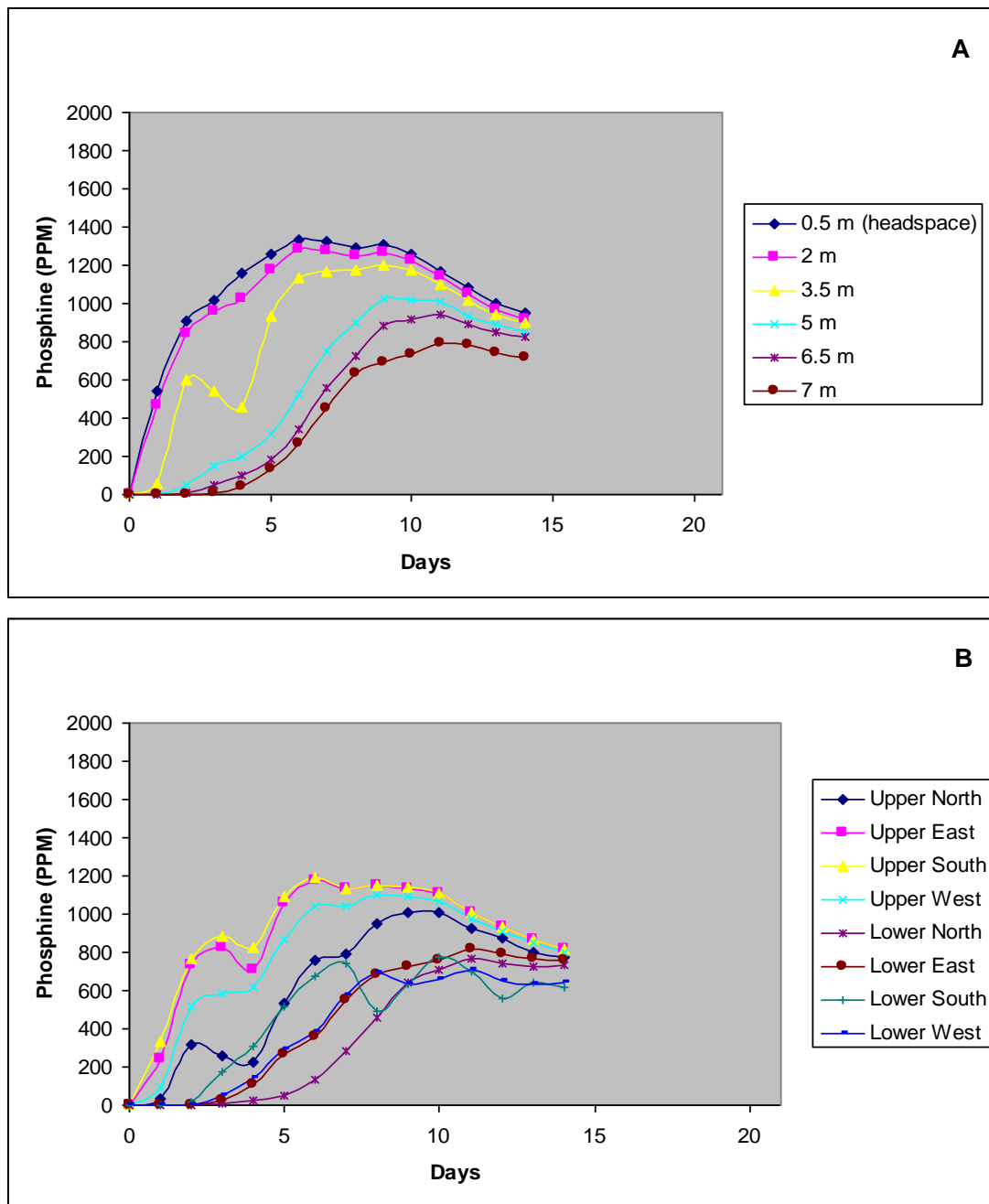


Figure 3. Phosphine concentrations measured at different locations in a sealed silo containing sorghum (13.0% mc). (A) - sampling points along central vertical axis, (B) - peripheral sampling points. Grain temperature was 10.6°C and mean minimum and maximum air temperatures were -0.2 and 17.1°C (mean 8.5°C). The silo pressure halving time was 120 seconds.

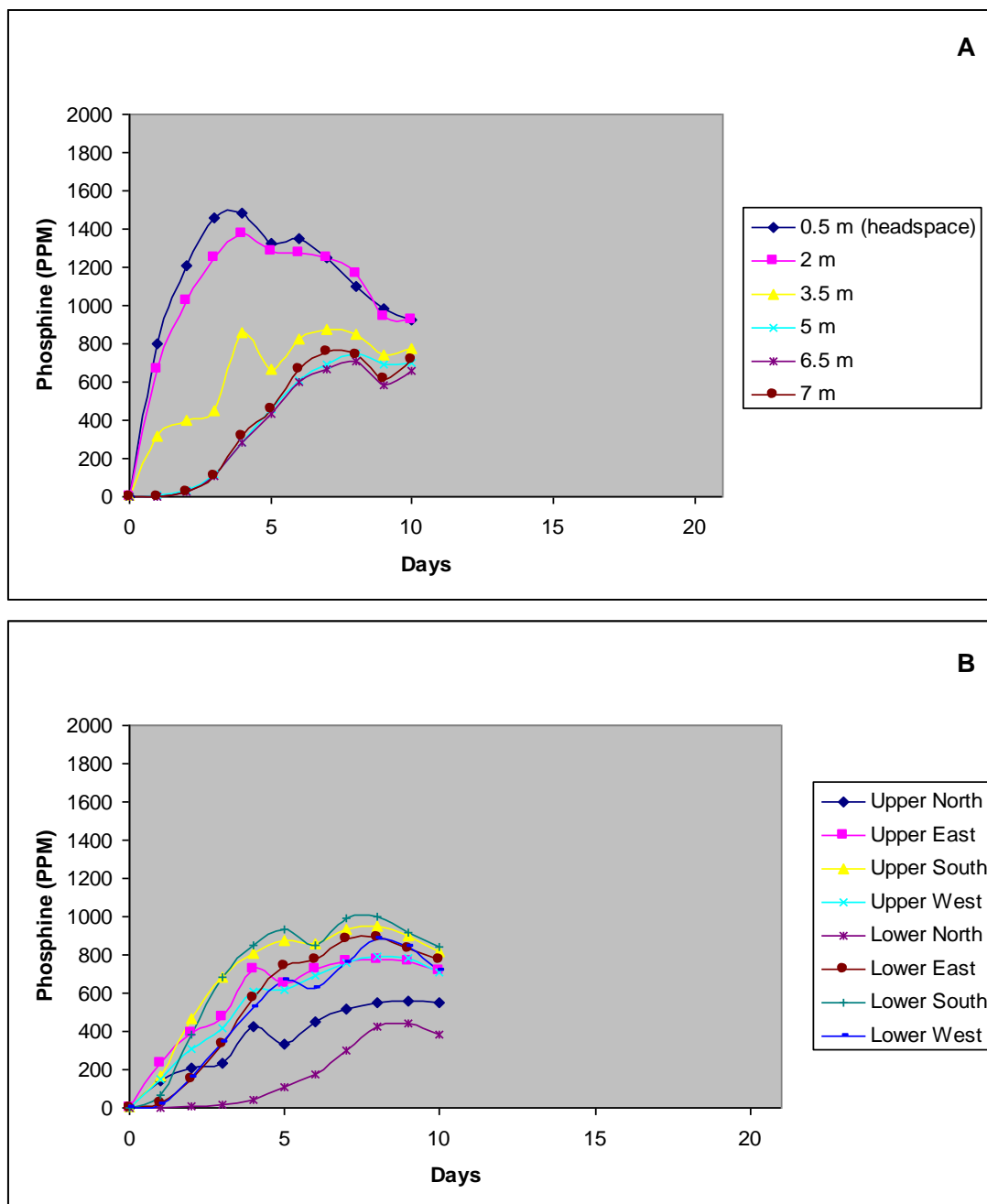


Figure 4. Phosphine concentrations measured at different locations in a sealed silo containing sorghum (13.2% mc). (A) - sampling points along central vertical axis, (B) - peripheral sampling points. Grain temperature was 15.8°C and mean minimum and maximum air temperatures were -3.6 and 17.1°C (mean 6.8°). The silo pressure halving time was 120 seconds.

Table 10. Effect of phosphine fumigation of sorghum on mixed-age populations of *Rhyzopertha dominica* (strong resistant) and *Sitophilus oryzae* (weak resistant) exposed in cages.

Strain	Cages	Live adults (mean \pm SD, n = 3)	
		4 d ^a	6 wk ^a
July 2008 fumigation			
<i>R. dominica</i>	Control	58.3 \pm 21.8	66.3 \pm 28.0
	Upper	0.0 \pm 0.0	0.0 \pm 0.0
	Lower	0.0 \pm 0.0	2.0 \pm 1.0
<i>S. oryzae</i>	Control	14.0 \pm 5.3	334.3 \pm 103.3
	Upper	0.0 \pm 0.0	9.7 \pm 7.4
	Lower	0.0 \pm 0.0	13.3 \pm 5.1
August 2008 fumigation			
<i>R. dominica</i>	Control	132.3 \pm 36.7	162.3 \pm 48.6
	Upper	0.0 \pm 0.0	1.3 \pm 0.6
	Lower	0.0 \pm 0.0	1.3 \pm 0.6
<i>S. oryzae</i>	Control	97.0 \pm 6.6	402.7 \pm 43.5
	Upper	0.0 \pm 0.0	4.7 \pm 2.3
	Lower	0.0 \pm 0.0	2.0 \pm 1.7

^aMixed-age populations were incubated at 25°C after removal from the silos.

Table 11. Population reduction in mixed-age populations of *Rhyzopertha dominica* (strong resistant) and *Sitophilus oryzae* (weak resistant) exposed in cages during phosphine fumigation of sorghum.

Cages	Temperature (°C)	Phosphine (ppm) ^a	Reduction (%)	
			<i>R. dominica</i>	<i>S. oryzae</i>
July 2008 fumigation (14 d duration)				
Upper	10.7	912 \pm 295	100	97.1
Lower	10.2	409 \pm 333	98.4	96.2
August 2008 fumigation (10 d duration)				
Upper	13.7	777 \pm 224	99.6	99.1
Lower	13.2	281 \pm 232	99.6	99.6

^aMean \pm SD

Table 12. Effect of phosphine fumigation on natural infestation of *Sitophilus zeamais* in sorghum.

Sample	Live adults (mean \pm SD, n = 15)	
	4 d after collection	6 wk after collection
Pre-fumigation	4.3 \pm 5.5	176.9 \pm 282.3
Post-fumigation	0.0 \pm 0.0	0.0 \pm 0.0

Table 13. Response to phosphine of the *Sitophilus zeamais* population collected from infested sorghum in 2008. (Adults exposed for 20 h and mortality assessed 14 days later.)

Measurement	Result
LC ₅₀ (95% fiducial limits) (mg L ⁻¹)	0.0054 (0.0049-0.0057)
LC _{99.9} (95% fiducial limits) (mg L ⁻¹)	0.020 (0.016-0.027)
Mortality (%) at FAO discriminating dose	100

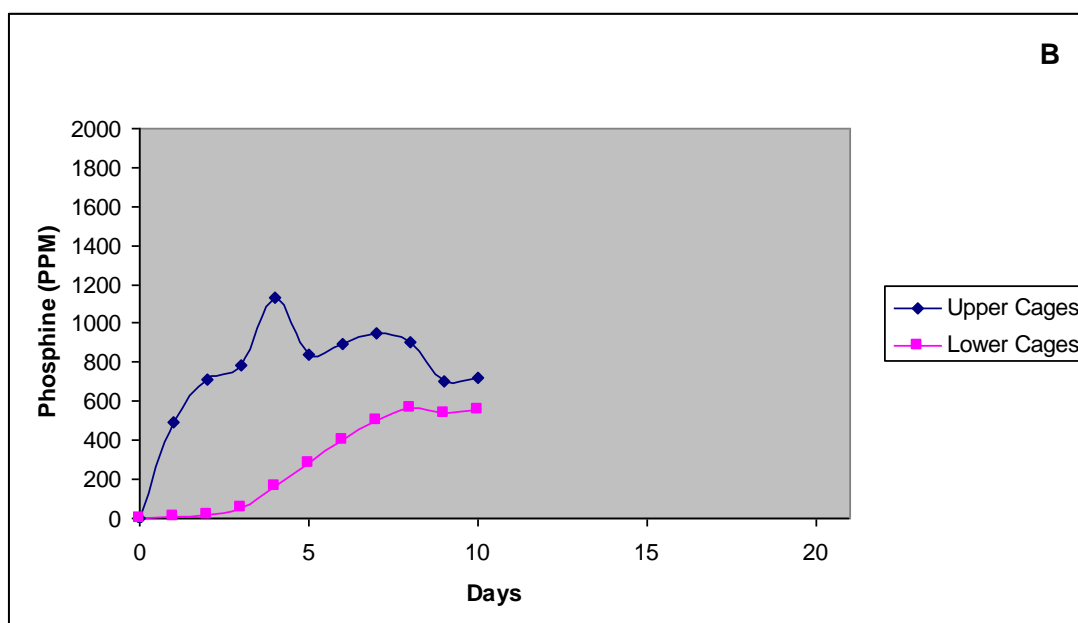
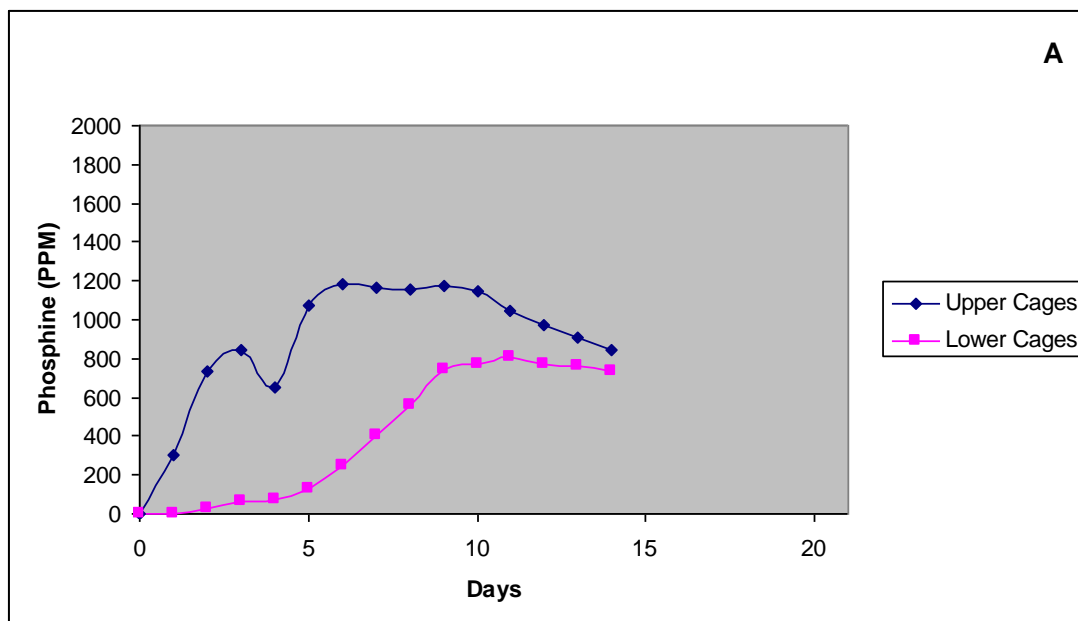


Figure 5. Phosphine concentrations measured at the location of the upper and lower cages in silo fumigations of sorghum. (A) – Trial 3 - grain temperature was 10.6°C and mean minimum and maximum air temperatures were -0.2 and 17.1°C (mean 8.5°C). The silo pressure halving time was 120 seconds. (B) Trial 4 - grain temperature was 15.8°C and mean minimum and maximum air temperatures were -3.6 and 17.1°C (mean 6.8°). The silo pressure halving time was 120 seconds.

1.2.3.2 Trials in Western Australia

Ten trials were completed in Western Australia in 90 m³ silos which were 7.1 m high and 4.5 m wide. There were five trials on wheat, four trials on barley and one on field peas. The dose of phosphine applied was 1.1 g m⁻³ of silo volume. Phosphine was measured at six points along the central vertical axis i.e. 0.5 (headspace), 1.5, 3, 4, 5.5 and 7.1 m, and temperature was measured at the 1.5 and 4 m points. Ambient temperature was recorded on site with an electronic data logger. Grain temperature was calculated as the mean of the temperatures measured at 1.5 and 4 m.

Wheat was fumigated for 21 days in Trial 1 (Figure 6). The phosphine concentration x time profiles achieved depended on where they were measured. Concentrations peaked earlier and tended to be higher in the upper parts of the grain bulk. Maximum mixing had occurred by day 7 with almost identical concentrations at all depths except at the 7 m point which was almost always lower with a mean concentration of 348 ppm. Loss of phosphine was estimated to be 7% per day based on analysis of the mean concentrations for days 7-21.

Wheat was fumigated for 22 days in Trial 2 (Figure 7). The phosphine concentration x time profiles achieved depended on where they were measured. Concentrations peaked earlier and tended to be higher in the upper parts of the grain bulk. By day 8 almost identical concentrations were achieved at all depths except at the 7 m point, and by day 13 concentrations at all depths which were almost identical. The lowest mean concentration was 471 ppm at 7 m. Loss of phosphine was estimated to be 8% per day based on analysis of the mean concentrations for days 5-21.

Field peas were fumigated for 14 days in Trial 3 (Figure 8). The phosphine concentration x time profiles varied depending on where they were measured. By day 5 there were similar concentrations at all depths except at the 7 m point which tended to be lower. The lowest mean concentration was 362 ppm at 7 m. Loss of phosphine was estimated to be 18% per day based on analysis of the mean concentrations for days 5-14.

Wheat was fumigated for 19 days in Trial 4 (Figure 9). The phosphine concentration x time profiles achieved depended on where they were measured. Concentrations peaked earlier and tended to be higher in the upper parts of the grain bulk. Maximum mixing had occurred by day 9 with similar concentrations at all depths. The lowest mean concentration was 456 ppm measured at 7 m. Loss of phosphine was estimated to be 6% per day based on analysis of the mean concentrations for days 4-19.

Wheat was fumigated for 12 days in Trial 5 (Figure 10). The phosphine concentration x time profiles achieved depended on where they were measured. Concentrations were low and variable at 7 m because the base plate was knocked off accidentally for 24 hours before being refitted. Concentrations measured at other points tended to be similar probably because the silo was only 30% full. Loss of phosphine could not be estimated from the mean because means for only 4 days were available for analysis. The lowest mean concentration was 72 ppm measured at 7 m and the fumigation must be regarded as a failure.

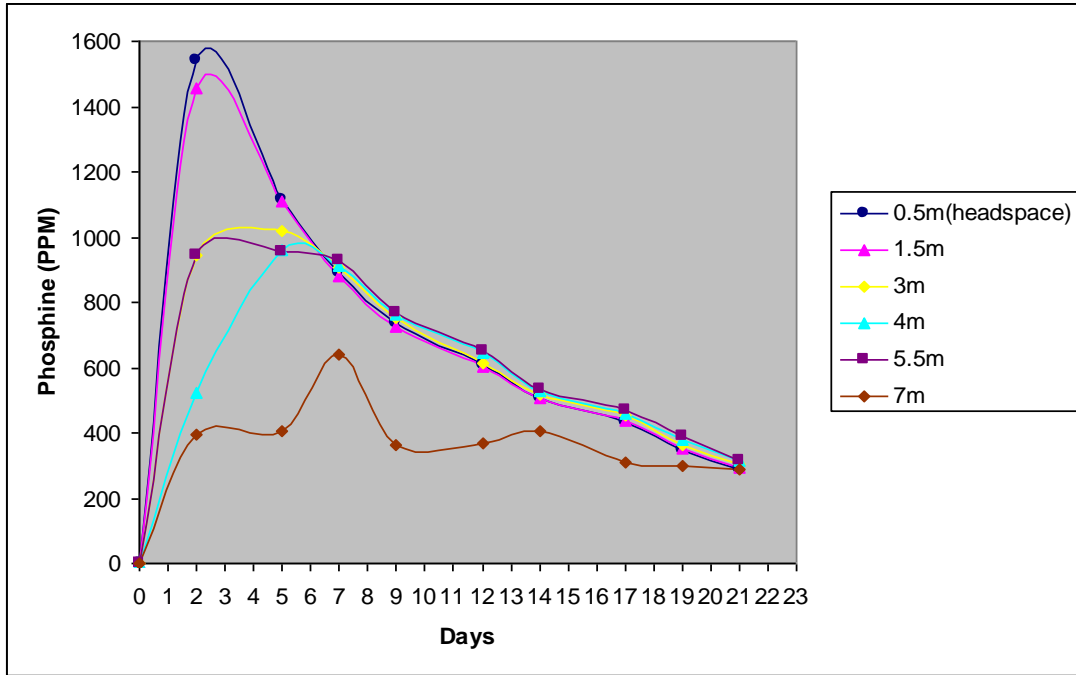


Figure 6. Phosphine concentrations measured at different depths in a sealed silo containing wheat (9.8% mc). Grain temperature was 21.0°C and mean minimum and maximum air temperatures were 17.0 and 31.6°C respectively (mean 24.3°C). The silo pressure halving time was 210 seconds.

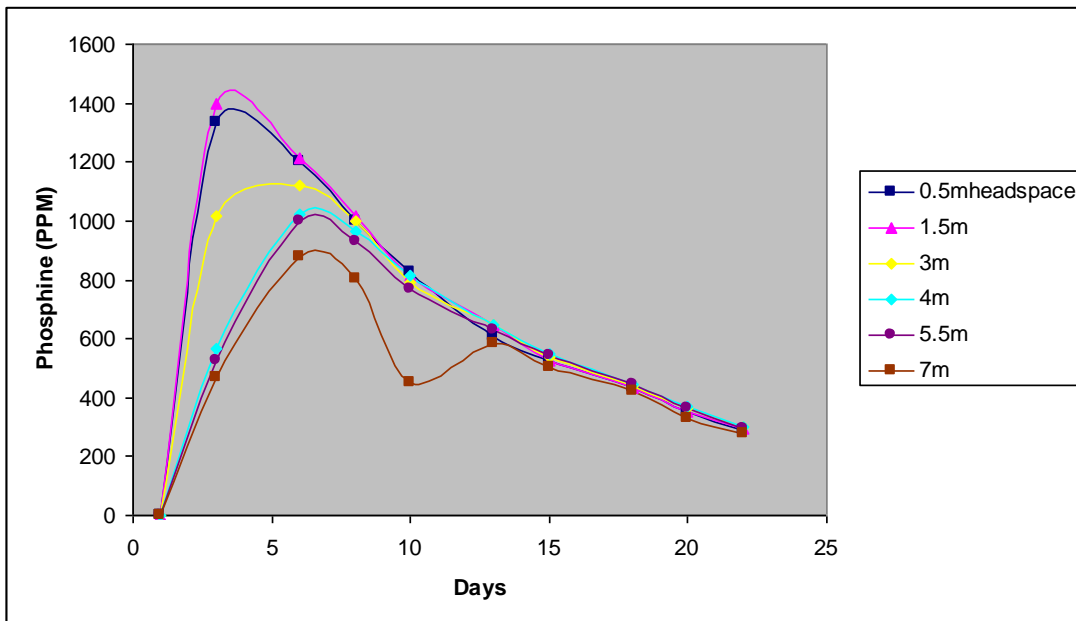


Figure 7. Phosphine concentrations measured at different depths in a sealed silo containing wheat (9.0% mc). Grain temperature was 19°C and mean minimum and maximum air temperatures were 17.0 and 31.6°C respectively (mean 24.3°C). The silo pressure halving time was 250 seconds.

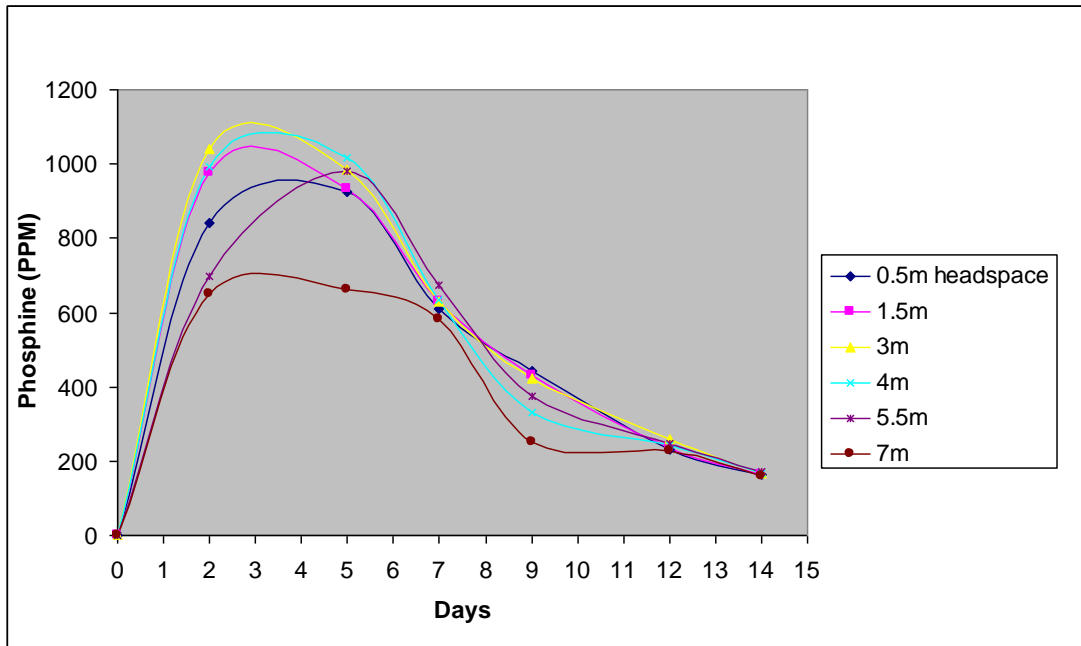


Figure 8. Phosphine concentrations measured at different depths in a sealed silo containing field peas (8.5% mc). Grain temperature was 19.8°C and mean minimum and maximum air temperatures were 17.0 and 31.6°C respectively (mean 24.3°C). The silo pressure halving time was 60 seconds.

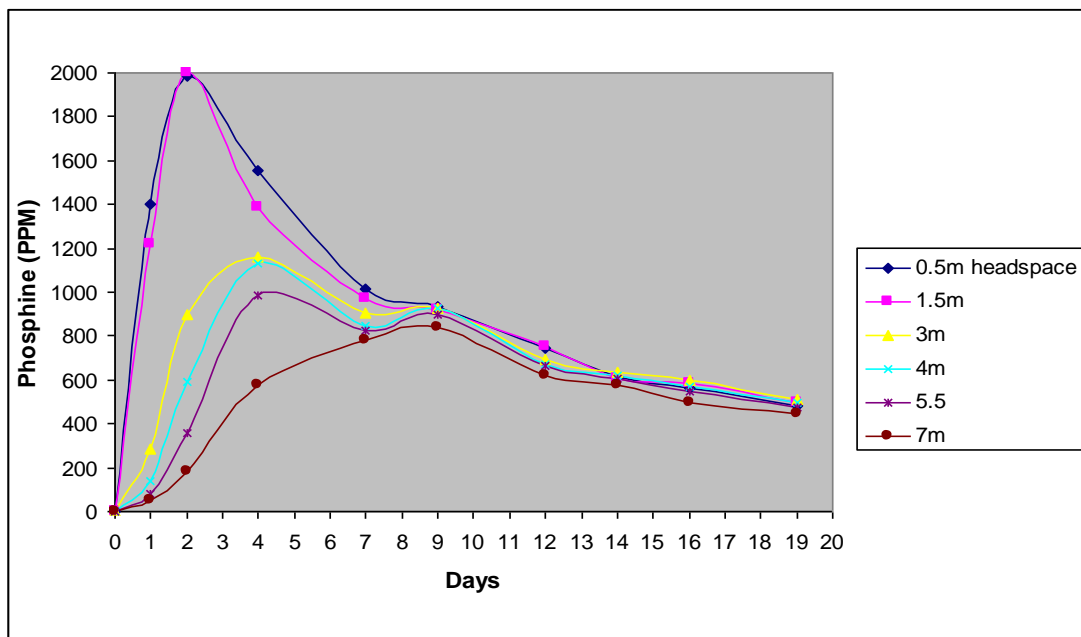


Figure 9. Phosphine concentrations measured at different depths in a sealed silo containing wheat (9.7% mc). Grain temperature was 20.1°C and mean minimum and maximum air temperatures were 13.1 and 27.7°C respectively (mean 18.7°C). The silo pressure halving time was 210 seconds.

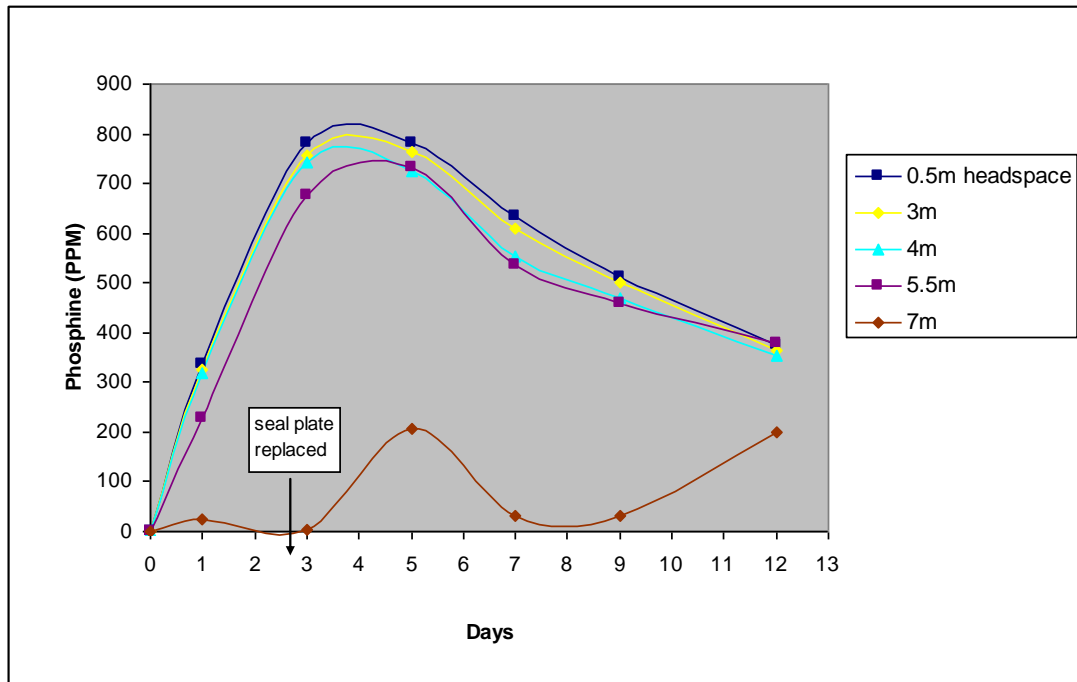


Figure 10. Phosphine concentrations measured at different depths in a sealed silo containing wheat (11.0% mc). Grain temperature was 21.4°C and mean minimum and maximum air temperatures were 9.7 and 23.3°C respectively (mean 16.5°C). The silo pressure halving time was 120 seconds.

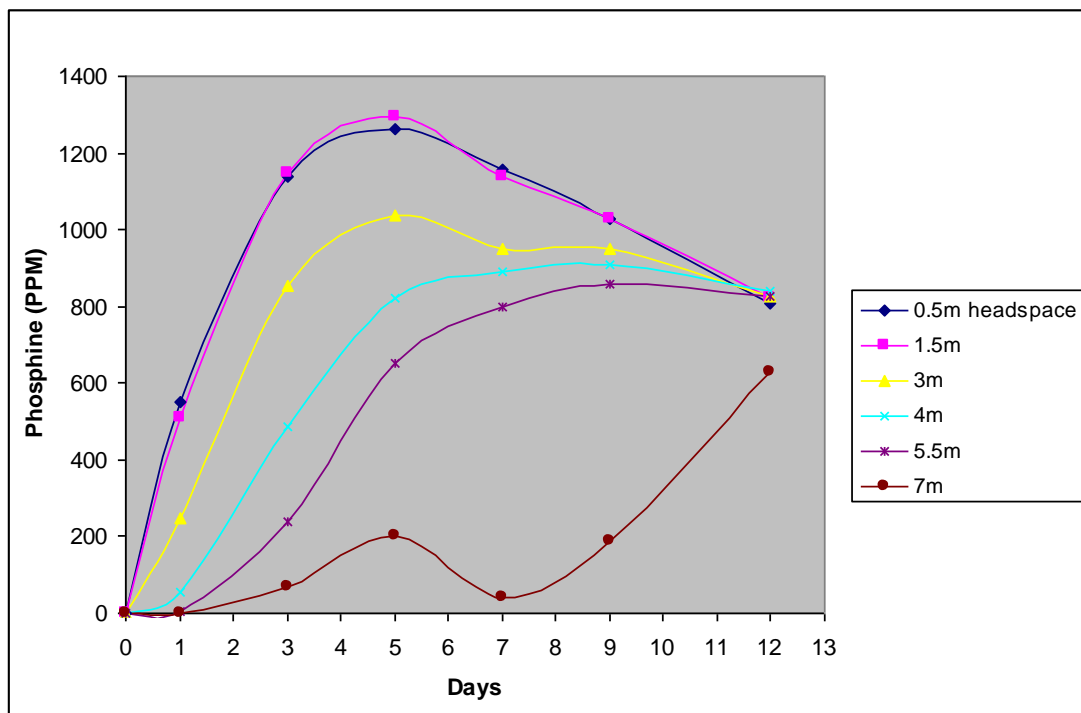


Figure 11. Phosphine concentrations measured at different depths in a sealed silo containing wheat (9.7% mc). Grain temperature was 19.5°C and mean minimum and maximum air temperatures were 9.7 and 23.3°C respectively (mean 16.5°C). The silo pressure halving time was 180 seconds.

Wheat was fumigated for 12 days in Trial 6 (Figure 11). The phosphine concentration x time profiles achieved depended on where they were measured. Concentrations were lower and variable at 7 m because of a fault in the seal plate. Concentrations peaked earlier and tended to be higher in the upper parts of the grain bulk. Maximum mixing did not occur until day 12. The lowest mean concentration was 161 ppm measured at 7 m. Loss of phosphine could not be estimated from the mean because means for only 4 days were available for analysis.

Barley was fumigated for 10 days in Trial 7 (Figure 12). The phosphine concentration x time profiles achieved depended on where they were measured. Concentrations were lower and variable at 7 m with a mean concentration of 355 ppm. Concentrations peaked earlier and tended to be higher in the upper parts of the grain bulk. Maximum mixing occurred by day 6 with similar concentrations measured at all points except at 7 m. Loss of phosphine could not be estimated because means for only 4 days were available for analysis.

Barley was fumigated for 17 days in Trial 8 (Figure 13). The phosphine concentration x time profiles achieved depended on where they were measured. Concentrations peaked earlier and tended to be higher in the upper parts of the grain bulk. Concentrations tended to be lower and variable at 7 m with a mean concentration was 321 ppm. Maximum mixing had occurred by day 5 with similar concentrations at all depths except at 7 m. Loss of phosphine was estimated to be 10% per day based on analysis of the mean concentrations for days 3-17.

Barley was fumigated for 17 days in Trial 9 (Figure 14). The phosphine concentration x time profiles achieved depended on where they were measured. Concentrations peaked earlier and tended to be higher in the upper parts of the grain bulk. Concentrations tended to be lower and variable at 7 m with a mean concentration of 289 ppm. Maximum mixing had occurred by day 7 with similar concentrations at all depths except at 7 m. Loss of phosphine was estimated to be 7% per day based on analysis of the mean concentrations for days 5-17.

Barley was fumigated for 17 days in Trial 10 (Figure 15). The phosphine concentration x time profiles achieved depended on where they were measured. Concentrations peaked earlier and tended to be higher in the upper parts of the grain bulk. Maximum mixing had occurred by day 7 with similar concentrations at all depths. The lowest mean concentration was 422 ppm at 7 m. Loss of phosphine was estimated to be 12% per day based on analysis of the mean concentrations for days 5-17.

The grain temperature in most of the Western Australian trials was about 20°C. To test the effects of exposure to grain at this temperature alone, two open ended fine mesh cages containing wheat and 50 *R. dominica* adults were speared 1.5 m into the top of an aerated silo. After 15 weeks at a mean temperature of 20°C the cages were withdrawn and mean mortality was 29.0% (SD = 4.2). The wheat was incubated for 5 wk at 25°C and a mean of 8.5 (SD = 2.0) adult progeny were recovered. Thus 15 weeks at 20°C did not kill all *Rhyzopertha dominica* adults or prevent reproduction. This sort of grain temperature would be considered typical of what can be achieved in the grain belt of central Western Australian during the seed storage period from December to March. Consequently, phosphine fumigation is needed to ensure freedom from insects.

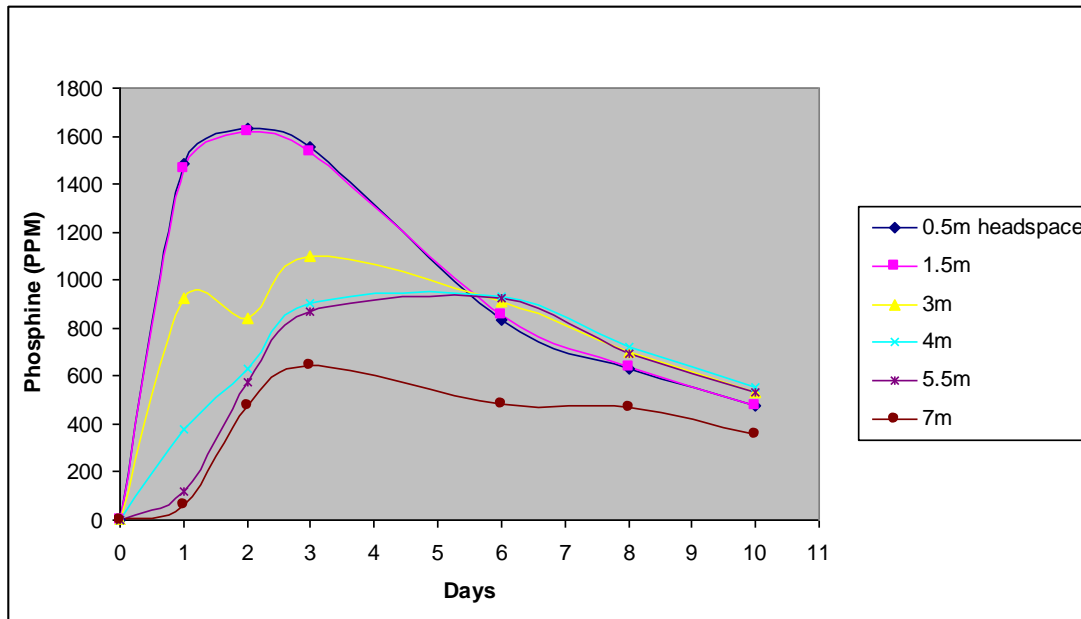


Figure 12. Phosphine concentrations measured at different depths in a sealed silo containing barley (9.4% mc). Grain temperature was 19.0°C and mean minimum and maximum air temperatures were 17.4 and 37.1°C respectively (mean 27.3°C). The silo pressure halving time was 80 seconds.

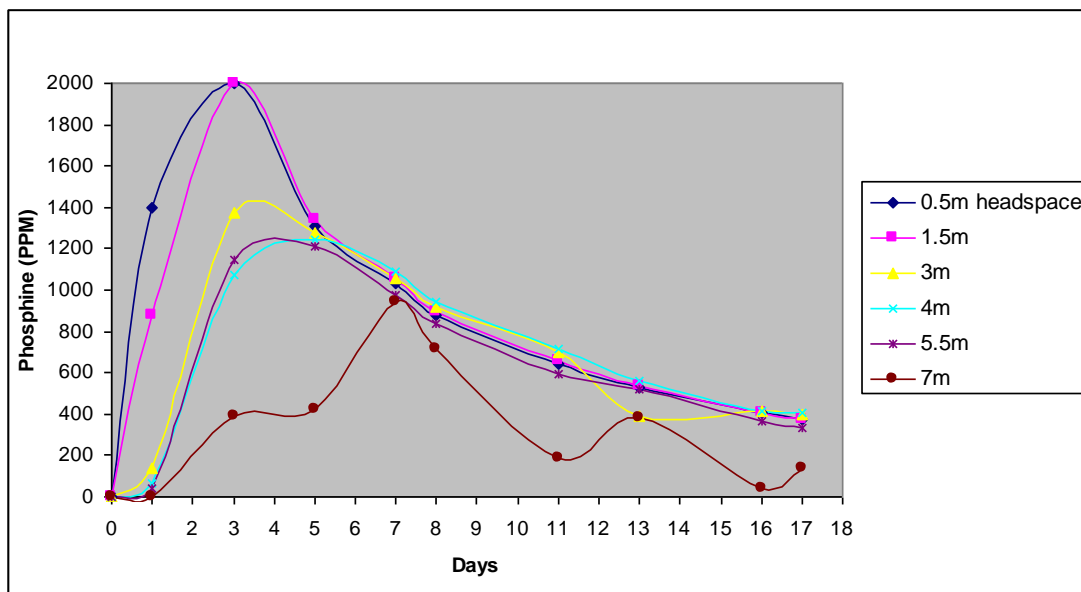


Figure 13. Phosphine concentrations measured at different depths in a sealed silo containing barley (10.0% mc). Grain temperature was 24.5°C and mean minimum and maximum air temperatures were 17.5 and 35°C respectively (mean 26.3°C). The silo pressure halving time was 90 seconds.

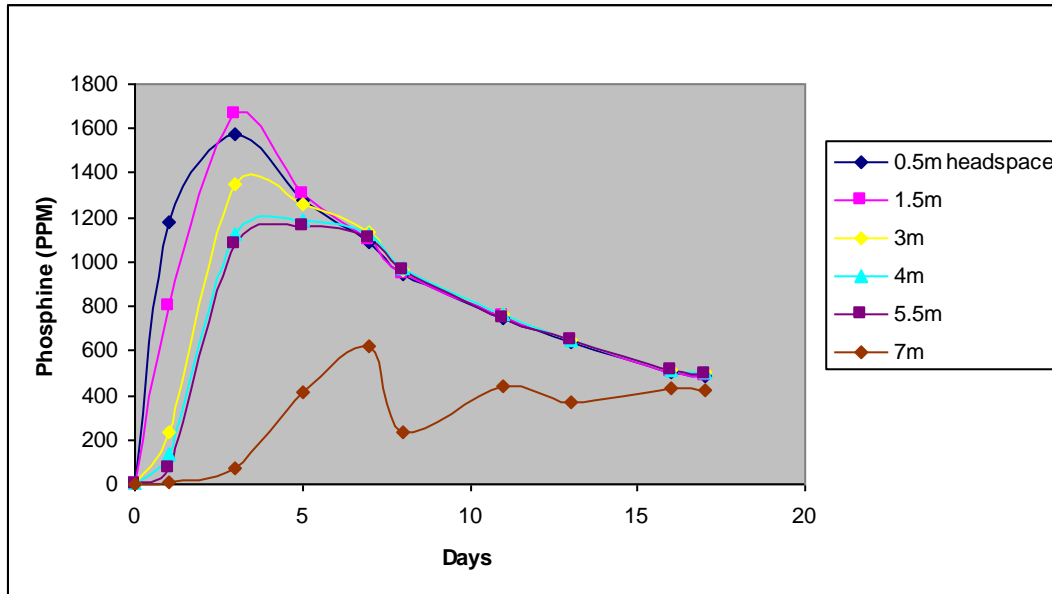


Figure 14. Phosphine concentrations measured at different depths in a sealed silo containing barley (8.7% mc). Grain temperature was 21.8°C and mean minimum and maximum air temperatures were 17.5 and 35°C respectively (mean 26.3°C). The silo pressure halving time was 60 seconds.

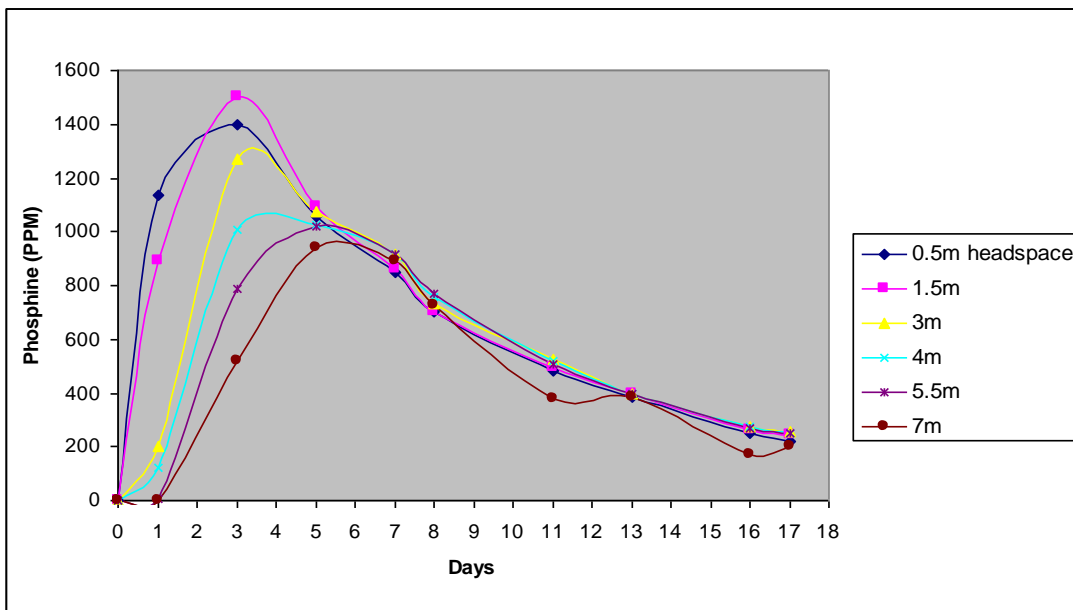


Figure 15. Phosphine concentrations measured at different depths in a sealed silo containing barley (10.0% mc). Grain temperature was 22.7°C and mean minimum and maximum air temperatures were 17.5 and 35.0°C respectively (mean 26.3°C). The silo pressure halving time was 60 seconds.

Although aluminium phosphide was the source of phosphine in these trials, the registered label for ECO₂FUME® and VAPORPHOS® can be used to estimate whether these fumigations would have controlled resistant insects. The label provides the minimum concentrations and exposure periods required to control resistant grain insects using phosphine. At 15-19°C, for example, exposure to 215 ppm for 14 days should control all major pests of cereals. At 15-20°C, 21 days at 70 ppm is required to control the pea weevil, *Bruchus pisorum*, in field peas. Eight of the 10 fumigation trials would have been successful using these criteria, although because concentrations were not constant they normally exceeded 215 ppm. There was a problem with the base plate in Trials 5 and 6 because concentrations measured at 7 m were low and variable, even though concentrations usually exceeded 215 ppm. This demonstrates the need for silo pressure testing and checking for leaks of silos prior to fumigation.

1.2.3.3 Trials in New South Wales

Seven trials were completed in New South Wales in 58 m³ silos which were 6.3 m high and 3.6 m wide and owned by the NSW DPI. There were three trials on oats, two on wheat, one on barley and one on triticale. The dose of phosphine applied was 1.72 g m⁻³ of silo volume. Phosphine was measured at three points along the central vertical axis i.e. 1, 3 and 5 m. Grain temperature was measured at 4 m and ambient temperature was monitored with an electronic weather station nearby. In one of the wheat trials, cages of mixed-age populations of two pest species were inserted into the grain bulk before being fumigated and the results were compared with those for cages inserted into an unfumigated barley bulk used as a control.

Oats were fumigated in Trial 1 for 13 days (Figure 16). The phosphine concentration x time profiles at 3 and 5 m were similar and those measured in the headspace were slightly higher (Figure 1). Concentrations peaked at 12 days indicating slow release of phosphine from the aluminium phosphide tablets, possibly because of the low moisture content. The lowest mean concentration achieved was 511 ppm at 5 m.

Triticale was fumigated in Trial 2 for 20 days (Figure 17). The phosphine concentration x time profiles at 3 and 5 m were similar and those measured in the headspace were slightly higher. Loss of phosphine was estimated to be 2% per day based on analysis of the mean concentrations for days 14-20. The lowest mean concentration achieved was 741 ppm at 3 m.

Wheat was fumigated for 12 days in Trial 3 (Figure 18). The phosphine concentration x time profiles at 1 and 3 m were similar and those measured at 5 m were slightly lower. Concentrations were levelling off at about 12 days indicating that the aluminium phosphide tablets were still releasing phosphine until about that time. The similarity between the 1 and 3 m measurements could be explained by the silo being only 40% full and the 3 m point being close to the grain surface. The lowest mean concentration achieved was 490 ppm at 5 m.

Oats were fumigated for 19 days in Trials 4 and 5 carried out at the same time under almost identical conditions (Figures 19 and 20). The phosphine concentration x time profiles were similar in the two trials and concentrations measured at 1, 3 and 5 m were very similar. The silos were only 40 and 30% full which probably explains the similar measurements at the three depths. Loss of phosphine was estimated to be 8-9% per day based on analysis of the

mean concentrations for days 14-19 in each trial. Mean concentrations during the trials were about 440 and 400 ppm in Trials 4 and 5 respectively.

The wheat fumigated previously in Trial 3 was refumigated for 15 days in Trial 6 after 2 days of passive ventilation (Figure 21). The phosphine concentration x time profiles were similar at 1 and 3 m and slightly higher than at 5 m. Concentrations were very stable at all depths during much of the trial indicating negligible loss from sorption or leakage. The similarity between the 1 and 3 m measurements could be explained by the silo being only 40% full and the 3 m point being close to the grain surface. The lowest mean concentration achieved was 621 ppm at 5 m. Unlike the first fumigation of this bulk in which concentrations continued to rise, concentrations in this trial levelled off after 5 days. It is possible that 2 days passive ventilation after the first fumigation was not long enough to ensure complete desorption of phosphine. Low levels of phosphine were measured at the end of ventilation of the first fumigation (3 ppm at 1.0m and 3.0 m and 23 ppm at 5.0 m). Alternatively, the wheat was older when it was refumigated and therefore less sorptive.

Barley was fumigated in Trial 7 for 15 days (Figure 22). Phosphine concentrations tended to increase throughout the trial. The phosphine concentration x time profiles at 1 and 3 m were similar and those measured at 5 m were slightly lower. The lowest mean concentration achieved was 534 ppm at 5 m.

Mixed-age populations of strong phosphine-resistant *Rhyzopertha dominica* and *Tribolium confusum* were placed, within cages, in the grain during Trials 1 and 2. Each cage was probed into grain to a depth of 1-2 m. On completion of the fumigation, insect cages were removed and the grain within checked for live adults. All adults (live and dead) were removed and the remainder of the grain placed in jars, with some culture medium, were stored for 10 wk at 25°C, 65% rh. At this time the grain was sieved and checked for live adult insects to determine whether eggs, larvae or pupae survived the fumigation. No life stage (egg, larvae, pupae or adult) of either species survived the 13 day and 20 day fumigations at grain temperatures around 11.5°C. The experiment was repeated in Trial 3, this time with strong resistant *R. dominica* and susceptible *Sitophilus oryzae* mixed-age populations. To ensure that insects did not die from cold alone, cages of insects were placed into an unfumigated control silo containing barley at a similar temperature. While insects survived the cold, no survivors of any life stage were found after the 12 day fumigation (Table 14).

Grain temperatures in the seven trials completed in New South Wales were in the range 10-12°C. Grain temperatures in the southern region of Australia regularly fall to around 10°C during winter. Active insects were observed in and around silos during the trials despite the low prevailing ambient temperatures. In addition, the increased uptake of aeration may result in grain temperatures below 15°C in other parts of Australia. Fumigation at grain temperatures of less than 15°C is not recommended but in practice would be occurring because growers are unlikely to measure grain temperature before fumigating. The New South Wales trials show that phosphine fumigation of grain at 10-12°C can result in high phosphine concentrations and that control of infestations of resistant insects is possible. The slow rise in phosphine concentration observed in some of the trials probably reflects the low grain moisture content (e.g. Figure 16).

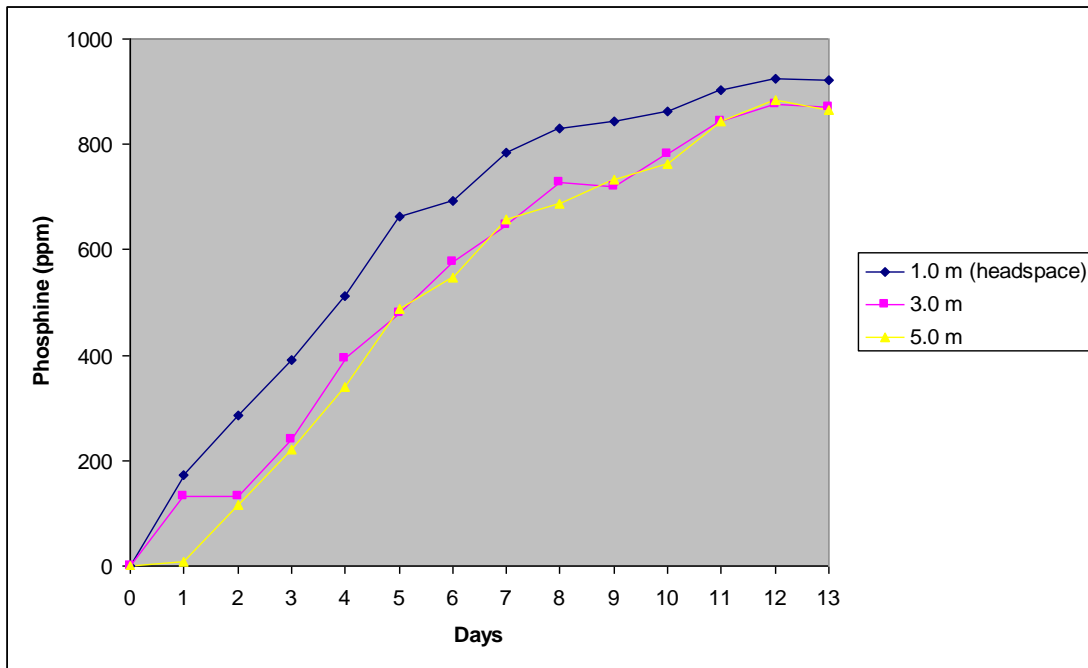


Figure 16. Phosphine concentrations measured at different depths in a sealed silo containing oats (7.2% mc). Grain temperature was 11.8°C and mean minimum and maximum temperatures were 0.0°C and 16.8°C (mean 8.4°C). The silo was 80% full and the silo pressure halving time was 155 seconds.

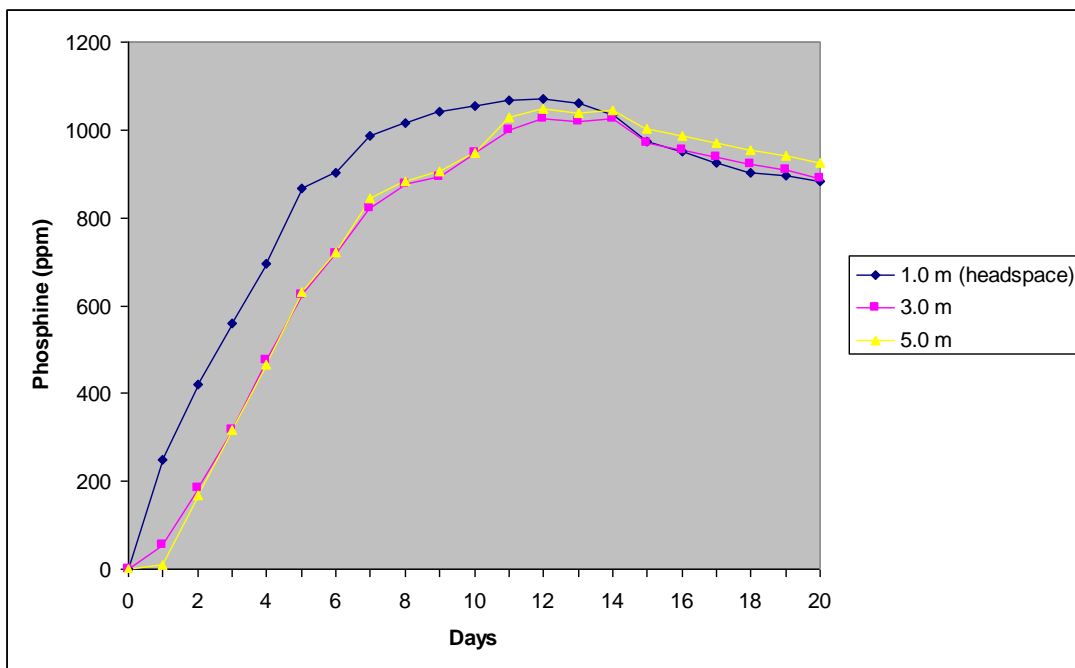


Figure 17. Phosphine concentrations measured at different depths in a sealed silo containing triticale (9.8% mc). Grain temperature was 11.2°C and mean minimum and maximum temperatures were 1.2 and 17.1°C (mean 9.2°C). The silo was 85% full and the silo pressure halving time was 185 seconds.

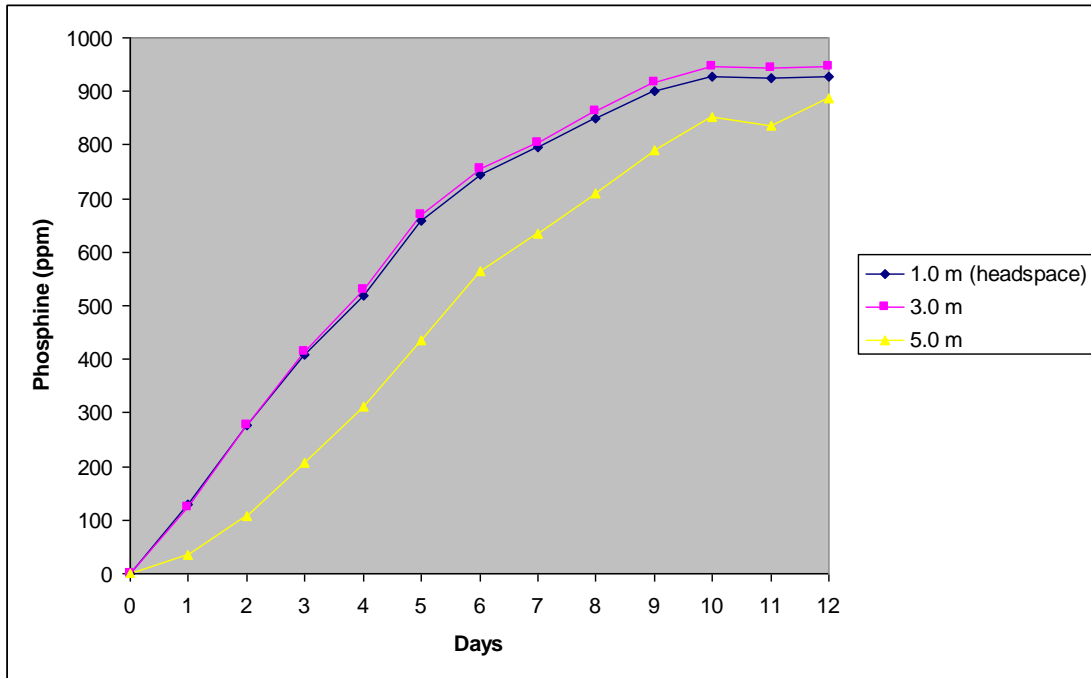


Figure 18. Phosphine concentrations measured at different locations in a sealed silo containing wheat (11.4% mc). Grain temperature was 10.3°C and mean minimum and maximum temperatures were 3.9 and 17.2°C respectively (mean 10.6°C). The silo was 40% full and the silo pressure halving time was 180 seconds.

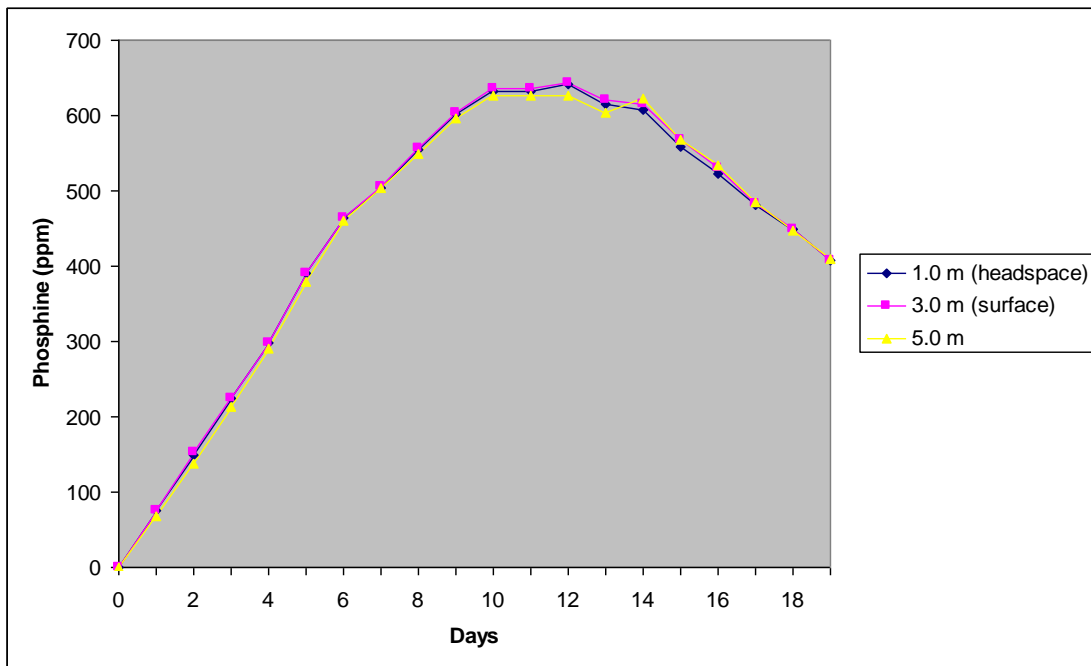


Figure 19. Phosphine concentrations measured at different locations in a sealed silo containing oats (8.2% mc). Grain temperature was 11.3°C and mean minimum and maximum temperatures were 3.9 and 18.5°C (mean 11.2°C). The silo was 30% full and the silo pressure halving time was 160 seconds.

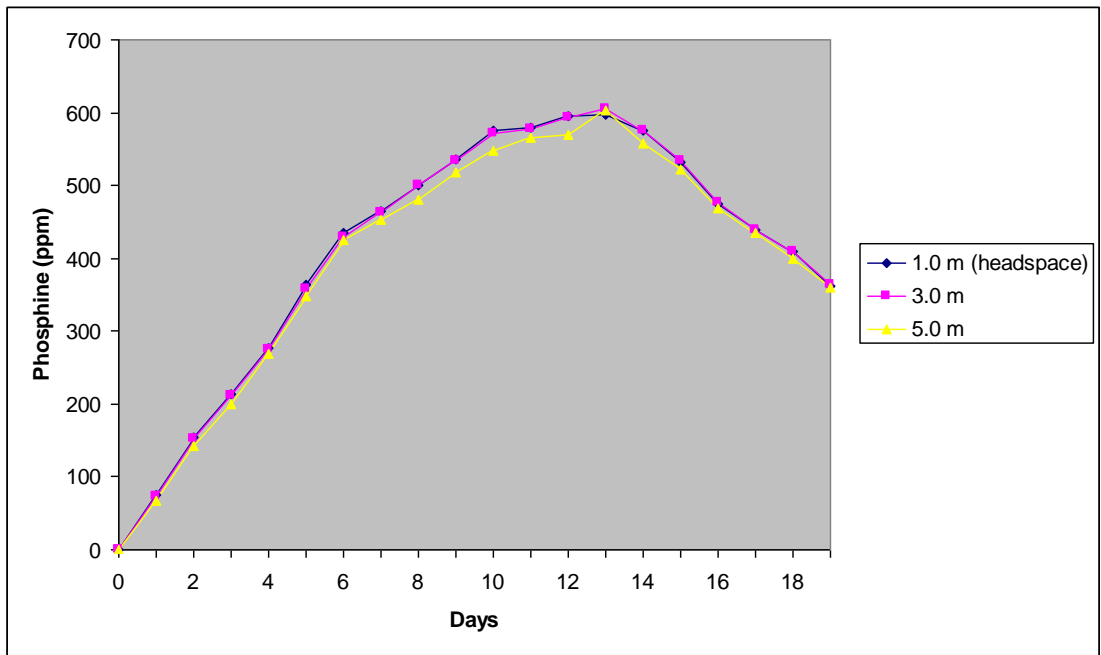


Figure 20. Phosphine concentrations measured at different locations in a sealed silo containing oats (9.1% mc). Grain temperature was 11.3°C and mean minimum and maximum temperatures were 3.9 and 18.5°C (mean 11.2°C). The silo was 40% full and the silo pressure halving time was 150 seconds.

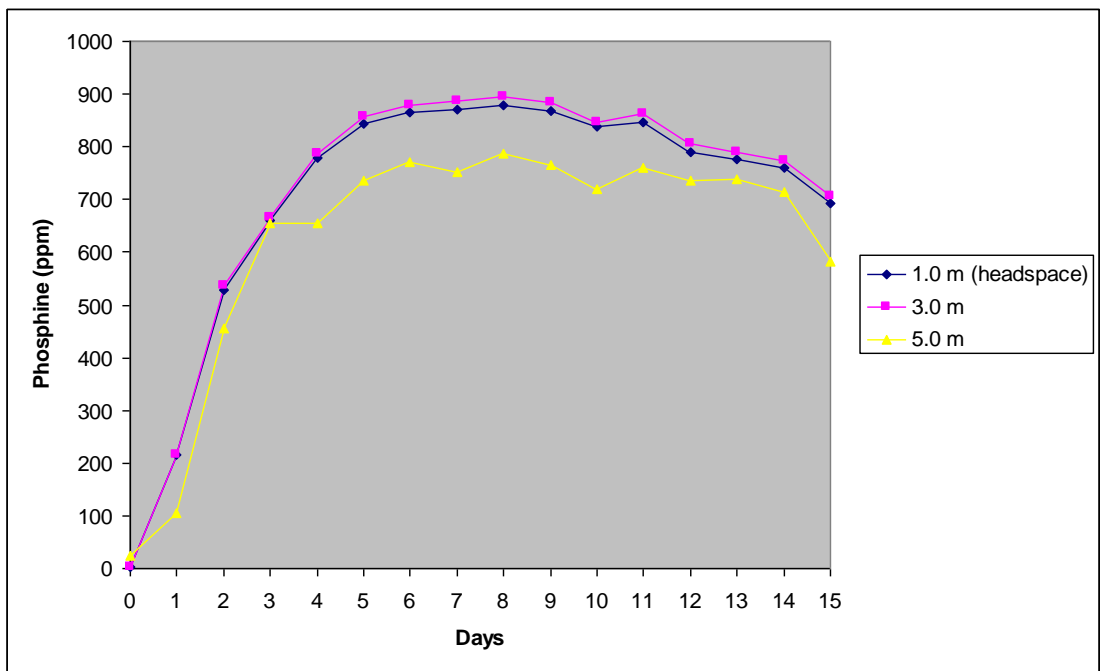


Figure 21. Phosphine concentrations measured at different locations in a sealed silo containing wheat (11.4% mc). This wheat had been fumigated previously in Trial 3 (Figure 18). Grain temperature was 11.1°C and mean minimum and maximum temperatures were 3.6 and 18.7°C (mean 11.2°C). The silo was 40% full and the silo pressure halving time was 180 seconds.

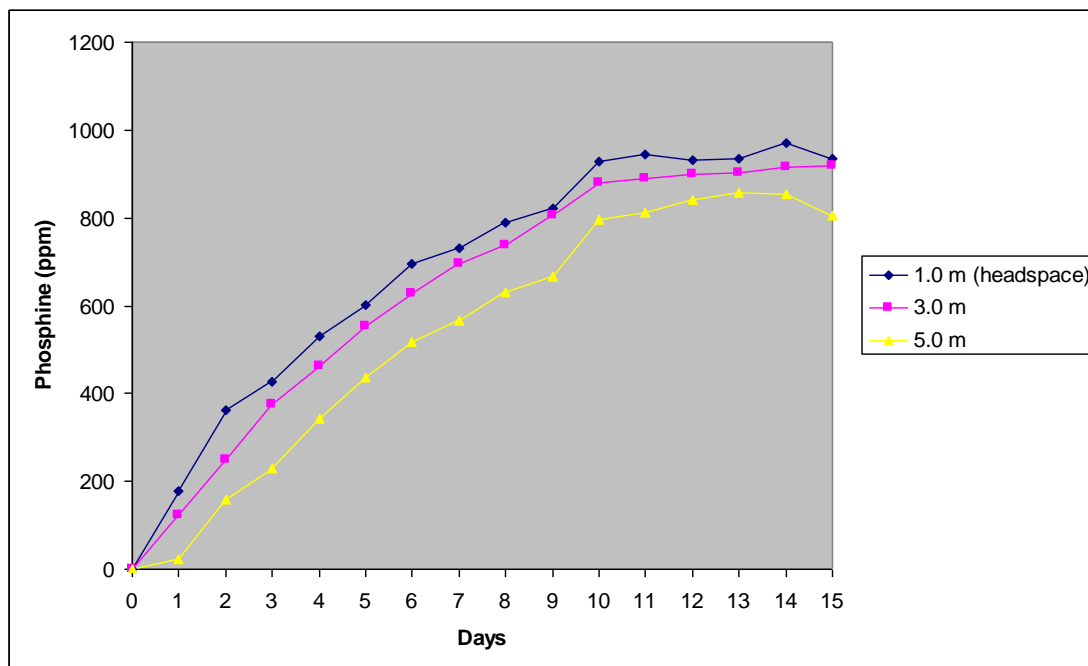


Figure 22. Phosphine concentrations measured at different locations in a sealed silo containing barley (11.4% mc). Grain temperature was 11.9°C and mean minimum and maximum temperatures were 3.6 and 18.7°C (mean 11.2°C). The silo was 70% full and the silo pressure halving time was 180 seconds.

Table 14: Initial and post-incubation counts (mean \pm SD) of live and dead adult *R. dominica* and *S. oryzae* after Trial 3 (12 days duration, grain temperature 10.5 °C).

Species	Treatment	Initial		10 wk post-fumigation	
		Live	Dead	Live	Dead
<i>R. dominica</i>	Control	64.5 \pm 14.8	6.0 \pm 0.0	60.0 \pm 17.7	0.5 \pm 0.7
	Fumigated	0.0 \pm 0.0	61.0 \pm 7.1	0.0 \pm 0.0	7.5 \pm 2.1
<i>S. oryzae</i>	Control	4.0 \pm 5.7	81.5 \pm 26.2	38.5 \pm 13.4	45.0 \pm 4.2
	Fumigated	0.0 \pm 0.0	81.0 \pm 2.8	0.0 \pm 0.0	1.0 \pm 1.4

1.2.4 Summary of key findings

Laboratory and field experiments were conducted to investigate different aspects of phosphine fumigation of cool grain. Laboratory experiments examined phosphine efficacy against resistant pests at 15°C, and effects of temperature and other factors on phosphine sorption in grain. Field trials were conducted in three states to collect data under practical conditions. The key findings are summarised below.

Laboratory experiments on phosphine efficacy:

- the relative importance of resistant strains from different species depends on temperature
- weak resistant *S. oryzae* populations were harder to control at 15°C than strong resistant *R. dominica* populations, despite published data showing that their responses to phosphine are similar at 25°C.
- complete control of strong resistant *R. dominica* was achieved in less than 14 days at either 240 or 720 ppm.
- the trends shown for *S. oryzae* show that 99-100% reduction would be expected by 14 days under the same conditions.

Laboratory experiments on phosphine sorption:

- sorption causes phosphine concentration to fall approximately exponentially (except for an initial phase lasting 1-2 h) implying that there is a constant percentage loss per day.
- sorghum is more sorptive than wheat so the margin for error is smaller for sorghum
- the rate of sorption was lower at lower temperatures resulting in higher concentrations for longer
- older grain tends to be less sorptive so delaying fumigation may result in higher concentrations for longer
- grain tends to be less sorptive if fumigated again (after adequate ventilation) but this is this could be explained by time in storage rather than refumigation per se.

Field trials in sealable farm silos:

- aluminium phosphide formulations were capable of releasing phosphine at low grain and ambient temperatures
- phosphine concentrations were often lower deeper in grain mass



- phosphine concentrations tended to be lower on northern side of the silo and higher on the southern side.
- control of resistant insects is provided adequate concentrations are achieved for long enough
- mortality of insects was not because of exposure to low temperatures alone

1.2.5 Cited references

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1.3. Implications for stakeholders

Successful fumigation of resistant insects requires sufficient phosphine concentrations for long enough to control all life stages of the insects. Lower temperatures maintain grain quality and reduce insect population growth, but phosphine is generally less effective at lower temperatures.

The relative importance of phosphine resistant strains of key pests changes with temperature. The most resistant Australian strains of two pests are known to respond similarly to phosphine but the project showed that one species became much harder to control in cool grain.

Sorption will be the major cause of loss of phosphine in a well-sealed silo. Rate of sorption was lower at lower temperatures, meaning that higher concentrations will be achieved for longer. Older grain tended to be less sorptive, so delaying fumigation may result in higher

concentrations for longer. Sorghum was more sorptive than wheat so the margin for error is smaller for this grain.

In farm silos phosphine gas was liberated from the aluminium phosphide formulations used despite the low grain and ambient temperatures (e.g. 10°C).

The results for the silo trials varied but three general observations were made. Lower concentrations tended to be measured deeper in grain mass, and concentrations measured higher in the grain mass tended to peak earlier. Current recommendations are for the aluminium phosphide formulation (e.g. tablets, blanket or belts) to be placed in the headspace to avoid potential contamination of the grain with unreacted aluminium phosphide. Previously, admixture while loading of silos with grain was common. Lower concentrations tended to be measured on the northern side, which tends to receive more direct sunlight.

The silo trials showed that control of resistant insect populations is possible subject to good gas-tightness and adequate exposure periods. Exposure to cool temperature alone was not the cause of insect mortality.

1.4. Recommendations

Phosphine fumigation of cool grain (i.e. 20°C or less) to control resistant insect populations is possible subject to good gas-tightness and adequate exposure periods.

Growers and others planning to fumigate cool grain in sealable silos should aim for the current silo pressure test standard of a 3 minute halving time for a full silo or a 5 minute halving time for a partially full silo.

Delaying fumigation may result in more effective fumigation because the sorptive capacity of grain decreases with age.

Growers and others planning to fumigate grain that has been aerated or has been harvested and stored in the cooler months of the year, measure grain temperature before fumigation to ensure that an appropriate exposure period is used.

The use of forced or passive recirculation methods (e.g. the thermosiphon), should be investigated to promote rapid and even distribution of phosphine from the headspace throughout the grain bulk.

Abbreviations/glossary

ABBREVIATION	FULL TITLE
CRCNPB	Cooperative Research Centre for National Plant Biosecurity
EPP	Emergency plant pest
ppm	Parts per million

2. Plain English website summary

CRC project no:	CRC50060
Project title:	Phosphine fumigation of cool grain
Project leader:	Dr Greg Daglish (QDPI&F)
Project team:	Dr Joanne Holloway (NSWDPI), Mr Chris Newman (DAFWA), Mr Philip Burrill (QDPI&F), Ms Hervoika Pavic (QDPI&F), Ms Kathryn Ellis (NSWDPI)
Research outcomes:	<p>This project assessed the efficacy of phosphine fumigation against resistant insects in cool grain, i.e. aerated grain or grain that has been harvested and stored in the cooler months of the year, and developed recommendations for Australia's grain storage industry. Grain temperatures of 20°C or less are considered cool. The project will contribute to keeping Australia's grain export industry free from insect strains resistant to phosphine.</p> <p>Increasing applications of phosphine to stored grain in silos that leak, or grain stored under the wrong conditions, has caused problems with insect pests building up resistance. Proof is needed to show whether phosphine fumigation of cool grain is effective in controlling resistant biotypes of insect pests of stored grain.</p> <p>The project has:</p> <ul style="list-style-type: none"> • provided reports describing new data on phosphine efficacy at low temperature against resistant grain insects. • provided reports describing new data on phosphine sorption by grain. • provided reports describing new data on phosphine fumigation of cool grain in sealed farm silos. • made recommendations to farmers and others within the grain industry. <p>Recommendations on phosphine fumigation of cool grain have been reported to representatives of the grain industry and national extension network at annual meetings of National Working Party on Grain Protection (NWPGP). Results, recommendations and technology transfer have been provided to the Australian grain industry via the NWPGP and industry</p>

	forums and publications. CRC partners, bulk grain storage operators and handlers have been informed of the findings.
Research implications:	<p>Successful fumigation of resistant insects requires sufficient phosphine concentrations for long enough to control all life stages of the insects. Lower temperatures maintain grain quality and reduce insect population growth, but phosphine is generally less effective at lower temperatures.</p> <p>The relative importance of phosphine resistant strains of key pests changes with temperature. The most resistant Australian strains of two pests are known to respond similarly to phosphine but the project showed that one species became much harder to control in cool grain.</p> <p>Sorption will be the major cause of loss of phosphine in a well-sealed silo. Rate of sorption was lower at lower temperature, meaning that higher concentrations will be achieved for longer. Older grain tended to be less sorptive, so delaying fumigation may result in higher concentrations for longer. Sorghum was more sorptive than wheat so the margin for error is smaller for this grain.</p> <p>In farm silos, phosphine gas was liberated from the aluminium phosphide formulations used despite the low grain and ambient temperatures (e.g. 10°C).</p> <p>The results for the silo trials varied but three general observations were made. Lower concentrations tended to be measured deeper in grain mass. Lower concentrations tended to be measured on the northern side. Concentrations measured higher in the grain mass tended to peak earlier.</p> <p>The silo trials showed that control of resistant insect populations is possible subject to good gas-tightness and adequate exposure periods. Exposure to cool temperature alone was not the cause of insect mortality.</p> <p>Growers and others planning to fumigate cool grain in sealable silos should aim for the current silo pressure test standard of a three minute halving time for a full silo or a five minute halving time for a partially full silo.</p> <p>The use of forced or passive recirculation methods (e.g. the thermosiphon) should be investigated to promote rapid and even distribution of phosphine from the headspace throughout the grain bulk.</p>
Research publications:	Daglish, G.J. and Newman, C.R. (in press) Trials on phosphine fumigation of grain in sealed farm silos in Western Australian following aeration cooling. <i>Australian Postharvest Technical Conference, Perth, 16-18 July 2006.</i>

	<p>Daglish, G.J. and Pavic, H. Effect of phosphine dose on sorption in wheat (2008). <i>Pest Management Science</i> 64, 513-518.</p> <p>Daglish, G.J., Pavic, H., Burrill, P.R., Holloway, J.C. and Newman, C.R. (2008) Combining the benefits of cooling and phosphine fumigation to meet the biosecurity challenge posed by grain insects. <i>Proceedings of an International Conference on Controlled Atmosphere and Fumigation in Stored Products, Chengdu, China, 21-26 September 2008</i>, pp. 489-492.</p> <p>Newman, C.R., Daglish, G.J., Wallbank, B.E. and Burrill, P.R. Investigation into control of phosphine-resistant <i>Rhizopertha dominica</i> in sealed farm silos fumigated with phosphine. <i>Proceedings of an International Conference on Controlled Atmosphere and Fumigation in Stored Products, Broadbeach, Australia, 8-13 August 2004, 2007</i>, pp 275-282.</p>
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