

## EFFICACY OF PHOSPHINE FUMIGATIONS ON BAGGED MILLED RICE UNDER POLYETHYLENE SHEETING IN INDONESIA

M. BENGSTON<sup>1</sup>, M. SIDIK<sup>2</sup>, H. HALID<sup>2</sup> AND E. ALIP<sup>2</sup>

<sup>1</sup>*Queensland Department of Primary Industries,  
80 Meiers Road, Indooroopilly, Australia 4068*

<sup>2</sup>*BULOG, Jl. Jend. G. Subroto 49, Jakarta, Indonesia*

### ABSTRACT

Phosphine (PH<sub>3</sub>) fumigation is the major means of pest control in milled rice in government godowns in Indonesia. In the tropical climate, insect infestation is severe and PH<sub>3</sub> fumigation is required every 3 months. The predominant pest species are *Tribolium castaneum* (Herbst) and psocids, chiefly *Liposcelis entomophilus* (Enderlein).

To investigate the efficacy of fumigation practices, six stack fumigation trials were carried out. The standard fumigation enclosure involved a single 0.125-mm polyethylene sheet covering a stack of around 300 t. The polyethylene sheeting was sealed to the concrete floor by weighting it with fumigation chains around the perimeter of the enclosure. The nominal PH<sub>3</sub> dosage was 2 g per t plus an allowance for the walkway space (where adjoining stacks are fumigated in one enclosure) of 5% of the dosage. PH<sub>3</sub> was generated from tablets containing aluminium phosphide. Throughout the fumigation, PH<sub>3</sub> concentrations were monitored using portable electronic meters which can determine carbon monoxide separately. In a 5-d fumigation, the concentration by time products (Ct's) exceeded 150 mg h L<sup>-1</sup>. Fumigations not in accord with the protocol — involving sheeting in poor conditions, fumigation enclosures with two sheets joined only by overlap, or fumigations without chains — all produced much lower Ct's. PH<sub>3</sub> concentrations have been discussed in relation to the rate of PH<sub>3</sub> release from the aluminium phosphide, the rate of its sorption on milled rice, the rate of its permeation through the polyethylene sheet and the rate of leakage. The PH<sub>3</sub> concentrations recorded suggest that complete kill of both species is possible in this type of fumigation, but reports of *L. entomophilus* strains from Indonesia with enhanced PH<sub>3</sub> tolerance suggest that complete kill is not always achieved in practice. Meters capable of monitoring PH<sub>3</sub> concentrations in the field have now been issued to operational staff and should reduce the frequency of fumigation failure.

### INTRODUCTION

In Indonesia, buffer stocks of grain are stored by the National Logistics Agency (BULOG). The major commodity is milled rice, which is stored in bags in godowns.

The storage interval varies with seasonal conditions and generally ranges from a few months to two years. Most of the cosmopolitan pest species are present, and in the tropical environment applied pest-control measures are necessary to prevent major losses (Sidik *et al.*, 1985)

These measures are based on spraying the storage fabric and the outsides of bag stacks with insecticide, plus insecticide fogging of the storage interior and phosphine (PH<sub>3</sub>) fumigation at intervals of approximately 3 months. Under these circumstances, the major pest species are *Tribolium castaneum* (Herbst) and psocids, chiefly *Liposcelis entomophilus* (Enderlein). From the field observations, it is not clear to what extent these infestations are due to fumigation survivors and to what extent to reinfestation. A Javanese strain of *L. entomophilus* has been reported to be quite tolerant to PH<sub>3</sub> (Pike, 1994), and PH<sub>3</sub> resistance has also been reported in strains of *T. castaneum* elsewhere (but apparently not as yet from Indonesia).

Despite the importance of duration and PH<sub>3</sub> concentration during fumigation, relatively few data have been published about commercial fumigation under tropical conditions. Fumigations of milled rice in Southeast Asia at an application rate of 2 g t<sup>-1</sup> — but under unspecified conditions — gave PH<sub>3</sub> concentrations 0.2–0.4 mg L<sup>-1</sup> over 100 h (Taylor and Harris, 1994). However, the same authors also reported that in Tanzania fumigations of maize under PVC sheeting, with a careful fumigation protocol, gave concentrations of 1.0–1.8 mg L<sup>-1</sup> over 7 d. The current paper reports the concentrations of PH<sub>3</sub> achieved in fumigations of stacks of bagged milled rice under plastic sheeting within a larger godown.

## METHODS

Six stack fumigations starting 29 August 1995 were carried out simultaneously in a typical godown in the BULOG storage complex at Gede Bage near Bandung in Java. The godown was 48 × 30 m. It had a concrete floor and walls to a height of 3 m, a steel frame and galvanised iron walls to a height of 5 m and a galvanised iron roof. The optimal filling capacity was 3,500 t bagged grain in three rows of four stacks each. The milled rice, in woven polypropylene bags each containing around 50 kg, was placed on timber dunnage in stacks typically around 300 t. The dunnage, in the form of pallets, provided an 0.1-m airspace below the stack and enhanced gas distribution. The concrete floor was made of slabs approximately 1.0 × 1.5 m with some surface irregularities. The joints had been filled with bitumen to within around 10 mm of the surface, and in some areas topped off to the surface with cement.

All fumigations were enclosed in 0.125-mm polyethylene sheeting which was sealed to the concrete floor with chains laid around the perimeter of the enclosure over the edges of the sheeting. On one of the experimental stacks, the fumigation chain was not used. Either one, two, three or four adjacent stacks were fumigated in a single enclosure; when required for this experiment, two fumigation sheets were joined by simple overlap of 1 or 2 m held by paper tape.

The application rate was 2 g PH<sub>3</sub> per t plus, where adjoining stacks were fumigated in one enclosure, an allowance of 5% for the walkway space. The PH<sub>3</sub> was generated from PHOSTOXIN® tablets each of which contains sufficient aluminium phosphide to evolve 1 g PH<sub>3</sub>. Approximately 20 tablets each were placed on shallow metal trays, and the trays were placed at even intervals around the perimeter of each stack on the floor and, where more than one stack was involved, along the walkways.

Details of the fumigation enclosures are given in Table 1.

TABLE 1  
Fumigation enclosures and grain

Enclosure number	Number of stacks	Quantity of grain (t)	Grain moisture (%)	Comments on the fumigation sheets
1	1	269	<14	Single sheet, new
2	3	776	<14	Single sheet, new
3	4	1239	14–15	Two sheets, new, and overlapped 2 m to join
4	2	415	14–15	Single sheet, used, and some patches
5	4	993	14–15	Two sheets, used, and some patches; overlapped 2 m to join
6	1	55	16	Single sheet, used and many patches, floor uneven, no chains

The volume of enclosure 1 was estimated at 347 m<sup>3</sup> (5.7 × 12.7 × 4.8 m) and the volume of milled rice at 321 m<sup>3</sup> (bulk density 0.838), so the unfilled volume, including the space underneath the stack, was 26 m<sup>3</sup>. The intergranular airspace was estimated at 143 m<sup>3</sup>, so the total gas volume was 169 m<sup>3</sup>. The calculated concentration of PH<sub>3</sub> applied was 3.2 g m<sup>-3</sup>.

The other enclosures will not be considered in detail.

PH<sub>3</sub> concentrations were measured daily throughout the fumigations. Nylon tubing 3 mm in internal diameter connected the metre to the sampling point on the floor, approximately 1 m inside the perimeter of the fumigation sheeting. There were two sample points at separated locations on one side of each enclosure excepting enclosure 1, in which there were three additional sample points (one on each side of the stack in the middle of a bag 2 m from the floor and a third one at the top of the stack in a central bag. At these additional sample points, a short length of stainless steel tubing was connected to the polyethylene tubing so that the steel tubing could be pushed into the bags.

Two similar PH<sub>3</sub> meters based on electrochemical sensors (Bedfont Model EC 80 PH<sub>3</sub> Fumigation Gas Meter, Bedfont Technical Instruments Ltd, England) which respond to both carbon monoxide (CO) and PH<sub>3</sub> were used. One meter incorporated a filter which when activated removed the PH<sub>3</sub>. This enabled measurement of PH<sub>3</sub> plus CO and then CO alone. The PH<sub>3</sub> readings for both meters were adjusted by subtraction of the readings for

CO. The means of all readings by the two meters differed by only 0.7%, and the PH<sub>3</sub> levels were therefore calculated as the mean of the results from the two meters. After completion of the fumigation, one meter was check-calibrated in the laboratory using a PH<sub>3</sub>-in-air mixture standardised by gas chromatography. The readings agreed within 2% (S. Pratt, personal communication). The PH<sub>3</sub> readings in parts per million were converted to milligrams per litre by division by a factor of 725.

The ambient conditions were measured by a maximum and minimum thermometer set 2 m above the floor on the side of a stack in the centre of godown. They ranged from 22°C overnight to 32°C during the day. The grain temperature was 27°C as measured in bags from the stack perimeter.

An estimate of the rate of PH<sub>3</sub> release was obtained by adjusting the parameters in a mathematical model of the release (Banks, 1991) to obtain a visual fit of the data from the first 4 d of the fumigation in stack 1. The model incorporates an initial phase where the quantity of PH<sub>3</sub> released was described by a power function of time, a transition point termed the crossover time, and a phase where the rate of production was proportional to the quantity of undecomposed aluminium phosphide remaining.

Before the crossover time  $t_c$ ,

$$m = A_1 a t^n$$

where  $m$  = quantity of PH<sub>3</sub> generated from the start of exposure to time  $t$ ;  $A_1$  = value of  $m$  at infinite time;  $a$  = constant;  $n$  = constant, here 1.15.

After the crossover time  $t_c$ ,

$$m = A_1[1 - \exp(-k_1(t - t_0))]$$

where  $k_1$  = constant;  $t_0$  = value of  $t$  where the exponential function is zero.

The model derives the maximum rate of evolution  $A_2$  from a quadratic equation relating  $A_2$  to the absolute humidity, which for 27°C and 70% r.h. is 18.05 g m<sup>-3</sup>:

$$A_2 = 4.49 \times 10^{-3}(18.05) - 3.25 \times 10^{-5}(18.05)^2.$$

The maximum rate of evolution is reduced to the rate permitted by the airflow ( $Q$ ) in m<sup>3</sup> h<sup>-1</sup> which brings moisture to the formulation. This airflow was fitted to the data.

The time to 50% release was calculated from the equation:

$$t_{50} = 0.5n/A_2(1 - \exp(-K_2Q))$$

where  $K_2$  = constant, here 146.95.

The total rate of loss of PH<sub>3</sub> was estimated for each day of interest by calculating the PH<sub>3</sub> level each day as a percentage of the PH<sub>3</sub> level measured the day earlier.

The rate of PH<sub>3</sub> loss by permeation through the intact polyethylene sheeting was calculated assuming a first order process.

$$C_t = C_0 \exp(-k \cdot A/V \cdot t)$$

where  $C_t$  = concentration at time  $t$  ( $\text{mg L}^{-1}$ );  $C_0$  = concentration at the nominated time of commencement ( $\text{mg L}^{-1}$ );  $k$  = permeation coefficient;  $A$  = surface area ( $\text{m}^2$ );  $V$  = volume of gas in the enclosure ( $\text{m}^3$ );  $t$  = time from commencement (h).

The value of  $k$  was estimated at  $0.2 \times 10^{-3}$  for surface area in  $\text{m}^2$  and volume in  $\text{m}^3$  from published data at  $25^\circ\text{C}$  for polyethylene sheeting of different thickness (Cooper and Bengston, 1979).

## RESULTS

Data on the  $\text{PH}_3$  concentration in each enclosure each day are given in Table 2. The  $\text{PH}_3$  readings on day 1 were somewhat variable, but there were no systematic differences in readings among the positions (including the one in the centre of the top of the stack); therefore the additional readings on enclosure 1 were not continued after the first day. There was no apparent delay in the penetration of  $\text{PH}_3$  into the polypropylene bags. The time to 50%  $\text{PH}_3$  release was estimated at 32 h and the release rate ( $0.016 \text{ g h}^{-1}$  per tablet) during the time interval was relatively constant. The time to 90% release was estimated at 66.4 h; 98% had been released by day 4, and 99.7% by day 5 (Fig. 1).

TABLE 2  
Mean ( $n = 4$ ) daily  $\text{PH}_3$  concentrations in  $\text{mg L}^{-1}$

Enclosure number	Day 1	Day 2	Day 3	Day 4	Day 5	Day 6	Day 7
1	1.55	2.08	2.39	2.41	2.33	2.12	–
2	1.39	1.60	2.24	2.28	2.15	2.01	1.84
3	1.04	1.35	1.55	1.51	1.34	1.28	1.08
4	0.89	0.99	1.05	1.00	0.84	0.70	0.55
5	0.82	0.91	0.74	0.57	0.42	0.28	0.16
6	0.99	0.40	0.14	0.06	0.02	0.02	0.01

The rate of loss due to permeation through the polyethylene sheeting was calculated as 1% per day. The total rate of loss from enclosures 1 and 2 after the fourth day was estimated at 7% per day.

Concentration by time (Ct) products for each fumigation enclosure are given in Table 3. There was a marked difference between the  $\text{PH}_3$  concentrations in enclosures 1 and 2 and those in the remaining enclosures; it is significant that the Ct products in the latter did not reach  $150 \text{ mg h L}^{-1}$ . The concentration of CO was verified as zero at day 1 and reached a maximum of 64 parts per million on days 5–7.

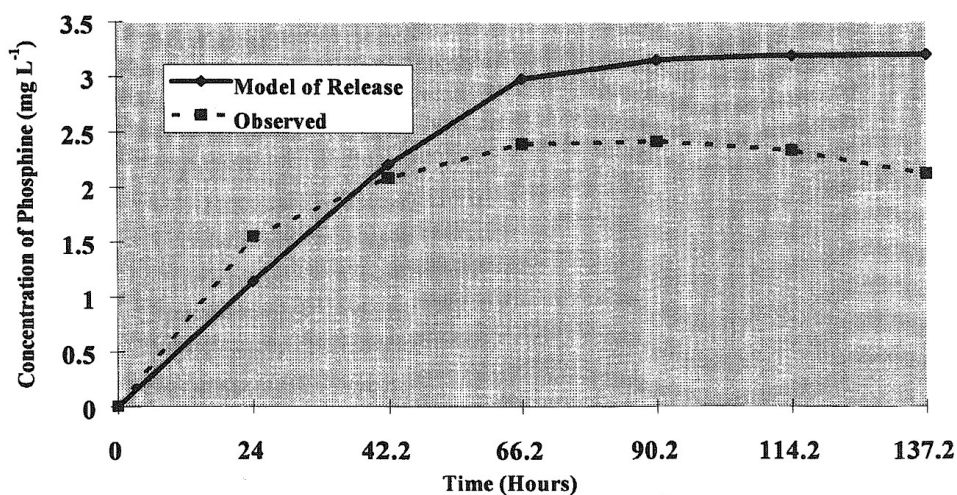


Fig. 1. Observed concentrations of  $\text{PH}_3$  in fumigations of milled rice in bags at  $27^\circ\text{C}$  and 14% moisture under polyethylene sheeting and modelled concentrations of  $\text{PH}_3$  released. The model of release (Banks, 1991) was calculated with the parameters: theoretical maximum  $\text{PH}_3$  concentration (total  $\text{PH}_3$  (g) in total gas volume ( $\text{m}^3$ ))  $3.2 \text{ g m}^{-3}$ , water vapour concentration  $18.05 \text{ g m}^{-3}$ ,  $Q = 0.002 \text{ m}^3 \text{ h}^{-1}$ ,  $t_{50} = 32.0 \text{ h}$ ,  $a = 0.00929$ ,  $t_c = 42.88 \text{ h}$ ,  $k_1 = 0.0626$ ,  $t_0 = 23.65 \text{ h}$ ,  $n = 1.15$ .

TABLE 3  
Concentration by time (Ct) products in  $\text{mg h L}^{-1}$

Enclosure number	5 d	7 d
1	229	–
2	212	310
3	145	204
4	103	137
5	76	91
6	38	39

## DISCUSSION

### Rate of release

The rate of release estimated here ( $0.016 \text{ g h}^{-1}$  per tablet) was significantly lower than the rate of  $0.034$  reported in an experiment at  $25^\circ\text{C}$  and 75% r.h. involving a single tablet in an open petri dish (Ducom and Bourges, 1993). The time to 90% release (66.4 h here) was also significantly below the 25 h reported in that experiment and the 45 h quoted to obtain the maximum release under unspecified conditions in the laboratory (Heseltine,

1973). The current data are nevertheless consistent with the release rate implied in reports of field fumigations of 100 t lots of maize in which peak  $\text{PH}_3$  concentrations sometimes occurred at day 4 (Taylor and Harris, 1994). The airflow rate of  $0.002 \text{ m}^3 \text{ h}^{-1}$  ( $25 \text{ ml min}^{-1}$ ) estimated in the current experiment was well below the rate of  $0.005 \text{ m}^3 \text{ h}^{-1}$  used to model release of  $\text{PH}_3$  from the tablet formulation (Annis and Banks, 1993). Possibly the technique of placing tablets in groups reduced the rate of release.

#### **Total rate of loss**

The rate of sorption, the rate of permeation and the rate of leakage cannot be separated on the basis of field data, but the total rate of loss (7% per day) observed — and estimated for the interval in which  $\text{PH}_3$  fumigation had virtually ceased — was at the lower end of the anticipated range. It was achieved in fumigations without a floor sheet, but obviously such a result depends on the presence of a reasonably gastight floor.

Losses due to sorption for specific commodities are influenced by many factors, including temperature, moisture, concentration, previous moisture content and previous fumigations. The rate of sorption is relatively high during the initial hours and thereafter becomes semi-logarithmic (Banks, 1993). Relatively few estimates of the rate of sorption on milled rice have been published, but an apparent first order rate constant for a full system at  $25^\circ\text{C}$  and 60% r.h. has been measured as 0.1 per day (Banks, 1990) and, when allowance is made for the filling ratio here, this suggests a possible loss of 9% per day. This estimate should be further reduced since, although no detailed fumigation history was available, it is likely that the rice had been fumigated prior to the current experiment.

A leakage rate of 5% has been suggested for a large well-sealed storage (Annis and Banks, 1993). The location of the enclosures in a godown reduced exposure to wind and sun and thus the rate of loss which might have occurred had they been in the open. There was minimal headspace and this would minimise the effect of temperature fluctuations.

#### **Carbon monoxide levels**

Stored dry grain is known to produce significant levels of CO (Whittle *et al.*, 1994), but in the 7-d fumigations measured here, the CO levels were low. These low levels would not significantly affect  $\text{PH}_3$  readings made with a meter which did not separate the response of the two gases.

#### **Effect of fumigation enclosure on gas concentration and Ct product**

The experiment provided a good illustration of the unsatisfactory results likely to occur when operating procedures may lead to a poor standard of fumigation enclosure. Fumigations involving sheets with holes, joins by simple overlap, or lack of fumigation chains all gave gas concentrations likely to lead to fumigation failure by allowing insect survival. Such fumigations in the longer term also lead to the development of resistance. In contrast, where prescribed fumigation procedures were used, the gas concentrations were above  $1 \text{ mg L}^{-1}$  from day 1 and throughout the fumigation, and the Ct products were above the  $150 \text{ mg h L}^{-1}$  which has been quoted as the fumigation target (Annis and Banks,

1993). The concentrations were above  $1.7 \text{ mg L}^{-1}$  for 5 d, and this has been estimated as the minimum necessary to kill 99% of eggs of *L. entomophilus* (Pike, 1994). This suggests that fumigations carried out according to the standard fumigation protocol are adequate to control all insects. However, the presence in Indonesia of *L. entomophilus* strains with enhanced  $\text{PH}_3$  tolerance suggest that complete kill is not always achieved in practice. This is supported by the reports of low gas concentrations in fumigations reported by others (Taylor and Harris, 1994). Meters capable of measuring  $\text{PH}_3$  concentrations in the field have now been issued to operational staff and should reduce the frequency of fumigation failure.

#### ACKNOWLEDGEMENTS

The work was financially supported by the Australian Centre for International Research (ACIAR). This support is gratefully acknowledged.

#### REFERENCES

- Annis, P.C. and Banks, H.J. (1993) A predictive model for phosphine concentration in grain storage structures. In: *Proc. Int. Conf. on Controlled Atmospheres and Fumigation in Grain Storages* (Edited by Navarro, S. and Donahaye, E.), Winnipeg, Canada, 11–13 June 1992, Caspit Press Ltd., Jerusalem, 241–260.
- Banks, H.J. (1990) Behaviour of gases in grain storages. In: *Fumigation and Controlled Atmosphere Storage of Grain. Proc. Int. Conf.* (Edited by Champ, B.R., Highley, E. and Banks, H.J.), Singapore, February 1989, 96–106.
- Banks, H.J. (1991) Influence of water and temperature on release of phosphine from aluminium phosphide-containing formulations. *J. Stored Prod. Res.* **27**, 41–56.
- Banks, H.J. (1993) Uptake and release of fumigants by grain: sorption/desorption phenomena. In: *Proc. Int. Conf. on Controlled Atmospheres and Fumigation in Grain Storages* (Edited by Navarro, S. and Donahaye, E.), Winnipeg, Canada, 11–13 June 1992, Caspit Press Ltd., Jerusalem, 241–260.
- Cooper, L.M. and Bengston, M. (1979) Tobacco beetle and its control. *Austral. Tobacco Grow. Bull.* **21**, 36–40.
- Ducom, P. and Bourges, C. (1993) Comparison of rate of release of phosphine from some commercial formulations generated under 75% relative humidity and four temperatures. In: *Proc. Int. Conf. on Controlled Atmospheres and Fumigation in Grain Storages* (Edited by Navarro, S. and Donahaye, E.J.), Winnipeg, Canada, 11–13 June 1992, Caspit Press Ltd, Jerusalem, 293–297.
- Heseltine, H.K. (1973) A guide to fumigation with phosphine in the tropics. *Trop. Stored Prod. Inf.* **24**, 25–36.
- Pike, V. (1994) Laboratory assessment of the efficacy of phosphine and methyl bromide fumigation against all life stages of *Liposcelis entomophilus* (Enderlein). *Crop Protection* **13**, 141–145.
- Sidik, M., Haryadi, H. and Pranata, R.I. (1985) Pest problems and the use of pesticides in grain storage in Indonesia. In: *Pesticides and Humid Tropical Grain Storage Systems* (Edited by Champ, B.R. and Highley, E.), ACIAR Proc. No. 14, 37–43.



- Taylor, R.W.D. and Harris, A.H. (1994) The fumigation of bag stacks with phosphine under gas-proof sheets using techniques to avoid the development of insect resistance. In: *Stored Product Protection. Proc. 6th Int. Working Conf. on Stored-Product Protection* (Edited by Highley, E., Wright, E.J., Banks, H.J. and Champ, B.R.), Canberra, Australia, 17–23 April 1994, CAB International, Wallingford, Oxon, UK, 210–213.
- Whittle, C.P., Waterford, C.J., Annis, P.C. and Banks, H.J. (1994) The production and accumulation of carbon monoxide in stored dry grain. *J. Stored Prod. Res.* **30**, 23–46.