

**Fig. 18** Extinction of insects at 3 time instances. Red color indicates zones with 99.9% insect mortality.

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## Phosphine distribution during fumigation of wheat in steel bins: extended abstract

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## Abstract

Phosphine is a widely used fumigant for controlling insects in stored grain, but fumigation effectiveness is often compromised by suboptimal distribution of the gas. Leaks in the grain bin wall and roof, foreign material in the grain, and phosphine placement contribute to regions of insufficient concentration of fumigant, resulting in insect survival and leading to phosphine-resistant insect populations. Phosphine distribution was studied during field tests in temporarily sealed bins to compare distribution from conventional probed tablets to the distribution using a closed-loop recirculation system. The results showed uneven distribution patterns and leakage over time with conventional probed tablets, which resulted in some areas in the lower half of the grain

mass receiving no phosphine and some other locations remaining below the target phosphine concentration for the entire period of fumigation. The closed-loop fumigations with the same phosphine dosage yielded much more uniform phosphine concentrations, but suffered from equal or greater phosphine leakage losses.

**Keywords:** grain storage, phosphine resistance, stored product insects, closed-loop fumigation.

## Introduction

The fumigant phosphine is extensively used for stored grain insect control and is considered one of the most effective insect control measures when properly applied (Philips et al., 2012). With this widespread use and high expectations, the effectiveness of phosphine fumigations is a fundamental concern for all users; however, there is little or no control of where gas may go during conventional fumigation. Improper application or leakage from the storage structure can result in insufficiently treated areas in the bin that will harbor surviving insects and likely select for resistant insects in the survivors. Thus, ineffective fumigations increase grain losses and contribute to the development of pesticide resistance in stored grain insects.

Conventional phosphine fumigation methods include probe and tarp, automatic dispenser, and gravity fumigation (Kenkel et al., 1993; Noyes et al., 1995). Phosphine is usually applied to grain as aluminum or magnesium phosphide in pellet or tablet form. The pellets or tablets react with water vapor in the air to produce phosphine gas. In gravity fumigation diffusion is used to distribute phosphine gas throughout the grain mass. There is little or no control of where gas may go during conventional fumigations. Each of these conventional methods offers increased risk of exposure during insertion of fumigant into the grain and the distribution of phosphine is often suboptimal. Leaks in the grain storage bin and foreign material in the grain can lead to regions of insufficient concentration of fumigant. Phosphine is also available in gaseous form mixed with carbon dioxide which can be directly injected into a grain storage bin. In the probe and tarp method, Noyes et al. (1995) recommended using a probe to place about three-quarters of the fumigant dosage 0.3 to 1.5 m below the surface of the grain mass and placing the remaining fumigant in aeration ducts in the base of the structure. Tarps can then be applied to partially filled bins to limit the fumigated volume and minimize leakage. In probe and tarp fumigation, workers must enter the grain bin and are exposed to entrapment hazards and fumigants during the tarping process.

A concentration of 200 ppm for 100 hours is the guideline to kill common stored wheat pests in Oklahoma (Noyes and Phillips, 2004) and in Kansas. It is virtually impossible to completely seal existing grain storage bins so that some phosphine does not leak out over the course of the fumigation. When sufficient levels of phosphine are not maintained for the duration required to eradicate all life stages of insects, the surviving life stages can continue to infest the grain. Furthermore, the surviving insects are likely to be the most resistant members of the population. Incomplete fumigations are a significant cause of development of phosphine resistance, which has been reported in stored grain pests (Benhalima et al., 2004; Lorini et al., 2007). Resistance to phosphine is a critical concern for grain storage managers because of the widespread use pattern.

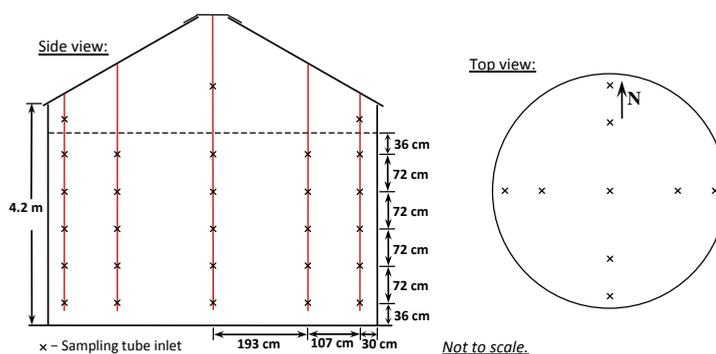
A safer and more effective alternative to traditional fumigation practices is the use of closed-loop fumigation (CLF) systems in grain handling and storage facilities. The typical CLF system uses a small fan and duct system to recirculate fumigant in the grain storage bin by drawing it out of the headspace and injecting it back into the bottom of the grain storage bin. The fumigant rises up through the grain until it enters the headspace where the cycle repeats. After several cycles through the grain storage bin the fumigant is evenly distributed. Recommended CLF flow rates of 0.0016 to 0.008 m<sup>3</sup>/min per m<sup>3</sup> grain (0.002 to 0.010 cfm/bu) provide several air changes through the grain storage bin per day to provide sufficient mixing in the usual time that phosphine pellets/tablets react (Noyes et al., 2002). CLF systems that distribute fumigant evenly throughout a grain storage bin can allow the use of less phosphine in a fumigation because the manufacturer's recommended application rates are elevated to allow for unequal fumigant distributions in typical grain storage bin (Kenkel et al., 1993; Noyes and Kenkel, 1994; Noyes et al., 1995; Hardin et al., 2009).

Phosphine is chemically stable at the normal conditions inside a grain storage bin and diffusion through the envelope of the structure is generally negligible, while the major loss of phosphine is through leakage from the structure through cracks and other openings. Pressure from wind and thermal buoyancy are the primary forces that drive the exchange of fumigant with the air outside the structure (Cryer, 2008). Wind flowing around a grain storage bin induces areas of high and low pressure. Wind velocity, direction, and the presence of other structures all affect the pressure distribution on the grain storage bin, and in turn, influence leakage (Mulhearn et al., 1976; Banks et al., 1983; Bibby and Conyers, 1998). CLF systems produce a pressure differential across the grain mass that can significantly contribute to leakage in a grain bin that is not sufficiently sealed.

The objective of this study was to evaluate the distribution of phosphine in temporarily sealed grain storage bins during conventional fumigation with probed tablets and compare to distribution during closed-loop fumigation of the same bins.

## Materials and Methods

The fumigation experiments were conducted in two corrugated steel bins each containing 95 metric ton of hard red winter wheat. Bins were 6.6 m in diameter with 4.2 m eave height and 6.0 m peak height. The wheat was center-loaded in the bin and leveled at 3.6 m deep. Plastic sampling tubes (3 mm inside diameter) were attached to support cables, which were installed with two in each cardinal direction plus one in the center with five sampling tubes on each support cable (Fig. 1). This provided nine sampling tube inlets at each of five depths in the grain mass plus three in the headspace, giving 45 sampling points distributed through the grain mass out of 48 total sampling points in the bin. The tubes ran outside the bins through an opening designed for that purpose with the ends arranged in a grid on a board for easy access. The bins were temporarily sealed using 4 mil plastic sheets covering all opening using contact adhesive. The sidewall to eave joint had been



previously sealed with caulk.

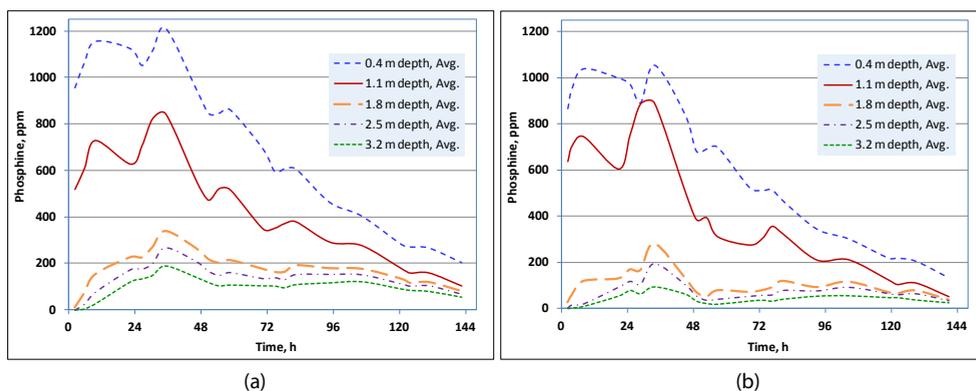
**Fig. 1** Experimental bin showing sampling tube locations.

Each bin was fumigated at the minimum label rate of 90 tablets per 27 metric ton of grain for both conventional and CLF fumigations. In the conventional fumigations the tablets were evenly dispersed among three depths of 1.2, 0.6, and 0.3 m from the surface at nine locations near the nine support cables. In the CLF fumigations the tablets were dispersed across the top surface of the grain and circulation fans were run for 45 minutes every six hours throughout the fumigation. After phosphine application, the concentrations of phosphine gas at various depths were measured manually with a Dräger X-am 5000 (Drägerwerk AG & Co., Lübeck, Germany) personal monitoring instrument using a Dräger X-am 1/2/5000 pump to draw the gas from the grain mass through the sampling tube and the lines of the gas sensor. The readings were collected approximately at 4-8 h intervals for 5 to 6 days. Phosphine concentrations were averaged for all nine sampling points at each depth in the grain and for the three sampling points in the headspace and the resulting means

graphed versus time. The full data from all 45 sampling points in the grain were analyzed to determine statistics such as mean, standard deviation, minima, and maxima for each sampling time.

## Results

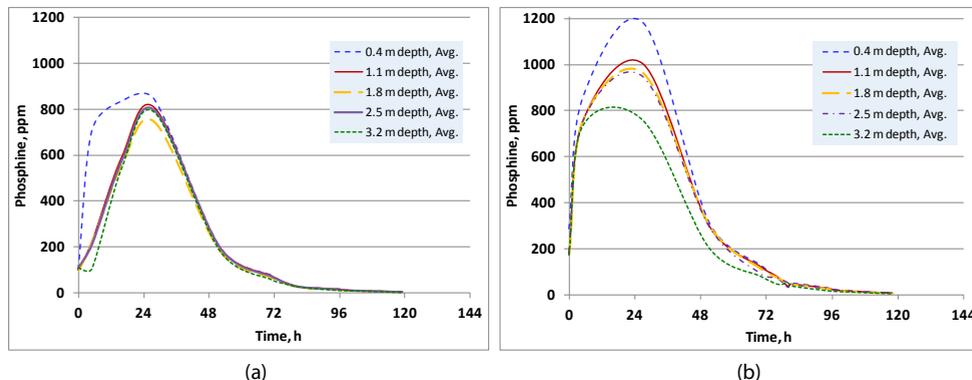
Figure 2 shows phosphine concentrations during a six-day conventional fumigation in the two bins. The two monitored depths with the highest readings (0.36 m and 1.1 m) had average doses above 200 ppm for most of the first five days, with only minor differences in the trends between the two bins. These two depths with the highest readings were the two nearest the top surface and fell within the range of depths where the pellets were introduced. The three lower depths monitored (1.8, 2.5, and 3.2 m), all below the depths where the pellets were placed, received average doses at each level less than 200 ppm with a few exceptions between 20 and 60 h. The peak readings at those three lower depths occurred at 34 to 35 h in both bins and one of the six average readings at those depths (from three readings each in two bins) reached 339 ppm at that peak (Fig. 2), while the rest were all below 300 ppm at the peak. The readings at the upper two depths also peaked at 34 to 35 h indicating the aluminum phosphide tablets were spent shortly after that time and the subsequent declining phosphine readings resulted from continued leakage out of the bins. In general, bin 2 had slightly lower average phosphine readings at all depths and at all times than did bin 1. These lower readings were likely due to bin 2 having slightly greater overall leakage around the upper portions of the bin, where maximum concentrations occurred, than did bin 1. The upper two depths in these bins (0.4 m and 1.1 m) had average concentrations above 200 ppm for the recommended 100 h (Jones et al, 2008), but lower three depths did not in either bin.



**Fig. 2** Phosphine concentrations during conventional, probed-tablet fumigation in (a) bin 1 and (b) bin 2, each containing 95 metric ton of wheat at 24°C.

Some of the individual sampling points at the lower three depths had zero readings for much of, and occasionally all of, the six-day conventional fumigation. The maximum number of sample points with a zero phosphine reading was 26 and 24 for bin 1 and 2, respectively, which occurred at the first reading (2.3 h). The minimum number of sample points with a zero phosphine reading was six and ten for bin 1 and 2, respectively, which occurred at 104 h. After 104 h the number of sample points with a zero reading began to increase again in both bins. It was also observed that 14 of 45 monitored locations in both grain masses received no phosphine (all readings during the fumigation were zero) or nearly no phosphine (majority of readings during the fumigation were zero). Both bins had similar patterns of phosphine average dosage for all depths, but there was slightly more variation observed between the two bins for the highest monitored depths (Fig. 2 and 3). However, the greater variability at the upper two depths could be due to the overall higher phosphine readings for those depths having proportionally higher deviations than in the lower three depths with the lower readings.

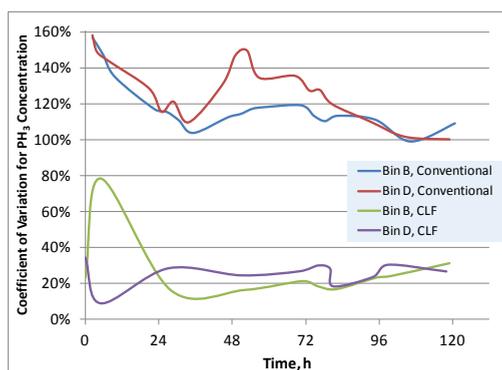
Figure 3 shows phosphine concentrations during a five-day CLF fumigation of the two bins. All peak readings, which occurred during the first 48 hours of fumigation, were in the same range as the highest peak readings in the conventional fumigations, 800 to 1200 ppm, with no depths having very low peaks (below 300 ppm) as seen at many of the lower depths in the conventional fumigations.



**Fig. 3** Phosphine concentrations during CLF fumigation in (a) bin 1 and (b) bin 2, each containing 95 metric ton of wheat at 24°C.

In bin 1, the distribution of average readings at each depth showed very little variation with only one data point early in the fumigation, at 5 h, deviating from the uniform trends. The peak readings at 28 h in bin 1 were all very close to each other at approximately 750 to 850 ppm. In bin 2, the peaks at 27 h varied from 800 ppm to 1200 ppm, but these were much more uniform than the peaks in the conventional fumigations. For both bins, average concentrations remained above 200 ppm for all five heights for 48 h, but no depths stayed above 200 ppm for more than 60 h. In general, bin 1 had slightly lower average phosphine readings at all depths and at all times than did bin 2. These lower readings may have been due to bin 1 having slightly greater overall leakage around the lower portions of the bin, which received the maximum pressurization from the circulation fan, than did bin 2.

The variation between readings at different depths in both bins for both conventional and CLF fumigations was evaluated by calculating the coefficient of variation from the mean and standard deviation of the 45 concentration values in each bin (Fig. 4). The conventional fumigations showed much larger values of coefficient of variation because of the large deviation between readings at different locations, especially between different depths (Fig. 2), within the grain mass. The CLF fumigations almost always had coefficients of variation under 30% except one data point in one test early before the recirculating airflow had produced uniform distribution.



**Fig. 4** Coefficient of variation over time for the average of the phosphine concentrations at 45 locations in the four bins during fumigations.

## Discussion

Flinn and Reed (2008) found similar results to ours in tall concrete bins when fumigating with pellets. In the absence of wind or chimney effects the phosphine gas did not move far from the pellets so that locations in the bins without pellets did not receive lethal concentrations of gas for fumigation. When there were significant chimney effects in those tall bins due to temperature differences, the phosphine gas moved to other locations and moved out of the bins through leakage from openings in the top and bottom of the bins. Cook (2016) measured phosphine gas concentrations during CLF fumigation of small (45 to 50 metric ton), well-sealed metal bins. Gas was circulated using a thermosiphon system (Boland, 1984). The CLF systems of Cook also maintained relatively uniform, but higher, phosphine gas concentrations during fumigations similar to our CLF fumigations. With the well-sealed bins in that study, phosphine gas concentrations always remained above 100 ppm for 125 h. The longer maintenance of gas concentrations was clearly a result of more effective sealing on those bins compared to the temporary sealing of our bins.

Fumigation treatments reported by Jones et al. (2008) demonstrated some similarities and some differences in comparison to our results. In their tests, pellets were distributed uniformly in tall concrete bins while turning the grain in the conventional application and the resulting gas concentrations were compared to those in identical bins under CLF fumigation. In the CLF bins, the same number of pellets were distributed on the top surface of the grain with phosphine gas then distributed with intermittent running of a recirculation fan. In the conventional bins the three monitored locations, top, middle, and bottom never reached 200 ppm of phosphine gas at any time during 72 h of monitoring, which is like the locations in our conventional fumigation bins that were not in close proximity to the tablets. CLF bins in their tests maintained an average phosphine gas concentration above 1000 ppm for approximately the last 60 h of the same test period, which indicates there was much less leakage from those concrete CLF bins than from our steel CLF bins.

Our measurements showed uneven phosphine distribution patterns and leakage over time when fumigating with conventional and CLF techniques. With the conventional probed tablet fumigation, the uneven distribution of phosphine at the minimum label rate resulted in effective doses in only some portions of the bin. The distribution of phosphine gas was much more uniform when using the CLF fumigation method. Both types of fumigations exhibited wind-driven leakage that was often excessive, while the CLF bins also exhibited continual high leakage due to the fan pressures in the bin and ductwork during the intermittent fan operation. Leakage driven by wind effects and recirculation fan pressure in these temporarily sealed bins prevented lethal phosphine gas dosages for the recommended length of time in all or part of the bin in all tests.

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## Fumigation of Apples and Sunflower Seeds with Phosphine – Desorption Behavior and Aroma Profiles

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Since many decades, fumigations of stored products are an accepted and worldwide used method to control pest organisms. Infested stored goods can be treated with anoxia and chemical fumigants to eradicate pests very effectively and without any movement of the products. Stored-product insects present a serious problem causing economic loss and contamination of food destined for animal or human consumption as well as a direct physical damage of materials and objects.