

EVALUATION OF SEALED STORAGE SILOS FOR GRAIN FUMIGATION

by

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## Abstract

Fumigation of stored grain is a common way to kill stored-grain insect pests. However, fumigating in unsealed structures is the leading cause of control failures and subsequent development of insect resistance. Sealing the storage structure is the only practical way to ensure a complete kill of all insects at all life stages. The cost, effort, and feasibility of sealing a U.S. corrugated steel silo during construction was evaluated and compared against an Australian sealed silo designed for fumigation. Gas monitoring and thermosiphon recirculation equipment was installed on both silos. Fumigation efficacy was evaluated using pressure half-life decay times, fumigant concentrations, insect bioassays, and grain quality data. Three fumigations with phosphine (PH<sub>3</sub>) pellets or tablets and two with VAPORPH<sub>3</sub>OS<sup>®</sup> cylinderized PH<sub>3</sub> and ProFume<sup>®</sup> cylinderized sulfuryl fluoride (SF) were performed in each silo for a total of ten experimental treatments. The Australian silo required 266 man-hours to construct and cost \$180 for additional sealing, compared to 359 man-hours and \$3,284 for constructing and sealing the U.S. silo. The Australian silo had a maximum pressure half-life decay time of 163 s versus 50 s for the U.S. silo. At application rates of 1.5 g/m<sup>3</sup> of PH<sub>3</sub> both silos maintained an average concentration of approximately 0.28 g/m<sup>3</sup> for 14 days. With thermosiphon recirculation the average minimum-to-maximum PH<sub>3</sub> concentration ratio in the U.S. silo was 0.52, compared to a ratio of 0.17 when fumigating without thermosiphon recirculation. Greater than 99% adult mortality was observed in all insect bioassays which included PH<sub>3</sub> resistant strains of *R. dominica* and *T. castaneum*. The average emergence from fumigated bioassays was 7 adult insects, compared to an average of 383 adults for the non-fumigated controls. Grain stored for 10 months in the sealed silos increased from approximately 11.5% to 17% m.c. in the top 0.3 m of grain, and decreased in test weight from approximately 77 to 65 kg/hL. Although the Australian

silo retained higher fumigant concentrations than the U.S. silo, fumigations were successful in both. Long-term storage in sealed silos is a concern because grain quality can deteriorate due to condensation and mold in the top grain layer.

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## **Dedication**

To my marvelous and longsuffering wife, Heather. I love you, baby.



## **Preface**

The world population is approximately 7 billion and is expected to grow to over 9 billion by 2050. For the food supply to keep up with this population growth, farmers must produce more food in the next 40 years than has been grown in the history of civilization (Sheeran 2012). According to the Food and Agricultural Organization of the U.N., cereal grains account for nearly half of the calories consumed by humans worldwide, and are therefore a key component in global food security. Kader (2004) estimated that nearly one third of the total grain harvested worldwide is lost before consumption or sale. Reducing the amount of grain lost after harvest is an important strategy to fight hunger and poverty. Many methods may be employed to preserve stored grain quality and quantity; this research project considered fumigation in sealed silos.

## Chapter 1 - Introduction

For thousands of years grain has been stored in sealed containers (De Lima 1990; Reed 1992; Sigaut 1980). In ancient times, agricultural societies stored their surplus grain in underground pits covered with mud or bricks. This kept the grain safe from the elements and restricted the entry of rodents, birds, and insects. Storing grain in mud or clay pots and jars continues in some places in the world today (Calderon 1990). Modern scientific research on sealed storage of grain was started in France in the 1800's by Ternaux, and others there continued it throughout that century (Sigaut 1980). In the 1920s, Dendy and Elkington (1920) performed a series of experiments studying the control of grain insect pests using grain stored in airtight jars.

More recently, chemical fumigants have been used to protect grain in sealed storage. However, the last half of the 20<sup>th</sup> century saw many fumigants lose their approved use status because of their negative environmental impact or because of toxicity and safety concerns (Bell 2002, Mills 2000). One fumigant still in use is phosphine (PH<sub>3</sub>) gas. Phosphine is inexpensive, easy to obtain, and relatively easy to use, making it the most widely utilized grain fumigant worldwide. However, its effectiveness and longevity may be under threat due to widespread PH<sub>3</sub> resistant insect populations (Opit et al. 2012).

Phosphine can be applied to stored grain via solid pellets and tablets, or via pressurized cylinders. Care must be taken that the storage structure is sealed before fumigation, but this is not always the case. In the U.S., the common bolted steel silos are neither manufactured as sealed nor sealed sufficiently after construction. Leaks present in the silo will invariably result in fumigant concentrations that fail to kill the more resilient insect pests (Winks 1986). In addition

to low levels of fumigant, extremely high levels can also be problematic. Nakakita et al. (1974) found that as adult *Sitophilus zeamais* were exposed to increasing concentrations of PH<sub>3</sub> (up to 20,000 ppm), they underwent narcosis and the higher levels of PH<sub>3</sub> had no effect on account of their metabolism being inhibited.

In the event of an incomplete kill, PH<sub>3</sub> resistant insects continue to produce offspring, thus creating a population of insects which are resistant to the fumigant. Resistance to fumigants is a global problem, and the number of resistant insect populations has increased significantly in the last decade (Newman 2010; Bell 2000; Prickett 1987).

Because phosphine is relied upon so heavily, insect resistance is a major problem for grain producers, merchants, and processors. If phosphine loses its ability to kill insect pests, grain producers and handlers will have a much more difficult (and expensive) time maintaining grain quality.

Due to the potential for PH<sub>3</sub> resistance development and the need to maintain its long-term effectiveness, and considering the success that sealed storage has had in Australia, research into the feasibility of sealed grain storage to ensure long-term efficacy of PH<sub>3</sub> fumigation in the U.S. is timely and warranted.

## **Chapter 2 - Objectives**

The objectives of this research were to:

1. Document and calculate the effort, costs, and feasibility of sealing bolted steel hopper bottom grain silos while under construction. (Addressed in Chapter 3)
2. Evaluate fumigation success or failure in sealed grain silos using fumigant concentration and insect bioassay data. (Addressed in Chapter 4)
3. Evaluate thermosiphon recirculation equipment for facilitating fumigant dispersal in sealed grain silos. (Addressed in Chapter 4)
4. Assess the suitability of storing fumigated grain for extended periods in sealed silos. (Addressed in Chapter 4)

# **Chapter 3 - Construction, Sealing, and Pressure Testing of Corrugated Steel Grain Silos**

## **Introduction**

The primary goal of grain storage is preserving grain quality. One of the major threats to grain quality during storage is insect infestation. Stored grain insect pests deteriorate grain by damaging whole kernels which facilitates mold growth. Insect pests can be controlled by fumigation which is the addition of toxic gas to the inside of the grain storage structure that kills all insects present. For a fumigation to be successful the gas concentration and exposure time to insects need to be held for a sufficient amount of time to kill all insects at all life stages. When the fumigation is complete, fresh air is forced through the grain to remove the fumigant.

The fumigated structure should be sealed prior to the fumigation because leaks allow the fumigant to escape, resulting in control failures where less than 100% of the insects are killed. Fumigating grain in leaky structures has been cited as the main reason for control failures that can lead to insect resistance (Leesch et al. 1995). In unsealed or poorly sealed silos the fumigant leaks out, creating selection pressure for resistant individuals in the insect population.

The vast majority of U.S. on- and off-farm grain storage structures are not engineered to be sealed for adequate levels of gas-tightness. Instead, they have to be sealed temporarily before fumigation which, especially in larger silos, can add substantial labor and material costs and results in greater risk of fumigation failures from inadequate sealing than with silos sealed by

design. A sealed structure keeps the fumigant within the grain mass long enough to achieve a complete kill of insects.

### **History of Sealed Storage**

Modern bulk grain storage silos made of concrete and steel keep grain dry, but are not usually airtight. Sealed storage here refers to a grain structure that is sufficiently airtight to contain a fumigant long enough to completely kill all grain insect pests at all life stages. The level of sealing differentiates an unsealed, conventional bulk storage structure that is designed primarily to keep water out, from a hermetic storage structure that is designed to prevent the passing of air or gas through the structure fabric.

In 1963, export regulations enacted by the Australian Government required that wheat bound for export from Australia be inspected and free from infestation of live insects (Barker and van Graver 2004). A reliance on residual insecticides, especially malathion, rapidly led to resistance in target grain insect pests such as *Rhyzopertha dominica* (F.), *Tribolium castaneum* (Herbst), *Oryzaephilus surinamensis* (L.), and *Sitophilus oryzae* (L.). The Working Party on Grain Protectants was formed in 1973 to develop alternative protectants to malathion. However, resistance to the replacement protectants began to develop, especially in *R. dominica* (van Graver and Winks 1994). Even today, widespread resistance to most grain protectants exists in Australia (Collins 2006).

Around the same time, customer preferences in overseas and in particular Australian markets began calling for reducing pesticide residues in grain products. Pesticide residue-free grain is a

major marketing emphasis for Australian grains (Anon. 2014). With shrinking options due to increasing insect resistance to grain protectants, a long-term solution to grain protection other than chemical protectants was needed, with more of a focus on phytosanitary handling and storage of grains and treatments with non-residue leaving fumigants. An outreach initiative was started to seal existing grain storage structures to ensure successful phosphine (PH<sub>3</sub>) fumigation based on previous work by Banks and Annis and others. In the 1980s, Cooperative Bulk Handling (CBH), a co-op that handles grain in Western Australia, invested in sealing its grain storage structures to be used for controlled atmosphere and fumigation application. Phosphine resistance monitoring programs (Collins et al. 2002; Newman 2010) and effective sealing standards (Banks and Annis 1981; Banks and Ripp 1984) led to extension efforts to educate farmers about the benefits of sealed grain storage and responsible fumigant management (Delmenico 1993; Chantler 1983). Outreach efforts also encouraged silo manufacturers to engineer more readily sealable silos (Ellis 1983). Fumigation research developed advances such as use of the thermosiphon to passively disperse fumigants within grain using solar radiation. Boland (1984) began using it in a concrete silo and that was followed by others (Newman et al. 2012).

Recognizing the threat to PH<sub>3</sub> as an effective fumigant due to insect resistance and human safety risks, in 2010 the Australian Government published standard AS 2628 for sealed grain silos (AS 2628-2010). According to this standard, a grain silo can be considered “sealed” only when an applied pressure on the inside of the structure depletes by 50% in no less than three (for older silos) to five minutes (for new silos). For example, if a new silo is pressurized to an internal pressure of 500 Pa, it should be sufficiently airtight to lose no more than 250 Pa in five minutes

(Banks and Ripp 1984; Newman 1990). The pressure can be applied to the silo using an air compressor via a tire valve installed on the sidewall or a vacuum cleaner through a port with a shutoff valve. This test is known as the half-life pressure decay test, or variable pressure test, and is easy to perform with a simple U-tube manometer.

In the early days of sealed silos, Banks and Annis (1981) stated that the standard was difficult to achieve in silos and bunkers smaller than 300 MT, and that pressure testing was difficult in storages greater than 10,000 MT. Ripp was able to successfully perform a pressure test on a structure of 260,000 MT (Banks and Ripp 1984) and today silos as small as 10 MT are sold as sealed and conform to the Australian sealing standard.

Another method for testing the level of gas tightness in a silo or building is the equilibrium pressure-flow test, or constant pressure test. This test measures the airflow rate required to maintain a given pressure inside a silo. It is commonly used by the heating, ventilation, and air conditioning industry (HVAC) to determine the permeability of structures to predict air exchange between the inside of structures and the outside. It has also been used in silos and flour mills (Meiering 1982, Chayaprasert 2010). This test is considered to be more accurate but requires sophisticated equipment to perform. It is therefore not widely used for testing grain silos.

Sealed storage should not be confused with hermetic (i.e., airtight) storage. Hermetic storage is designed for zero air exchange between the inside and outside of the structure, whereas sealed storage allows for some amount of leakage. The goal is that leaks do not cause fumigant concentrations to fall below levels that kill all life stages of all insects (Boland 1984). In



addition, if a structure does not meet the pressure standard, a control failure is not necessarily inevitable. The five minute pressure decay time indicates a level of sealing that minimizes the amount of leakage due to wind and chimney effects. While leakage via pressure differences is greater than that due to wind or chimney effects, this level of sealing allows the structure to hold the fumigant long enough to kill all insect life stages without having to add more fumigant.

Navarro (1998) recommended pressure decay times ranging from 1.5 minutes for a small (up to 500 m<sup>3</sup>), filled silo, and up to 6 minutes for a large (2000 to 15,000 m<sup>3</sup>), empty silo.

### **Sealed Storage in the U.S.**

Sealed storage is not as developed in the U.S. as it is in Australia. Even though agricultural extension papers as far back as the early 1920s emphasized the importance of sealing enclosures prior to fumigation (Flint 1921), control failures due to unsealed structures remained commonplace through the 20th century (Noyes et al. 2000). Nevertheless, some of the same pressures that spurred the development of sealed storage in Australia may encourage a similar development in the U.S.

Widespread insect resistance to grain protectants has arisen in the U.S. (Subramanyam and Harein 1990). In the late 1980s and early 1990s, populations of malathion resistant *T. castaneum*, *O. surinamensis*, and *R. dominica* were found on farms in Minnesota and Oklahoma.

Fumigants once approved for use in raw grain such as ethyl dibromide, carbon tetrachloride, carbon disulfide, and methyl bromide have not been reissued approval because of concerns about environmental impact, harmful residues in the grain and worker safety (Bell 2000; Haritos et al.

2006; Donahaye 2000). If  $\text{PH}_3$  loses its efficacy due to strong insect resistance, stored grain managers will have a much more difficult and expensive task of protecting grain from insect damage.

Another factor that may contribute to developing sealed storage is the growing interest in non-chemical control of insect pests such as under modified (MA) or controlled (CA) atmosphere storage. Similar in principle to fumigation, under MA and CA the composition of air inside the storage structure is altered to create a lethal atmosphere to insect pests and promote the safe storage of products. Though the terms are often used interchangeably in the literature, CA is usually used when stored products are treated with  $\text{CO}_2$  or  $\text{N}_2$  gases to displace air in order to expose the insects to a high  $\text{CO}_2$  or low  $\text{O}_2$  environment in the structure, respectively. The atmosphere is continually monitored and maintained (i.e., controlled) using tanks of compressed gas or gas generation equipment. The term MA usually refers to an atmosphere within a sufficiently sealed structure that is altered to a low  $\text{O}_2$  or high  $\text{CO}_2$  state through the respiration of organisms present. It is also used as a general term to refer to any structure in which the proportion of atmospheric gases and/or pressure have been altered to preserve stored product quality or control pests such as when treating organic grains with  $\text{CO}_2$ . In the early 1980s, the Environmental Protection Agency (USEPA) approved these non-chemical, naturally occurring gases for disinfesting raw and processed agricultural commodities. The organic food sector is growing, and uses CA and MA to disinfest not only grains but also other foods like nuts, fruits, and vegetables (Dilley 1990). Some major U.S. grain handling companies investigated and adopted the use of CA (Jay and D'Orazio 1983), including for disinfesting organic rice and blue

corn intended for human consumption. Hermetically sealed storage structures are designed to maintain the low O<sub>2</sub> environment that CA and MA storage require.

Phosphine and other fumigants are toxic to humans and many instances of injury and death caused by exposure to fumigants can be found in the literature (Sudakin 2005, Singh et al. 1996, Anon. 1999). Another benefit of sealed silos is prevention of gas leaks into worker areas and into the nearby environment which increases worker and bystander safety.

### **Fumigation in Steel Silos**

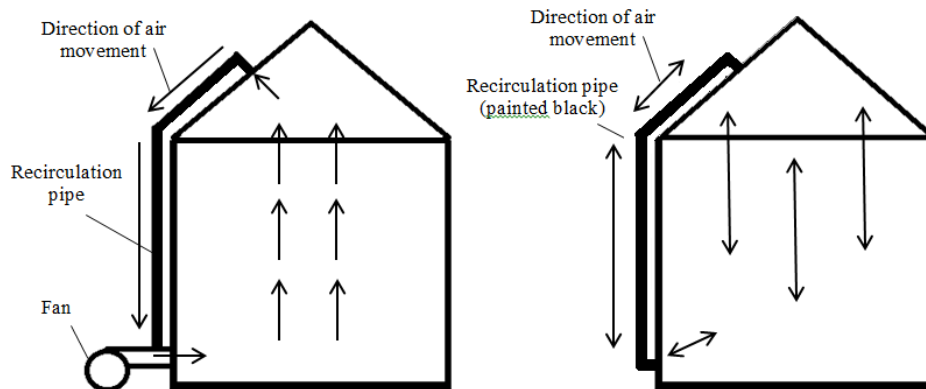
Steel grain silos consist of rounded steel sheets that are either welded or bolted together, a sloped steel roof, and a floor which may be flat or a conical shaped hopper to facilitate unloading of grain. The wall sheets can be smooth or corrugated for more strength, and come in various gauges. Silos have capacities from less than 500 bushels to over 1 million bushels. Welded silos are easier to seal successfully because the sidewall seams are already sealed with the weld. In contrast, when sheets are bolted together there is a possibility for air to escape between the seams. Rubber stripping is usually placed between the sheets in a bolted silo to seal seams. Grain inlets, discharge chutes, air vents, aeration ducting, and the junctions between the wall and floor and the wall and roof are places to seal. To minimize the potential for leakage, in some regions silos are painted with a reflective white paint that helps to reduce solar induced temperature rise in the silo which leads to pressure differences between the inside and outside of the silo. Pressure differences are a main cause of air exchange between the silo and external environment.

The grain surface in a silo can also be covered with impermeable tarpaulins to reduce fumigant leakage. This has been done with varying degrees of success (McGaughey and Akins 1989). It is labor intensive because someone has to climb inside the silo and drape the tarpaulin over the grain surface and ensure it is tucked into the grain along the outer edge in order to keep the fumigant trapped below the tarp.

For bolted steel silos, Noyes et al. (1999) recommended sealing the roof eave gap using closed-cell foam strips with adhesive backing. For narrow gaps of less than 6.4 mm, they recommended using urethane or silicone caulk. Urethane is more expensive but has greater adhesion, UV resistance, and durability. Additionally, grain conveying, loading and discharge equipment, distributors, downspouts, and aeration fans need to be caulked to make the silo airtight (Newman 1990). It is recommended that steel silos should not remain sealed after fumigation or CA treatment because of the chance for moisture condensation in the headspace (Casada and Noyes 2001). Sealable ventilation fans should be added to sealed silos to eliminate humid air from condensing on the underside of the silo roof and dripping onto the top of the grain. This also aids in clearing a silo of gas after the fumigation is complete.

Uniform gas distribution within the storage structure is important when treating grain with a fumigant. Pockets of low fumigant concentrations can form due to lack of gas circulation allowing insects in those locations to survive. Gas circulation is achieved either by forced or natural convection currents within the grain mass. Winks (1992) suggested that fumigation using low velocity forced convection recirculation of gas in a leaky silo has a greater chance for fumigation success than relying on natural convection currents to recirculate gas in the same silo.

Figure 3.1 shows a closed loop recirculation design that uses a fan to draw gas from the headspace and force it up through the grain mass from the plenum (left) and a closed loop design in which recirculation depends on natural convection currents (right). In this example the passive recirculation design utilizes an externally installed black pipe that is heated by solar radiation. That creates a convection current which enhances gas distribution through the grain mass in the structure. The black pipe is known as a thermosiphon because the temperature difference between the air inside the pipe and the air inside the silo draws (i.e., siphons) the gas from the lower connection at the bottom of the silo (i.e., plenum) to the upper connection on the roof (i.e., headspace) or vice versa. These natural convection currents vary in velocity and direction as a result of varying temperature differences throughout the day. In both cases the silo is sealed to the outside and the gas is distributed only through the closed loop system.



**Figure 3.1** Closed loop fumigant recirculation using forced (left) versus natural (right) convection currents.

In the U.S., the majority of grain is stored on-farm (NASS 2014) and most storage structures are corrugated steel silos. These silos are usually open-eaved to facilitate the removal of moisture-laden air from the headspace. In Australia in the 1970s approximately 25% of grain was stored

on-farm (Banks and Ripp 1984). Research to investigate the viability of sealing on-farm storage was undertaken to ensure successful fumigations with the dual goals of achieving insect- and residue-free grain and ensuring the future of PH<sub>3</sub> as an effective fumigant. Currently, nearly all grain silos sold in Western Australia are engineered to comply with Australian Standard 2628 for sealed silos, and adoption is growing in Eastern Australia. Partly as a result of successful adoption of sealed silos, today farmers have the capability to store approximately 50% of grain harvested (GRDC 2013).

A study was undertaken to compare the materials and time effort required to successfully seal a U.S. corrugated steel silo versus an Australian sealed silo. The Australian silo was designed as a sealed silo and the U.S. silo was a conventional open-eave design. The Australian silo was used as a benchmark for gas-tightness due to its advanced gastight design and construction. The U.S. silo was sealed as it was being constructed with the goal of making it as gastight as feasible. The two sealed silos were equipped with closed-loop fumigant recirculation systems and subsequently tested for gas tightness using the half-life pressure decay test. Efforts focused on minimizing gas loss during multiple fumigation cycles. The goal of this study was to document and calculate the effort, costs and feasibility of sealing these two hopper bottom metal silos while being constructed.

## **Materials and Methods**

### **Silo Construction and Sealing**

Two hopper bottom grain silos were constructed at the Grain Science Complex at Kansas State University, Manhattan, KS. A SCAFCO 1503HBT hopper bottom silo was donated for this

project by SCAFCO Grain Systems Company, Spokane, WA. A Bird's model 2250 Australian sealed silo was shipped to Kansas from Bird's Silos and Shelters in Popanyinning, Western Australia. Table 3.1 shows the silo size and capacity specifications.

**Table 3.1.** Specifications of the Australian (Bird's) and U.S. (SCAFCO) grain silos

	Bird's Silos Model 2250	SCAFCO Grain Systems Model 1503HBT
Diameter	4.40 m (14.4 ft.)	4.57 m (15.0 ft.)
Peak height	6.93 m (22.7 ft.)	7.73 m (25.3 ft.)
Volume	63.3 m <sup>3</sup> (2,235 ft <sup>3</sup> )	71.9 m <sup>3</sup> (2,539 ft <sup>3</sup> )
Bushels (corn)	1,795	2,039
Metric Tons	45.6	51.8

### Site preparation

The silos were constructed and placed on two existing 4.9 m diameter concrete pads (Figure 3.2). The support legs were bolted to the concrete pads to secure the silos. There were existing electricity hookups and plenty of room on the paved driveway to assemble the silo components before placing them on the pads.

For both silos a polyurethane adhesive sealant, Bond+Seal (Wurth USA, Ramsey, NJ) was used. The sealant was applied between all wall, roof and hopper sheet seams, small gaps (less than approximately 6 mm), and around other equipment attachments to the silo such as the thermosiphon and PH<sub>3</sub> reaction chamber. All metal-on-metal contact surfaces were cleaned with alcohol before applying sealant to the surface and connecting those using bolts or rivets.



**Figure 3.2** Site of silo construction and placement at Kansas State University, Manhattan, KS.

### **Pressure testing**

Half-life pressure decay tests were undertaken for each silo under empty and filled states.

Pressure tests were performed in the early morning during calm conditions and when the silo roof and wall temperatures were in equilibrium with the ambient temperature. This avoided pressure increases in the silo due to solar radiation and wind effects that would have otherwise skewed the pressure test results. As a result, most tests were performed between 7:00 and 8:00 am.

The U-shape pressure relief valve piping was used as a monitor for the pressure tests (Figure 3.3). Positive pressure was applied to the inside of the silo using a portable wet/dry vacuum cleaner via one of the ball valves on the 4-way tap installed in the Thermosiphon piping (Figure 3.4). The pressure induced by the vacuum cleaner displaced the oil in the U-shape pressure relief



valve. Two horizontal lines 25 mm apart were drawn on the U-shape pressure relief valve for use with the pressure decay tests. When the oil reached the top line (Figure 3.5) the ball valve was shut and the vacuum turned off to seal the pressurized silo. The time it took for the oil level to recede from the top line to halfway between the top line and equilibrium line was measured.



**Figure 3.3** U-tube pressure relief valve piping connected near the bottom portion of the thermosiphon piping. Also used as a monitor for pressure testing of the sealed silos.



**Figure 3.4** Vacuum cleaner set on “blow” connected to ball valve and pressurizing one of the two sealed silos during a pressurization test.



**Figure 3.5** U-shape pressure relief valve used as a monitor during a pressure decay test showing the silo before pressure was induced (left) and pressurized to approximately 170 Pa (right).

When the hydraulic oil is level with the bottom line of the U-shape tube, the silo is in equilibrium with normal atmospheric pressure. To determine the pressure indicated when the oil reached the top line of the U-shape piping the U-shape pressure relief valve piping was used as a manometer. The silo was kept under pressure using the vacuum cleaner through one ball valve. The other ball valve was opened to allow enough air to egress the silo so the oil was steady at the top mark. A digital manometer was connected to a small port in the thermosiphon pipe to read the pressure inside the silo.

## **Results and Discussion**

### **SCAFCO silo**

Gas tightness is easier to achieve the fewer holes and seams a silo has. The SCAFCO silo had more than 1800 bolt holes and over 152 m of seams, with a total of 57 individual sheets including the wall, roof and hopper sheets. With so many potential leakage sites, a liberal amount of sealant was used between sheets and around bolt holes. The biggest problem areas for sealing silos are the roof-wall and wall-hopper (or wall-ground) junctions. Special attention was paid to these areas during assembly.

### **Roof assembly**

The silo roof consisted of 23 A-shaped sections, including one double wide section for the entrance hatch. Each section had a ridge on both sides which overlapped with the ridge on the adjacent section (Figure 3.6). The seams along the ridge were potential gas leakage points considering the relatively few bolts along the large linear surface. A thick bead of sealant was placed along the ridge between adjacent sections (Figure 3.7).



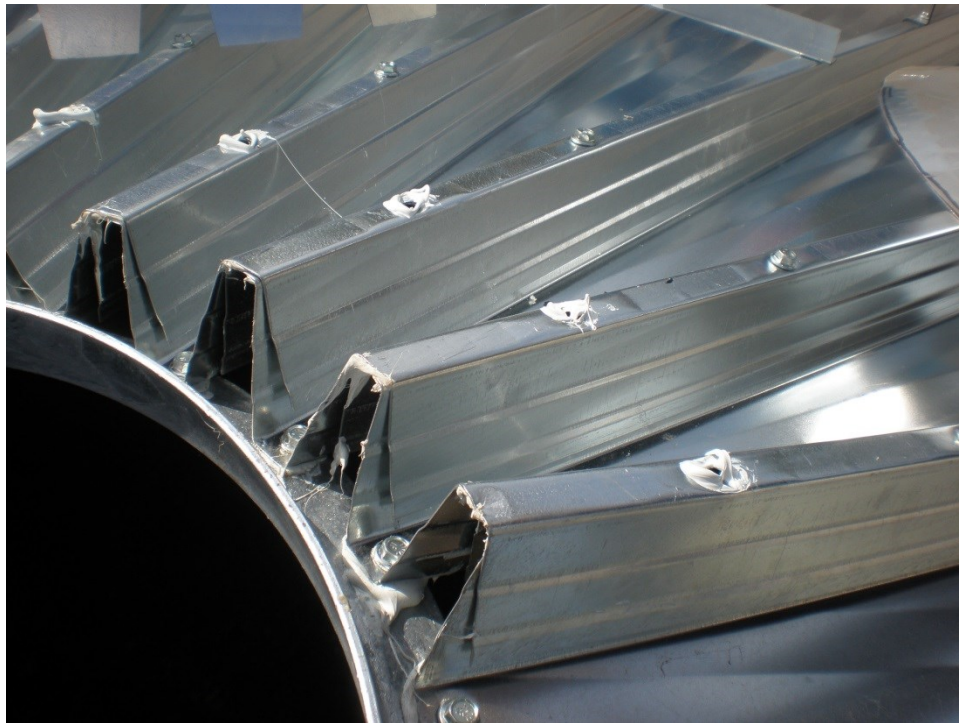
**Figure 3.6** A-shaped sections with overlapping ribs formed the roof of the SCAFCO silo.



**Figure 3.7** Placing sealant along the overlapping ribs between roof sections.

The ridges were joined by six bolts along the top of the ridge. The top end of each section was attached to a central roof ring with two bolts. The gaps between the ring and the roof section ridges were identified as potentially major gas leakage points (Figure 3.8).

To cover the gaps, metal flashing was fabricated that was attached to the roof sections using self-tapping screws and hammered tightly into place against the gaps (Figure 3.9). They were then completely covered with sealant (Figure 3.10).



**Figure 3.8** The large end openings of the roof ribs posed a problem for sealing.



**Figure 3.9** Metal flashing piece to be screwed onto the roof rib opening to cover the ends of the roof ribs.

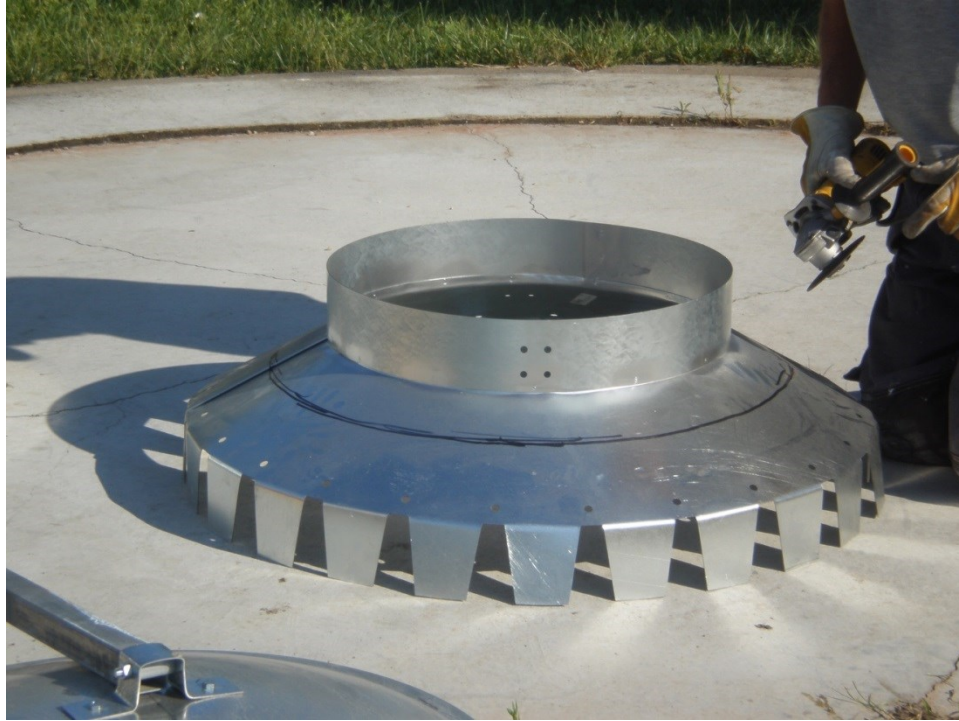
The silo user instructions indicated to keep the bolts loose until all of the sections were placed so they could be adjusted. However, the sealant began to dry well before all sections could be put into place. Adjusting the sections even slightly separated the ribs and sealant, compromising the airtight seal. For this reason, the bolts were tightened immediately. Unfortunately, because the sealant acted as glue between the sections, there was little flex in the roof which made it impossible to attach the last few sections together. As a result, bolts had to be loosened on several sheets to give the roof enough flexibility to fit the last sections together. After all of the roof sections were finally in place, all loose bolts were tightened.



**Figure 3.10** Metal flashing pieces on the ends of the roof rib openings after covering with sealant.

The top lid section provided by SCAFCO was not designed to be gastight. It was a typical “bottle cap” design with the cap sitting directly on top of a thin metal lip and overlapping it to keep out rain (Figure 3.11). Instead of using this lid section, an airtight cap and lip from Bird’s Silos and Shelters was installed (Figure 3.12). The metal lip was rounded so that the closed-cell foam strip installed on the underside of the cap gave an airtight seal when it came in contact with the lip. Ratchet straps were used to secure the airtight cap on the lip.

The entry hatch on one of the roof panels had a cover door that was not sufficiently sturdy to provide an airtight seal. The hatch door was reinforced with angle iron (Figure 3.13) and a strip of closed-cell foam was affixed to the underside where the door met the lip around the hatch. This provided a good airtight seal when closed and latched.



**Figure 3.11** Bottom part of the “bottle cap” lid section supplied by SCAFCO. The thin round lip made it difficult to attain an airtight seal with the cap (not shown) and so was cut out and replaced.

### **Wall assembly**

Each sidewall ring consisted of five sheets. The sheets were connected to each other with 32 bolts on the vertical seams and 32 bolts (for the bottom sheets) or 13 bolts (for the top sheets) on the horizontal seams. Before bolting the sheets together, a bead of sealant was placed between the sheets where they overlapped and each bolt hole was encircled with sealant. The sheets were then overlapped, bolted together, and tightened using an impact wrench.





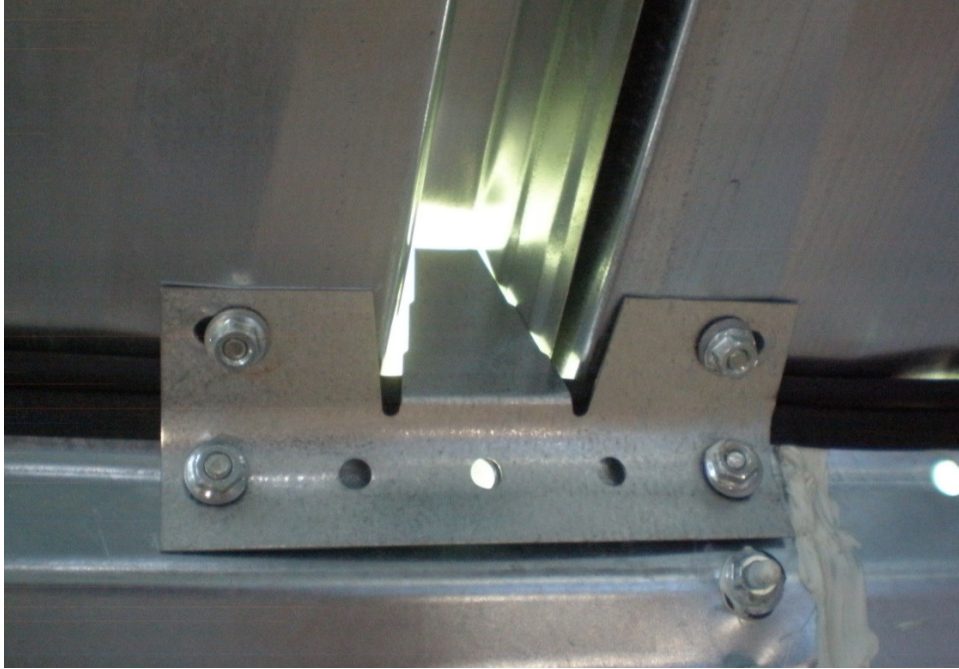
**Figure 3.12** Lid from Bird’s Silos and Shelters installed on the SCAFCO “bottle cap” lid section. The thicker, round-edged lip allowed for an airtight fit with the foam strip installed on the underside of the cap (not shown).



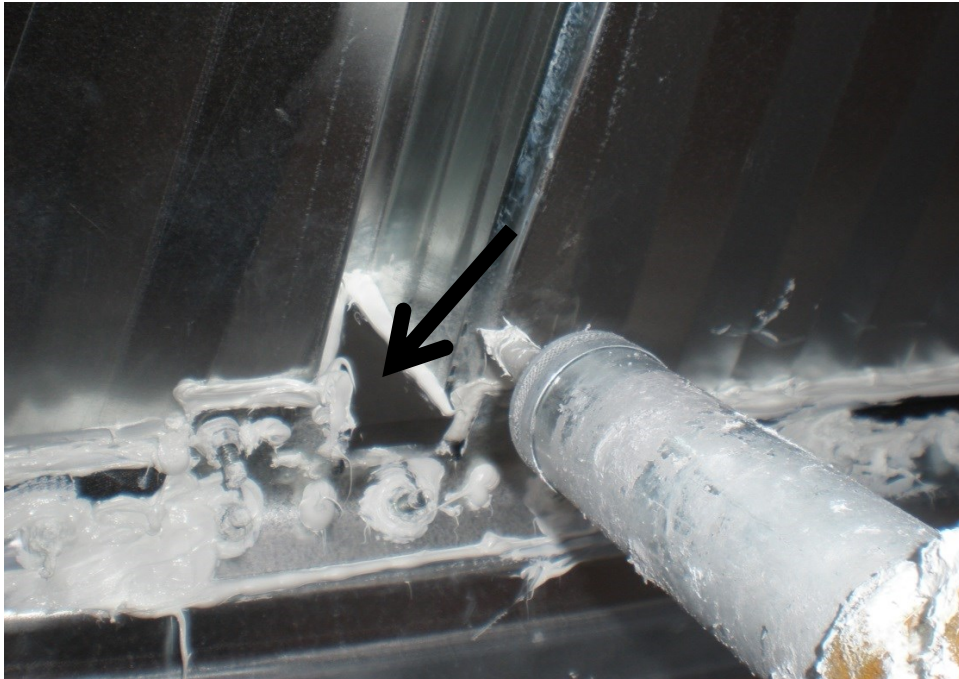
**Figure 3.13** Reinforced entry hatch door installed on one of the roof panels.

### **Roof-wall attachment**

The user instructions advise attaching the roof panels to the top ring while building the roof, however, the roof was completely assembled on the ground before attaching it to the top ring. On the first attempt the roof did not fit when trying to attach it to the sidewall so the bolts were loosened and the roof panels were adjusted to make the roof fit. This led to the roof ribs being shifted and caused the sealant between the roof ribs to come apart and create gaps for air to leak through. Most of these air leaks were found during later leakage testing and then resealed. A sealing kit was included with the SCAFCO silo that consisted of rib clips that closed most of the gap near the wall created by the roof ribs (Figure 3.14), foam sealing blocks to fill in the rest of this gap (Figure 3.15), and a rubber gasket that clipped onto the top wall sheet to provide a seal between it and the roof (Figure 3.16).



**Figure 3.14** “Rib clip” used to attach the roof to the top wall sheet and close most of the gap created by the roof rib.

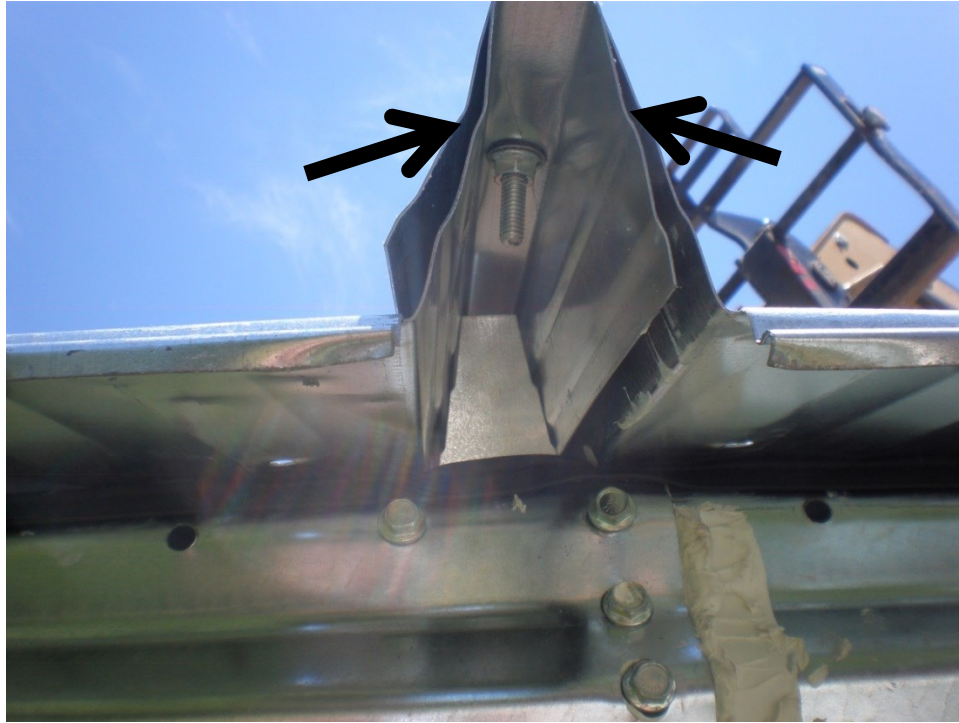


**Figure 3.15** Arrow pointing to the foam block affixed to the rib clip. Sealant was placed around the interior of the entire roof/sidewall interface.



**Figure 3.16** Rubber gasket that was installed around the entirety of the top wall sheets to help seal the seam between the top wall and roof panels.

The sealing kit was designed to keep moisture (i.e., rain) out of the top of the silo. It was not adequate to prevent gas from entering or exiting the silo. The rib clips were attached to the upper edge of the top ring. The clips were designed to both attach the roof to the sidewall, and to close the gap between the ridges of the roof sections. Adhesive spray was applied to the interior side of the rib clips and the foam blocks were stuck to these to close the gap around the metal flashing (Figure 3.14). Sealant was applied to the entire interior seam between the roof and top wall sheets. The seams of the ridges between adjacent sheets were difficult to apply sealant into, as the nozzle of the sealant gun was too large to fit into the tight space (Figure 3.17).



**Figure 3.17** Ridge where the roof sheets overlapped. Arrows indicate where sealant could not be easily applied due to the narrow space and leaks detected during pressure testing could not be fixed.

When the roof and top wall ring were put together, the assembly was lifted into the air by a small crane using a metal ring with a diameter larger than the top roof opening. The metal ring was attached to the crane with straps, placed inside the roof assembly, and used to lift the roof assembly. Two more wall ring sections were assembled and then attached, sealing the seams and bolt holes as described above.

### **Leg and hopper assembly**

The silo support legs were attached to the lower sidewall ring and then fastened to the concrete pad using anchor bolts (Figure 3.18). The hopper cone consisted of 19 sheets, with more than 120 bolt holes per sheet. Because of close tolerance, the hopper sheets were placed in position

and bolted together, but the bolts were not tightened until all hopper sheets were in place (Figure 3.19).



**Figure 3.18** Anchor bolt securing a support leg of the SCAFCO hopper silo to the concrete pad.

Sealant could not be applied around each individual bolt hole as it was on the wall sheets because it would have dried out before all bolts were ready to be tightened. Instead, sealant was applied into the gaps between the hopper sheets as much as possible before the bolts were tightened (Figure 3.20). At the bottom of the hopper, each sheet was bolted to a center ring to form the bottom outlet. A square metal plate was attached to that ring to which the  $\text{PH}_3$  reaction chamber was welded.

Sealant was applied to the seam between the hopper sheets and bottom sidewall sheets. Because of the angle, it was more effective to apply the sealant from the inside of the silo. This was time consuming because someone had to use a ladder braced against the bottom of the hopper bottom to reach the hopper-sidewall junction and apply the sealant.



**Figure 3.19** All of the hopper sheets were put in place before applying sealant between the sheets and tightening the bolts.



**Figure 3.20** Tightening the bolts on the hopper after application of polyurethane sealant into the gap between sheets.

### **Bird's silo**

The Bird's silo was intended to be used as a sealed silo for grain fumigations, and thus was designed to reduce the number of potential leak sites. For this reason, the sidewall rings are constructed out of only one sheet so there is only one seam per ring. The roof and hopper sections are both constructed out of one sheet each and had no potential for leaks other than where they connected to the wall rings. However, to ship the silo to Kansas, these sections had to be cut into four pieces each at the factory and reassembled on-site. The Bird's silo consisted of a total of 14 individual sheets. It required about 600 rivets to fasten the wall sheets together and connect the roof to the top wall sheet plus about 140 self-tapping screws to attach the silo to the hopper base.



## Roof assembly

The roof section pieces were welded together to create a single circular sheet with a “V” cut out (Figure 3.21). The roof sheet was formed into a cone and the two edges of the “V” were welded together (Figure 3.22). A ring was installed on the underside to provide structural stability to the roof. A hole was cut in the peak of the roof to accommodate the cap and lid. The lid was designed by Bird’s to be opened and closed from ground level by a winch and pulley system (Figure 3.23). When closed, the pressure of the pulley on the close fitting cap over the round-edged lip provided an airtight seal.



**Figure 3.21** Inspecting the welded hopper sections before forming the sheet into a cone and welding it to the base structure.



**Figure 3.22** Lifting of the roof sheet of the Bird's silo and forming it into the shape of a cone before welding the final seam.



**Figure 3.23** The roof cap opening and closing apparatus designed to provide an airtight seal with the help of a winch operated from ground level.

### Wall assembly

Each of the four wall rings was cut into three sections at the factory to fit them inside the shipping container for the journey from Australia to Kansas. The ring sections were manufactured approximately 10 cm longer than normal so they could be overlapped and connected together. Sealant was applied to the 10 cm overlap between each section. They were then joined with two rows of pop rivets to form the ring (Figure 3.24). The pop rivets were covered with sealant on the inside of the silo. The roof section was lifted onto the top wall ring and attached using pop rivets (Figure 3.25). Sealant was applied on the inside of the silo on the seam between the roof and top sidewall ring.

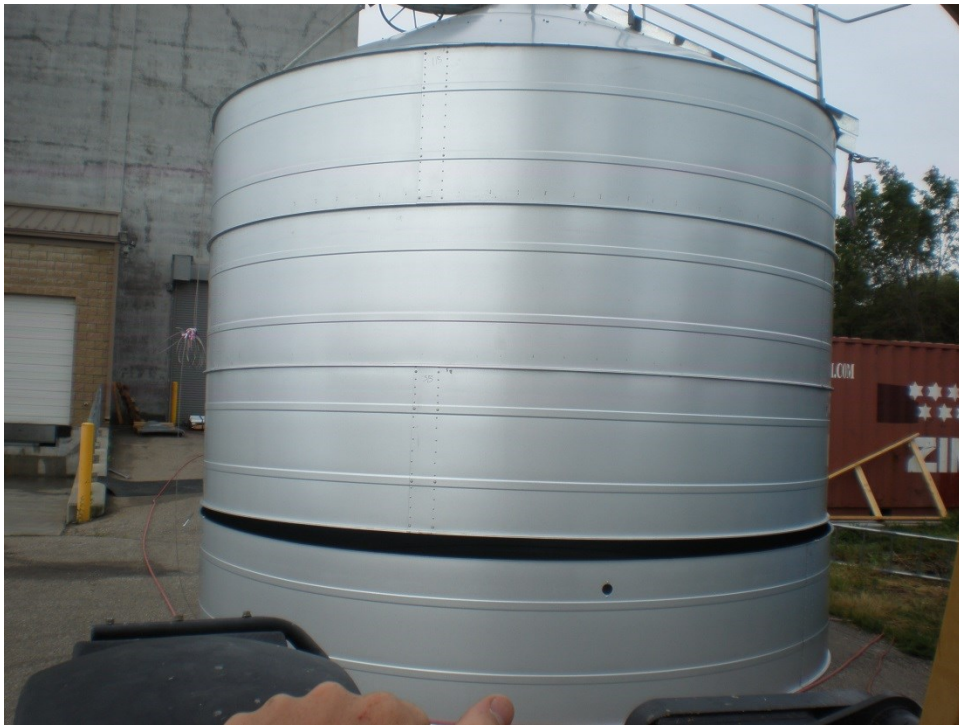
The roof and top ring were lifted and set on the lip of the next ring (Figure 3.26). The rings were attached to each other using pop rivets placed at approximately 10 cm intervals around the entire circumference of each ring seam (Figure 3.27).



**Figure 3.24** The wall sheet sections were overlapped approximately 10 cm. After sealant was applied they were connected with pop rivets.



**Figure 3.25** The roof section was lifted onto the top wall ring and attached with pop rivets.



**Figure 3.26** After a wall ring was finished, the completed sections were lifted onto that ring and connected with pop rivets.



**Figure 3.27** Close-up view of connecting the wall rings to each other using pop rivets.

### **Base and hopper assembly**

The base structure of the Bird's hopper silo was fabricated from 5 cm diameter steel pipe. Twenty four vertical supports were welded between two rings which required a person on-site with welding skills. The supports were approximately 1.5 m tall. The hopper was assembled in the same manner as the roof which provides consistency in manufacturing and for assembly. The section pieces were laid out on the ground and welded together, then formed into a cone. The hopper cone was lifted and set into the base structure and welded to it along its edge. The bottom of the hopper cone was approximately 0.6 m above the ground (Figure 3.28). The completed roof and wall sections were lifted using the crane and placed on top of the base and hopper. The bottom lip of the bottom ring fit snugly over the outer edge of the hopper cone base ring (Figure 3.29). Self-tapping screws placed every 10 cm were used to attach the silo to the hopper cone

base (Figure 3.30). Sealant was applied to the hopper-sidewall interface on the inside and outside. A hole was cut in the bottom of the hopper cone to accommodate the butterfly valve and  $\text{PH}_3$  reaction chamber housing.



**Figure 3.28** The preassembled hopper cone was placed inside the base support structure and welded to it.

### **Additional equipment on both silos**

A slide gate discharge was included with the SCAFCO silo, but because of the difficulty in sealing the slide gate, the bottom of the silo was modified to use a butterfly valve instead (Figure 3.31). The butterfly valve was custom-made based on the design of the one that came with the Bird's silo.



**Figure 3.29** Self-tapping screws were used to attach the bottom ring of the silo to the hopper cone base.



**Figure 3.30** The author attaching the Bird's silo to its base assembly using self-tapping screws.

On both silos,  $\text{PH}_3$  reaction chambers provided by Bird's were installed beneath the butterfly valve. To make the chamber, an 8 cm deep ring was welded to the hopper bottom piece around the butterfly valve. The operating shaft of the butterfly valve extended through the ring (Figure 3.32). Closed-cell foam sealing tape was placed on the bottom lip of the metal housing.



**Figure 3.31** Butterfly valve installed at the bottom of the silo hopper cone for discharging the grain. The narrow gap around the opening allows fumigant in and out of the  $\text{PH}_3$  reaction chamber installed below (see Figure 32).



A metal bowl with the same diameter as the upper ring clipped onto the housing, creating an airtight  $\text{PH}_3$  reaction chamber with a volume of approximately  $0.3 \text{ m}^3$  (Figure 3.32). A perforated plate was inserted into the bottom of the bowl for placement of  $\text{PH}_3$  fumigant tablets or pellets.

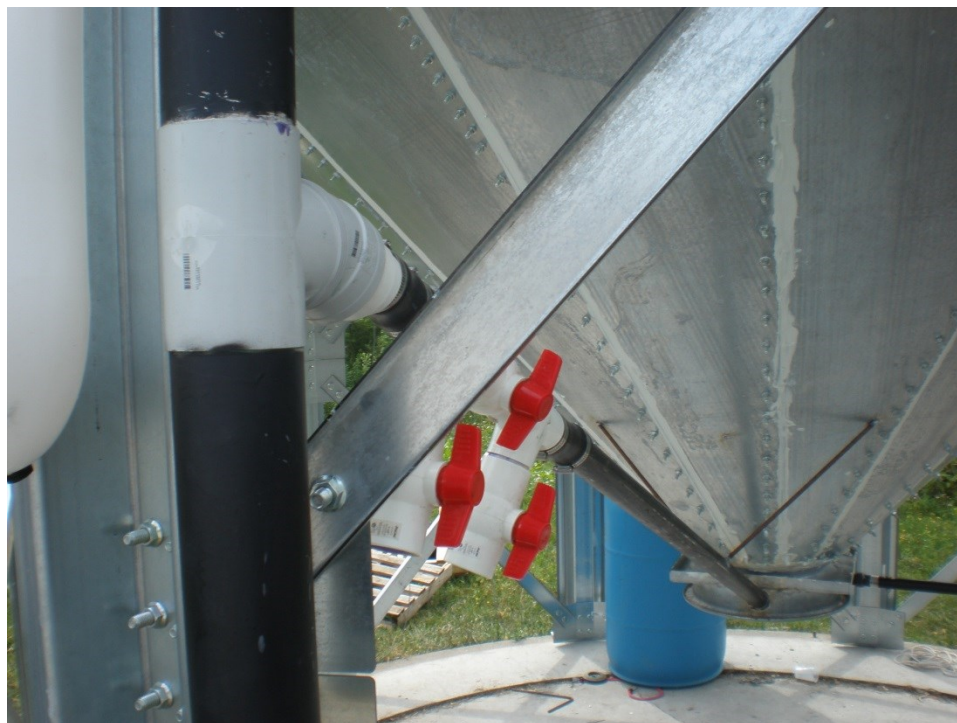


**Figure 3.32** Phosphine reaction chamber installed below the butterfly valve of the hopper bottom silo.

### *Thermosiphon Closed Loop Recirculation System*

To facilitate gas recirculation inside the silo, a closed loop recirculation system was installed. It consisted of a 38 mm diameter pipe connecting the  $\text{PH}_3$  reaction chamber to the 90 mm black PVC thermosiphon pipe near the sidewall-hopper interface (Figure 3.33). The 90 mm thermosiphon pipe was installed at the south side of the silo where it ran upwards to the eave. After making two  $90^\circ$  bends it ran along the roof to approximately 0.5 m from the peak where it connects into the headspace (Figure 3.34). A vertical section extends below the thermosiphon at

the transition to the pressure relief valve. There is an airtight removable cap below this section to remove any condensation that could collect in the thermosiphon. As a result of the gap around the butterfly valve (Figure 3.31), the thermosiphon piping provided a closed loop system in which the air and fumigant mixture inside the silo could be circulated.



**Figure 3.33** Close-up view of the pipe connection between the  $\text{PH}_3$  reaction chamber and the thermosiphon. The ball valve and taps can be utilized for introducing other gases including cylinderized  $\text{PH}_3$  and turning the thermosiphon on or off.

### *Pressure Relief Valve*

An important component of the closed loop recirculation system is the pressure relief valve. A 90 mm white PVC pipe in the shape of a U was connected about 0.5 m above the transition from the small diameter piping to the thermosiphon (Figures 3.3, 3.4 and 3.5). The U-tube was open to the

atmosphere via a 90 mm diameter and 1.5 m long PVC pipe that ends in a 180° bend to prevent rain from entering.



**Figure 3.34** Thermosiphon piping extending along the south roof panel and connecting into the headspace of the Bird's silo.

To seal the pipe from outside air, the bottom of the U is filled with light hydraulic oil to approximately 1 cm above the top bend of the U. Solar heating of the silo wall and roof, barometric pressure changes, and pressurization from applied gases such as cylinderized  $\text{PH}_3$  or  $\text{CO}_2$  can build up pressure inside the silo that may cause structural damage. When pressure increases, air is pushed from the silo into the thermosiphon pipe and through the hydraulic oil in the U-tube to the atmosphere, relieving the pressure difference between the inside and outside of the silo. When pressure decreases, i.e., from barometric pressure swings or from unloading of grain, outside air is drawn through the hydraulic oil of the U-tube into the thermosiphon pipe and ultimately the silo. The pressure relief valve may not be of large enough capacity to equilibrate

the silo pressure during unloading of grain, however. Care should be taken to allow for adequate venting (e.g., by keeping the roof hatch open) to avoid excess suction pressure during grain unloading.

### **Evaluating the gas-tightness of the sealed silos**

Table 2 shows the half-life times from the 13 pressure decay tests conducted. In addition to the initial 7 tests in July and August 2015 after the silos were constructed, each silo was pressure tested before being fumigated. When the oil is level with the top line it indicates an inner silo pressure of 140 to 170 Pa based on measurements taken with the digital manometer. Wind speeds and air temperatures during the tests indicate reasonable conditions. These values are important because of the influence wind and temperature can have on the pressure within the silo (Navarro 1998). Most of the tests were performed under calm conditions (wind speeds of less than about 5 m/s), and during times when solar radiation did not increase the roof and wall temperature of the silo (ambient temperatures below about 25°C). The data collected does not indicate a readily apparent correlation between either wind speed or temperature and the half-life pressure decay times.

Neither silo reached the 3-5-minute minimum half-life pressure decay time prescribed by the Australian silo sealing standard AS2628. For the empty silos, the half-life times for the Bird's silo ranged from 42 seconds to 2 minutes 43 seconds, and for the SCAFCO silo from 0 to 50 seconds. For the partially and completely filled silos, the half-life times for the Bird's silo ranged from 4 seconds to 1 minute 53 seconds, and for the SCAFCO silo from 20 to 46 seconds.

Overall, the Bird's silo showed a better gas-tightness as reflected in the higher half-life times compared to the SCAFCO silo.

The Bird's silo showed a greater loss of gas-tightness over time compared to the SCAFCO silo. After reaching the highest half-life time on August 12, 2015 as a result of additional sealing efforts, once the first set of fumigations took place on August 18, 2015 the half-life times of the Bird's silo decreased by 50-71 seconds (31-44%) while those of the SCAFCO silo decreased by 4-19 seconds (8-38%). As additional fumigations took place in August and November of 2015 and March of 2016, the half-life of the Bird's silo decreased by up to 159 seconds (98%) compared to 30 seconds (60%) for the SCAFCO silo. Between the March and April 2016 pressure tests and additional sealing was performed on the Bird's silo.

These results indicate that stress on the silo from loading and unloading of grain may cause flexing of the silo structure that results in substantial degradation of sealing materials and pressure half-life decay times. This is a concern as silos are utilized for many years and undergo many loading and unloading cycles. Dramatic seasonal weather conditions such as high summer and freezing winter temperatures as well as wind, snow, rain and exposure to solar radiation may also affect the durability of sealing materials, and therefore the gas-tightness of a sealed silo more substantially than anticipated. Resealing a silo on an annual or seasonal basis would add substantial costs, and may prove to be impractical.

**Table 3.2** Half-life times, grain level, wind speed and ambient temperature for the pressure decay tests performed on the Bird's and SCAFCO silos while empty and partially and completely filled.

Date	Bird's Half-life (s)	SCAFCO Half-life (s)	Grain Level	Wind Speed (m/s)	Air Temperature (°C)	Actions performed
<b>2015</b>						
14-July	42	0:00	Empty	2.3	26.9	
15-July	-	0:20	Empty	2.2	21.6	Fix SCAFCO leaks
16-July	66	-	Empty	2.8	27.0	Fix Bird's leaks
16-July	103	-	Empty	3.4	28.6	Fix Bird's leaks
16-July	151	-	Empty	3.7	29.5	Fix Bird's leaks
10-August	-	0:42	Empty	0.9	27.5	Fix SCAFCO leaks
12-August	163	0:50	Empty	0.3	21.6	Fix both silos' leaks
18-August	113	0:31	1/2 Full	2.2	20.7	Filled with grain
18-August	92	0:46	1/2 Full	2.6	20.1	No actions
31-August	31	0:23	Full	0.3	21.3	Filled with grain
25-November	5	0:25	Full	4.2	12.3	No actions
<b>2016</b>						
31-March	15	0:20	Full	6.5	9.6	No actions
25-April	40	0:20	Full	2.2	16.1	Fix Bird's leaks

Pressure testing itself may have had a deleterious effect on the half-life times. If there were areas where the sealant membrane was thin, it is possible that the introduction of high pressure from the vacuum cleaner – which was enough to force air through the pressure relief valve – without sufficient venting, could have compromised one or more of the sealant membranes.

### **Fixing leaks**

As part of the initial pressure tests, leaks were found in both silos. Some leaks were able to be fixed, and others were not. The tests were performed when the silos were empty, half-full, and full of grain. When pressure tests indicated short half-life times, (less than 3 minutes) leaks in the silos were discovered by spraying soapy water on the seams and bolt holes while the silo was under positive pressure. Once discovered, locations were marked and subsequently additional sealant was applied to these areas after thoroughly removing soap residue and drying them.

In the SCAFCO silo there were six major leaks detected along the ridge seams between the roof and wall sections. It is likely that when the already connected roof sections were pulled slightly apart to accommodate the last few roof sections, sealant was pulled apart when the sections were shifted. This must have created gaps through which air could escape. Due to the structure of the roof ribs, these leaks could not be addressed after the roof was constructed because the leak was between overlapping roof sheets and could not be reached.

Thirteen out of the 23 roof rib end points on the SCAFCO silo showed leaks. Additional sealant was applied to the outside of each leaky roof rib clip opposite the foam block, but could not be applied in between the overlapping ribs. The arrows in Figure 3.17 show the area that could not

be effectively sealed because the nozzle of the sealant gun was too wide to fit into the narrow space between roof ribs.

Other leaks detected in the SCAFCO silo were in the hopper-wall junction. There were 15 junctions where three sheets joined (i.e., two wall sheets and one hopper sheet, or two hopper sheets and one wall sheet), and 12 of these had leaks. No leaks were found where only two wall sheets were joined. Several leaks were found around bolts in the hopper, likely where sealant did not get completely around the bolt holes or where the neoprene gasket around the bolt was stripped out because too high of a torque was applied from the electric impact wrench during construction. The time spent on the SCAFCO silo in finding leaks, fixing them, and pressure testing again was about 60 man-hours.

On the Bird's silo, 11 leaks were found in total. Eight of these were on seams or joints where three sheets met. The other leaks were on the vertical seams of the wall sheets. The areas where these leaks were found were cleaned and dried, and more sealant was reapplied to the leak and surrounding seams and rivets. The time spent in finding the leaks, fixing them, and pressure testing again was about 12 man-hours.

If market demands eventually warrant engineering changes in silo design to allow for easier sealing, the roof – and hopper-wall junctions, top lid, and roof – especially the overlapping roof ribs – should be considered areas of greatest concern.



### **Time and labor to construct and seal silos**

Don Bird from Bird's Silos and Shelters and Chris Newman from Stored Grain Services, both from Western Australia, came to Kansas to lend their expertise in constructing the sealed silos. The construction crew ranged from three to eight people, depending on people's availability. The Bird's silo took 10 days from start to finish, and the SCAFCO silo took 26 days from start to finish. This time included unpacking of the containers to erecting and sealing the silos to pressure testing the completed silos. The silos were built concurrently, and had different people working on them on different days. Altogether, the Bird's silo took 266 man-hours and the SCAFCO silo took 359 man-hours to complete.

The polyurethane sealant came in 600 mL "sausage" tubes. Approximately 8 tubes were used on the Bird's silo and 28 were used on the SCAFCO silo. Given the SCAFCO silo was not designed to be sealed for gas-tightness, much more sealant was needed to cover the holes, gaps and seams compared to the Bird's silo.

Table 3 shows the breakdown of effort spent on various aspects of silo construction. Also included are the estimated times it would take experienced workers to construct each silo. Much more time was spent constructing the SCAFCO roof and hopper than what was needed for the Bird's, largely due to the effort that went into sealing. The Bird's silo required more time for assembling the sidewalls, as each ring was challenging to fit onto the one below it. Much time was spent aligning the rings before connecting them with rivets.

**Table 3.3** Effort spent in man-hours to construct each silo (including sealing) compared with the base time it would take experienced workers to build the same silo.

	Roof and Lid	Hopper / Base	Sidewalls	Assembly	Fixing leaks	Miscellaneous	Total
Bird's	48	60	60	66	12	20	266
SCAFCO	90	128	30	31	60	20	359
Bird's (Factory)	-	-	-	-	-	-	53
SCAFCO (experienced work crew)	-	-	-	-	-	-	96

### **Bird's silo**

It takes a work crew of about 53 man-hours to assemble a silo at the Bird's factory (Don Bird, personal communication). Extra time was needed for the construction of the silo in this study because the wall, roof and hopper sheets were cut in pieces prior to shipment and had to be welded on-site. Because the Bird's silo was designed to be sealed, the only "sealing time" parsed out of the following time and labor figures is the time spent finding and fixing leaks.

The roof took about 48 man-hours to piece together, weld, seal, and install the ground-opening airtight lid. The hopper and base assembly took approximately 60 man-hours to assemble and weld. Sixty man-hours were spent on putting the sidewalls together. To assemble the roof, sidewall rings, and hopper sections took 66 man-hours. Actual construction time was therefore 234 man-hours. Approximately 12 man-hours were spent finding, fixing, and re-testing leaks. The additional 20 man-hours from the total time spent was accounted for by people waiting for work, planning next steps, waiting for parts, and other activities.

Eight tubes of sealant were used to seal the Bird's silo, but this cost would already be figured into the list price of the silo as ordered from the factory, and the same is true for the airtight lid, thermosiphon, PH<sub>3</sub> reaction chamber, and pressure relief valve.

### **SCAFCO silo**

The fact that the SCAFCO silo used bolts rather than rivets made it more difficult to seal. The rivets used on the Bird's silo were highly airtight, and there were fewer of them. The neoprene washers on the bolts often shredded due to overtightening as the sheets were tightened together. The roof and hopper of the SCAFCO were major leak areas, whereas the Bird's silo used single welded sheets which could not leak. In the end, more than three times the amount of polyurethane sealant was necessary to seal and reseal the SCAFCO silo.

An experienced work crew of four could erect the same model SCAFCO silo in 2.5 to 3 days (20 to 24 hours), or 80 to 96 man-hours (Regan Heaton, personal communication). The extra time it took for sealing the SCAFCO silo was substantial. The roof, including sealing between the roof ribs and end gaps, and installing the modified sealed lid took approximately 90 man-hours to complete. One work day was spent adjusting the roof to get it to fit on the top sidewall ring. This could have been avoided had the roof sheets been attached to the top of the sidewall ring during construction as specified in the owner manual.

Approximately four hours were spent in fabricating the metal flashings that were installed to seal the ridge caps. These were made from scrap metal, but purchased new the sheet metal would be around \$10. Approximately 66% of the time, or about 60 man-hours were spent on sealing the

roof in addition to installing the sealing kit that was included with the silo. The sealed lid with remote ground-level opening has a list price of \$350. The ground-level opening device was not installed on SCAFCO silo as the slope of the roof prevented its full range of operation.

The sidewalls took only slightly more time to assemble than an unsealed silo would have. In a stock version silo, rope bead sealant is supposed to be applied between sheets. For this silo, extra care was taken to clean each seam with alcohol and encircle every bolt hole with sealant. It took about 30 man-hours to assemble the sidewalls, including the sealing which added up to an estimated four man-hours total.

For the hopper section, sealing the seams between adjacent hopper sheets took about four man-hours. Sealing the junction between the hopper and sidewall took more effort because of the ungainly use of the ladder required to reach the junction from inside the silo. This process took approximately 10 man-hours. Altogether, the time spent in sealing the hopper section was about 14 man-hours, and took a total time of 128 man-hours. This time included attaching the legs to the silo and anchoring them in the concrete pad.

Assembling the roof, wall, and hopper sections together took approximately 31 man-hours. Fabricating the butterfly valve outlet (list price \$244) and installing it took approximately 10 hours. This is about five times the amount of time it would take to assemble and install the rack and pinion gate outlet supplied by SCAFCO.

The thermosiphon and pressure relief valve equipment took approximately 10 man-hours to install and paint, and lists for \$350. The sealant used was negligible, but PVC cleaner, primer, and cement to assemble the piping cost approximately \$20.

Twenty eight tubes of sealant were used on the SCAFECO silo; 11 tubes on the roof, 7 on the sidewalls, and 10 on the hopper. The list price of the sealant is \$30, for a total of about \$840 for sealant.

Table 4 shows the breakdown of costs for constructing and sealing the SCAFECO and Bird's silos. Assuming a wage of \$15 per hour, the labor that went into sealing was \$1,470, or about 30.6% of the total cost of labor. For the SCAFECO silo, the costs of sealing including equipment, materials and labor totaled \$3,284. The total sealing cost represents approximately 25.7% of the cost of the SCAFECO hopper silo which has a list price of \$13,500.

Because the Bird's silo was designed as sealed, the extra sealing costs are only labor costs to find, fix, and re-test for leaks. This overall cost of \$180 is less than one percent of the total silo list price of \$7,200.

Assuming the proportion of sealing time to total construction time to be about 27% (98 man-hours to 359 man-hours for the SCAFECO silo), and estimating 96 man-hours for an experienced crew to construct a similarly sized silo, the estimate for an experienced crew to seal the silo is about 26 man-hours. The labor cost for sealing was reduced to 21.4% of the total cost of labor because during construction of the SCAFECO silo for this project, much time was spent fixing

mistakes, re-applying sealant, and otherwise spending more time than an experienced crew would on sealing. Assuming the same amount of sealing materials, the total sealing expense for an experienced crew was reduced from \$3,284 to \$2,207, or 16.3% of the total list price of \$13,500.

In 1984 Banks and Ripp (1984) estimated the average cost to be approximately A\$20 per MT for sealing a bolted steel silo and A\$8 per MT for adding the needed modifications for fumigation (i.e., recirculation ductwork, pressure relief valves and exhaust fans). This cost included labor and materials for retro sealing of silos already constructed but not yet sealed. Accounting for inflation and converting to U.S. dollars, the total amount would be \$60/MT (RBA 2016). In this study, the cost of sealing the SCAFCO silo while being constructed including resealing as a result of finding leaks during pressure testing was \$63/MT. For an experienced crew this may be reduced to \$43/MT. In comparison, the cost of sealing (and resealing) the Bird's silo, which was designed to be a sealed silo was \$4/MT. These costs are similar to the costs estimated by Banks and Ripp (1984). For larger storage structures such as concrete silos (up to 2,700 MT) and flat storage bunkers (up to 300,000 MT), Banks and Ripp estimated lower sealing and modification costs on a per-MT basis, ranging from \$5/MT to \$15/MT. It is likely that for larger metal silos there would be similar cost reduction for sealing and modifying for fumigation.

Temporarily sealing a silo like the ones in this project is relatively inexpensive (approximately \$50 for labor and materials). And even sealing larger silos (5000+ MT) would only cost slightly more (less than \$200 for labor and materials (Dolan Jamison, personal communication)).

**Table 3.4** Labor and material costs for constructing and sealing the silos, with comparative figures for temporarily sealing an existing silo prior to fumigation

	SCAFCO	Bird's	SCAFCO (Experienced work crew)	Temporary sealing (existing silo)
Silo list price	\$13,500	\$7,200	\$13,500	-
Labor cost for construction @ \$15/hr.	\$3,735	\$3,810	\$1,440	n/a
Labor cost for sealing @ \$15/hr.	\$1,470	\$180	\$393	\$30
Total labor cost@ \$15/hr.	\$5,205	\$3,990	\$1,833	\$30
Materials and equipment cost for sealing	\$1,814	n/a	\$1,814	\$50
All sealing expenses	\$3,284	\$180	\$2,207	\$80
Total cost of silo including construction and sealing	\$20,519	\$11,370	\$17,147	
Proportion of sealing labor of total labor cost	28.2%	4.5%	21.4%	100%
Proportion of sealing materials of silo list price	13.4%	n/a	13.4%	0.37%
Proportion of all sealing expenses of silo list price	24.3%	2.5%	16.3%	0.60%

A grain manager must decide whether the cost of permanently sealing a grain silo is worth the potential gains in fumigation success, as lower long-term labor costs would not likely be realized for many years. A hybrid approach may be to permanently seal areas with greater leakage potential such as the roof-wall junction, and use temporary sealing for vents, fans, and other areas that are not conducive to being permanently sealed.

### Conclusions

This study documented and evaluated the effort, costs and feasibility of sealing two hopper bottom metal silos while being constructed. The Australian silo was designed as a sealed silo and the U.S. silo was a conventional open-eave design. The two sealed silos were equipped with closed-loop fumigant recirculation systems and subsequently tested for gas tightness. The following was concluded:

- Neither silo had half-life pressure decay times that reached the 3-5 minute prescribed by AS2628. The longest half-life times were 50 seconds and 2 minutes 43 seconds for the SCAFCO and Bird's silos, respectively. The half-life times were shorter after loading the silos with grain, suggesting the pressures from grain loading may have broken some of the seals and opened paths for air to travel through the silo wall seams.
- After nine months the pressure decay times decreased by up to 38 to 44% for the SCAFCO and Bird's silos, respectively, through hot summer and cold winter weather. It is possible that exposure to temperatures above 35° C and below 0° C as experienced in Kansas, along with high winds and solar radiation deteriorated some of the sealant exposed to the elements.
- Permanently sealing a corrugated steel silo after-market is time consuming and labor intensive. The additional time spent in sealing, checking for and fixing leaks and installing the additional fumigation equipment was 98 man-hours. An experienced crew may be able to reduce this to about 26 man-hours for a similar sized silo.
- Additional needed equipment and sealing materials may add substantial cost to the silo. The sealed lid, thermosiphon, butterfly valve, pressure relief valve, polyurethane sealant, and metal flashing cost a total of \$1,814 or \$35/MT which represents about 13.4% of the initial cost of the SCAFCO silo.
- The total cost of modifying and sealing a silo during construction to prepare it for fumigation on a permanent basis was estimated to be \$63/MT, or 24.3% of the total list price. For an experienced work crew that cost may be lowered to \$43/MT, or 16.3% of the list price. A considerable amount of money may be saved in the short term by



temporarily sealing the silo prior to each fumigation which for an experienced work crew may cost only \$0.91/MT, or 0.37% of the list price.

## **Chapter 4 - Efficacy of Recirculation Fumigation in Sealed**

### **Australian and U.S. Steel Hopper Grain Silos**

#### **Introduction**

Reducing the amount of grain lost after harvest is an important strategy to fight hunger and poverty. Kader (2004) estimated that nearly one third of the total grain harvested worldwide is lost before consumption or sale. Insect pests are among the biggest contributors to post harvest losses. Insects destroy grain both by consuming it and by spoiling it with their filth. Insect activity and the presence of mold can raise the temperature of the grain to the point where the grain is ruined. In some cases, fires have been started in grain silos as a result of “hot spots” in the grain (Clark et al. 1998). Because of the close positive relationship between insects and the factors that contribute to poor grain quality, controlling insect infestations goes a long way in preserving the grain quality.

Fumigation is the addition of toxic gas to the inside of a stored grain structure to kill insect pests. The goal of fumigation is to kill all insects at all life stages. For a fumigation to be successful, a lethal concentration of the gas must be held for a minimum amount of time at an appropriate temperature. A common measure of this is known as the concentration time product (CTP). When the fumigation is complete, fresh air is forced through the grain to remove the fumigant.

A tightly sealed structure is a prerequisite for a successful fumigation. Fumigation in unsealed or leaky structures is likely the main cause of fumigation failures which leads to populations of

resistant insects (Leesch et al. 1995, Casada and Noyes 2001). In unsealed structures the fumigant leaks out and does not maintain a lethal concentration of gas. This creates selection pressure against susceptible individuals, and selection pressure for individuals carrying genes which confer resistance to the fumigant (Schlipalius et al. 2008, Collins et al. 2002).

In the early 1960's, export regulations in Australia required all exported grain to be free of injurious insect pests (van Graver and Winks 1994). Grain pests were controlled with fumigations and pesticide applications, but further export restrictions that limited chemical residues in grain began the grain industry's search for non-chemical means of insect control (Newman et al. 2006).

In the 1970s, a revival of sealed grain storage research began in Australia. Researchers developed methods of sealing existing grain storage structures and effective sealing standards (Banks and Annis 1981; Banks and Ripp 1983), led extension efforts to educate farmers about sealed grain storage and responsible management (Delmenico 1993; Chantler 1983), and worked with silo manufacturers to engineer more readily sealable silos (Ellis 1983).

The vast majority of U.S. on- and off-farm grain storage silos are not designed to be sealed for adequate levels of gas-tightness. Instead, they have to be sealed temporarily before fumigation which adds substantial labor and material costs and results in greater risk of fumigation failures from inadequate sealing than with silos sealed by design.

## **Fumigation**

Many fumigants have lost their approved use status because of their negative environmental impact or because of toxicity and safety concerns (Bell 2002; Mills 2000). Phosphine (PH<sub>3</sub>) gas is the most common fumigant used in the grain industry worldwide. Phosphine is formulated as solid pellets of aluminum or magnesium phosphide which sublime upon contact with moisture in the atmosphere. In gaseous form, it is also available in cylinderized containers. Phosphine tablets or pellets can be placed in the grain stream as the silo is being filled, placed on top of the grain or hung in the headspace in sachets, deep-probed into the grain mass from the surface, or applied at ground level using a separate chamber connected to the structure.

Phosphine is relatively inexpensive, easy to obtain, and easy to use. However, its effectiveness is under threat due to widespread phosphine resistant insect populations that have developed around the world including the U.S. (Collins 1998, Opit et al. 2012). Australia is the only nation with a country-wide phosphine resistance program (Subramanyam and Hagstrum 2011), and sealed storage fumigation is the backbone of this program. Efforts to develop sealed grain storage in Western Australia have kept PH<sub>3</sub> resistance in check (Delmenico 1993). Monitoring programs in Australia are in place to determine the extent and severity of resistant populations, but no such survey work exists in the U.S. (Opit et al. 2012), though it has been called for in the past (Zettler and Cuperus 1990).

## **Recirculation**

Gas distribution within the storage structure is important to consider when treating stored grain with a fumigant. Stored grain insect pests can be found throughout the grain bulk. For this

reason, lethal fumigant concentrations should reach all areas inside the structure. With poor distribution there may be areas of low fumigant concentrations in the grain mass, allowing insects in those locations to survive.

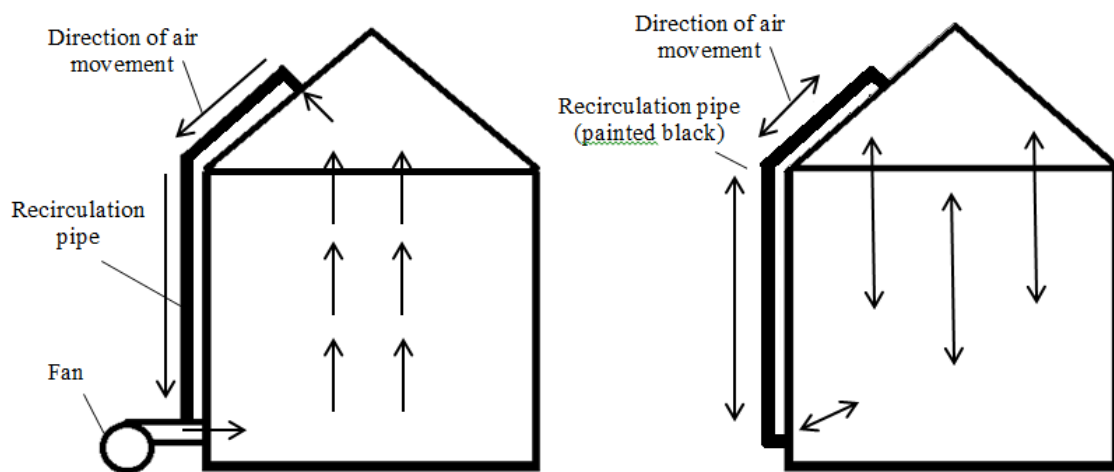
Dispersing the gas throughout the grain mass can be carried out in several ways. Pixton and Griffiths (1971) stated that the primary mechanism for moisture transfer within a grain mass was diffusion, but supplemented by convection. Heat and moisture in the form of water vapor continually move throughout the grain mass during storage. Moisture is transferred from high to low vapor pressure areas due to differing moisture contents in the grain being stored (Obaldo et al. 1991). Due to the relationship between temperature and vapor pressure, temperature gradients also drive moisture movement. The movement takes place more rapidly between areas with higher moisture and temperature gradients (Jayas 1995), but is nevertheless very slow (between 0.08 and 2.54 m per hour) even at temperature gradients of up to 25° C (Berck 1975).

Solar radiation on the metal wall of the silo creates interstitial air temperature gradients within the porous grain mass that can differ by an order of magnitude, even in small silos (Jian et al. 2009). This occurs because air warms faster than grain due to its higher thermal conductivity and lower heat capacity. The temperature gradients in turn produce convection currents due to the lower density of warm air.

Temperature gradients within the grain mass can vary at different locations within the silo because different sides of the silo receive different amounts of solar radiation during the day due to the sun's movement overhead, cloud cover, and shading from other silos or structures (Jian et al. 2009). Moisture gradients within the grain bulk also vary due either to moisture migration over time (Chang et al. 1994) or loading the silo with grain of different moisture contents. Thus,

the driving forces of natural heat and mass transfer within the grain mass are asymmetrical in the vertical, radial, and circumferential directions (Alagusundaram 1990).

Figure 4.1 shows two recirculation designs. The left image uses a low speed fan to force air and fumigant through the grain and out through a pipe connected to the roof of the silo. The right image shows a recirculation design based on natural convection currents. The pipe on the outside of the silo is painted black. When the sun shines on the black pipe, the air inside the pipe becomes warmer than the air within the grain mass. This temperature differential creates a convection current in which the warm, less dense air in the pipe is drawn upwards. Air in the bottom of the grain mass is pulled into the pipe, which is then warmed. This circulation distributes the fumigant throughout the structure. In contrast to forced air recirculation, this design allows air to reverse directions when the air in the thermosiphon pipe cools down in cloudy conditions or at night. In both cases the silo is sealed to the outside and the gas is distributed only through the closed loop.



**Figure 4.1.** Closed loop fumigation (CLF) with forced air circulation and convection based recirculation with thermosiphon.

### *Fumigant loss*

Often, the actual fumigant concentration does not reach the theoretical concentration expected because of fumigant loss. Banks and Annis (1984) stated that the major reasons for fumigation failures are overall fumigant loss and inadequate CTP in infested areas, either due to an inadequate overall dosage or a delay in fumigant reaching insect infested areas.

Several processes prevent gas concentrations from reaching their theoretical levels during fumigation, including sorption and various mechanisms of physical gas loss from the structure itself. In this context, sorption refers to loss of gas applied to a stored commodity that is not due to leaks in the storage structure. Rauscher et al. (1972) showed that  $\text{PH}_3$  was physically, and not chemically, adsorbed onto wheat flour, wheat bran, and oat flakes, and could be recovered with sufficient aeration and movement of the commodity. Reed and Pan (2000) suggested  $\text{PH}_3$  molecules find their way into grain kernels via diffusion along concentration gradients and undergo chemical reactions with grain constituents (lipids, protein) which detoxify the gas. Internally developing insects are exposed to the fumigant by the sorption of the gas into grain kernels prior to its detoxification. Fumigants can react with grain constituents and be lost in this manner as well (Banks 1986). At higher temperatures, relative humidity, and grain moisture contents, the sorption rate of  $\text{PH}_3$  is higher. Sorption rate of  $\text{PH}_3$  is between 5-20% per day in wheat (Banks 1986).

Fumigant loss also occurs from gas transfer from the inside to the outside of the structure.

Barometric pressure changes and wind cause pressure gradients across leaks in the fabric of the storage structure, and temperature differences between the outside ambient air and the air inside

the storage structure cause air exchange through the stack, or chimney, effect (Banks 1986). Ingress of outside air can dilute fumigant concentration within the structure (Banks 1989). Fumigating in sealed grain silos may benefit grain producers and handlers in warmer regions (such as the southern U.S.) where normal temperatures severely limit grain cooling options with ambient aeration alone because treating grain in sealed storage can preserve grain quality without causing excessive moisture shrink.

The overall goal of this project was to evaluate the feasibility of sealed silos for fumigation under U.S. (Kansas) conditions. Assessments included fumigation trials with  $\text{PH}_3$  and sulfuryl fluoride (SF), stored-grain insect pest control, and overall stored grain quality. Efforts focused on minimizing gas loss during fumigation in a U.S. bolted corrugated steel hopper silo. These results were compared with an Australian-design steel hopper silo that was used as the benchmark. In addition, the Australian pressure decay sealing standard (AS2628) and thermosiphon recirculation technology were evaluated.

## **Methods and Materials**

### **Grain silos used**

A 45.6 MT capacity sealed grain hopper silo from Australia (Bird's Silos & Shelters, Popanyinning, Western Australia) was shipped to Manhattan, Kansas and erected on site. A 51.8 MT capacity corrugated steel hopper silo typical to the U.S. (SCAFCO Grain Systems, Spokane, Washington) was also shipped to Manhattan, Kansas and erected on site.



The SCAFCO silo was not engineered to be airtight, but a sealing kit was included with the silo that contained a rubber gasket to be installed between the roof and top wall sheets, and foam blocks designed to close off the gap created by the roof ribs (Figure 4.2). The sealing kit was not designed to make the silo airtight, but to prevent excessive moisture from entering the silo. Nevertheless, the foam blocks helped close off gaps too large to be sealed with polyurethane sealant.



**Figure 4.2.** Rubber gasket attached between roof and edge of top wall sheet (left), and closed-cell foam block closing large gap at the end of the roof rib (right).

### **Fumigant recirculation**

Each silo was equipped with a ThermoSiphon to circulate and disperse the fumigant within the silo. The ThermoSiphon attached to the silo externally. The ThermoSiphon consisted of 38 mm steel pipe that followed the hopper slope upwards to the silo wall where it transitioned to a black 90 mm PVC pipe. The PVC pipe went vertically up the silo wall and continued along the roof slope where it opened into the headspace approximately 0.5 m from the peak (Figure 4.3). There was no forced air in the ThermoSiphon to circulate the fumigant, rather, it relied on convection

currents created when solar radiation caused temperature differentials between the air inside the black pipe and the air inside the silo.



**Figure 4.3.** Showing the ThermoSiphon recirculation pipe connecting the  $\text{PH}_3$  reaction chamber below the hopper (left) and the headspace (right).

The velocity of air inside the thermosiphon was measured using a hot wire anemometer during sunny and partly cloudy conditions, and at various ambient temperatures. The volumetric airflow rate was calculated by multiplying the air velocity by the area of the 90 mm thermosiphon pipe.

### **Fumigant application**

The grain was held inside the silo above a butterfly valve. Phosphine reaction chambers were installed on the bottom of each silo. The chambers consisted of a bowl that was clamped to the butterfly valve housing to make an airtight seal (Figure 4.4). A grate was placed inside the bowl to increase exposure of phosphine tablets or pellets to air. The fumigant exited the chamber in two ways, upward into the grain mass through the gap between the butterfly valve and its housing, and through the ThermoSiphon pipe (Figure 4.5).



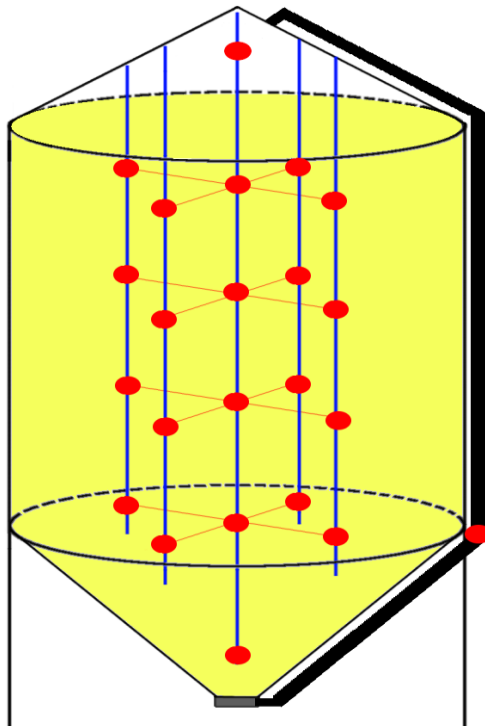
**Figure 4.4.** Airtight  $\text{PH}_3$  reaction chamber installed on the underside of the hopper bottom.



**Figure 4.5.** Butterfly valve and exit points for fumigant inside  $\text{PH}_3$  reaction chamber.

### Measuring fumigant concentration

To measure phosphine concentrations, 3 mm diameter plastic monitoring lines were attached to aircraft cable using zip ties and hung vertically in the silo. The cables were hung in the center and approximately 1 m from the silo sidewall in the north, south, east, and west directions. The north, south, east, and west lines had four readings each, terminating in the SCAFCO silo at 1.8, 2.8, 3.8, 5 m from the top of the grain surface. The center line had an additional reading at 6.1 m. In the Bird's silo the readings terminated at 1.4, 2.6, 3.8, and 4.5 m, with an additional reading on the center line at 5.1 m. Monitoring lines were also set up in the headspace and in the bottom of the ThermoSiphon pipe near the hopper/sidewall junction. Each silo had a total of 23 reading points (Figure 4.6).



**Figure 4.6.** Gas concentrations were measured from twenty three sampling points per silo (indicated by dots).

The monitoring lines exited the top of the silo through an elbow fitting and ran down the side of the silo to a table where the gas measurements were recorded. The elbow fitting was filled with closed cell expanding foam and the gaps between lines were sealed using an all-weather silicone sealant (Figure 4.7).



**Figure 4.7.** Monitoring lines exiting silo roof. The fitting was filled with closed-cell expanding foam and sealed with silicone caulk around the lines.

Phosphine concentrations were read using a Draeger X-am 5000 (Dräger, Inc., Lübeck, Germany) personal monitoring device using a Draeger X-am 1/2/5000 pump to draw the gas from the grain mass through the lines to the gas sensor. For fumigation trials 1-3, readings were taken approximately hourly from morning to for the first 72 hours of the fumigation, and then at intervals from 2-4 hours after that. A portable wet/dry vacuum cleaner was used to purge the

lines and draw gas samples through the lines more quickly before each reading. This resulted in an expedited measurement and recording process.

In trials 4 and 5, SF concentrations were read using a Spectros SF-ExplorIR (Spectros Instruments, Hopedale, MA) non-dispersive IR instrument. Readings were taken two or three times per day throughout the fumigation. An onboard pump in the instrument drew the gas through the lines.

Throughout trials 1 and 5, PH<sub>3</sub> readings were also taken outside the silos to check for leaks into the surrounding space using the handheld Draeger X-am 5000 personal monitoring device and X-am 1/2/5000 pump. Concentration readings were taken on the north, south, east, and west directions of the silos at distances of 0, 2, and 6 m.

### **Bioassays**

Insect bioassays were prepared to demonstrate the efficacy of two fumigations. Due to time and resource constraints, bioassays were prepared for fumigation trials 1 and 5 only. Centrifuge tubes of 50 mL (30 mm x 115 mm) were drilled at the top and bottom and fine brass mesh was glued to cover the end openings.

For trial 1, 20 unsexed adult *Sitophilus zeamais* were placed in a tube with whole corn, and 50 unsexed adult *Tribolium castaneum* were placed in a tube with 50% whole corn and 50% ground corn. Fifteen tubes with each species were prepared, five for each silo and five for control. After the initial preparation, the tubes were kept under controlled conditions (approximately 25° C,

66% R.H.) for 5 days to allow females to lay eggs within the tubes. One bioassay tube of each species was put inside a small burlap bag. Five of these bags were prepared and each one was placed 45 cm below the grain surface directly next to one of the five top-level monitoring line points. After five days under fumigation, the bioassays were retrieved and kept indoors for 24 hours. Live and dead adults were counted and separated from the food, which was recollected and placed in glass jars with screen tops. *S. zeamais* were sieved through a #12 screen and the *T. castaneum* were sieved using #12 and #18 screens above a pan. The jars with food were kept at 25° C and 66 % R.H. for six weeks to allow adult insects to emerge.

In fumigation trial 5, PH<sub>3</sub>-resistant *R. dominica* (Belle Glade, Florida) and *T. castaneum* (Red Level, Alabama) were included in the bioassays. Both strains were characterized as PH<sub>3</sub>-resistant by the FAO discriminating dose assay (*R. dominica* 87% and *T. castaneum* 100% resistant). PH<sub>3</sub>-susceptible lab populations of the same species were also used in the bioassays. Fifty unsexed adult PH<sub>3</sub>-resistant and thirty unsexed adult PH<sub>3</sub>-susceptible *R. dominica* were placed in a tube with 90% whole wheat flour and 10% mixture of flour and yeast. Fifty unsexed adult PH<sub>3</sub>-resistant and thirty PH<sub>3</sub>-susceptible *T. castaneum* were placed into a tube with 50% whole wheat kernels and 50% cracked wheat. Fifteen tubes of each species, with both PH<sub>3</sub>-resistant and PH<sub>3</sub>-susceptible populations were prepared and kept at 25° C and 66% R.H. for 7 days to allow females to lay eggs. The bioassays were placed in the silo in the same manner as in trial 1. After ten days the top lid and PH<sub>3</sub> reaction chamber were opened on the SCAFCO silo (under PH<sub>3</sub>). After fifteen days the Bird's silo (under SF) was opened and vented. A blower fan was placed beneath the silo to draw fresh air through the grain to vent the fumigants out. The bioassays were counted and processed post-fumigation in the same manner as in trial 1.

## **Fumigations**

The first three fumigation trials had a target of 300 ppm for 72 hours resulting in a CTP of 21,600 ppm-hours. This target was based on the minimum exposure time but the application rate was lower than would be used in the industry. Due to the risk of explosion at PH<sub>3</sub> concentrations above 17,000 ppm and the small reaction chamber used, trials were performed with lower doses first to ensure the fumigant would disperse readily and stay below explosive levels. Trials 1 and 3 utilized aluminum phosphide tablets. Each tablet weighed 5 grams and produced one gram of PH<sub>3</sub>. Trial 2 utilized aluminum phosphide pellets. Each pellet weighed 1 gram and produced 0.2 grams of PH<sub>3</sub>. In trials 4 and 5, cylinderized sulfuryl fluoride and PH<sub>3</sub> were used.

The first fumigation trial began on August 24, 2015. Aluminum phosphide tablets were placed in the reaction chamber of each silo. Readings were taken from approximately 6:00 am. to midnight hourly for the first 72 hours, and every two to four hours after that. At the end of the trial a final reading was taken on August 29 at approximately 4 pm (6 days total). Afterwards, the silos were ventilated by opening the top lid and removing the PH<sub>3</sub> reaction chamber.

The second fumigation trial began on August 31, 2015 at 12:00 pm. Aluminum phosphide pellets were utilized to provide the same concentration as in trial 1. Readings were taken approximately 10 times per day for four days, then once every other day until September 9 (10 days total).

Fumigation trial 3 began September 18 at 10:00 am. Phosphine tablets were used at the same rate as in the previous two trials. In this trial, the ThermoSiphon on the SCAFCO silo was closed using a ball valve. This forced the fumigant to permeate upwards through the grain and distribute throughout the grain mass without the benefit of recirculation. The ThermoSiphon on the Bird's



silo remained open. Readings were taken approximately 10 times per day for the first four days, then once daily until September 24 (7 days total).

For trial 4, VAPORPH<sub>3</sub>OS® (Cytec Industries Inc., Woodland Park, NJ) cylinderized PH<sub>3</sub> was used in the Bird's silo. The cylinder contained pure PH<sub>3</sub> which was blended with ambient air using a Horn Diluphos System® (Fosfoquim S.A., Santiago, Chile) (Figure 4.8).



**Figure 4.8.** Horn Diluphos System used to blend PH<sub>3</sub> and air prior to silo fumigation during trial 4.

The PH<sub>3</sub>-air mixture was applied to the Bird's silo via the ThermoSiphon and directed to the headspace (Figure 4.9). The system supplied a total of 204.9 g for a target concentration of approximately 2000 ppm. Sulfuryl fluoride (ProFume®, Dow AgroSciences LLC, Indianapolis, IN) was used in the SCAFCO silo. The ProFume® Fumiguide® computer program was used to calculate dose and target CTP based on target species (*R. dominica*), life stage (eggs and pupae),

temperature (15.5° C), and exposure time (3 days). The calculated dose was 1,360 g or 4,541 ppm in the size of silos used. For a fumigation of three days, this provided a CTP of 107,821 g-hours, which is near the maximum allowable CTP for the size of silo used (107,884 g-hr.). The cylinder of SF was connected directly to a monitoring line (center line, 3.8 m below the grain surface) to allow the SF to flow into the grain mass. The cylinder was set on a digital scale and fumigant was metered out in real time by observing weight loss on the scale (Figure 4.10). The reason the cylinder of SF was connected to the monitoring line instead of the 4-way valve as the PH<sub>3</sub> cylinder was to avoid leaks as the gas line connection did not fit snugly into the 4-way valve.

For trial 5, SF was applied to the Bird's silo in the same manner as in trial 4. In the SCAFCO silo, 212 g of PH<sub>3</sub> was applied via the thermosiphon into the headspace of the silo using VAPORPH<sub>3</sub>OS® for the same target concentration as in trial 4.



**Figure 4.9.** Attaching the VAPORPH<sub>3</sub>OS® to the ThermoSiphon. The gas was directed to the headspace using a valve on the ThermoSiphon.



**Figure 4.10** The amount of SF applied to the silo was measured by placing the cylinder on a digital scale, opening the cylinder valve to allow gas to flow, and closing the valve when the correct amount of SF had been applied.

### **Grain quality**

Grain moisture content and test weight of the corn were measured at the beginning of the storage period and after 2, 6, 8, and 10 months. A GAC-2500-UGMA (DICKEY-john Corporation, Minneapolis, MN) grain analysis computer was used for the measurements.

Before the first fumigation of 2016, in mid-March, insect probe traps were placed in both silos at the top of the grain in the center and in the cardinal directions approximately 1 m from the

sidewall. Prior to the fumigation starting April 1, the probe traps were recovered and the insects trapped were counted.

### **Data analysis**

The concentrations among the five points (center, north, south, east, and west) at each monitoring level, and the concentrations among the points within the grain mass along each vertical monitoring line were analyzed to quantify the movement and distribution uniformity of phosphine in the silos.

The leakage rates were calculated using the following equation:

$$L = \frac{C_{\text{peak}} - C_{125}}{125 \text{ hours}} \quad (4.1)$$

Where,

L = gas leakage rate, ppm/h

$C_{\text{peak}}$  = peak gas concentration

$C_{125}$  = gas concentration at 125 hours.

Because some fumigation trials were monitored longer than others, the leakage rates were calculated over 125 hours to have a standard measure.

The half-loss time (HLT) is the number of hours it took for the peak concentration to decrease by 50%. For example, in trial 1, the Bird's silo peaked at approximately 400 ppm after 32 hours.

The concentration dropped to 200 ppm after 76 hours which resulted in an HLT of 44 hours (76h – 32h = 44h).

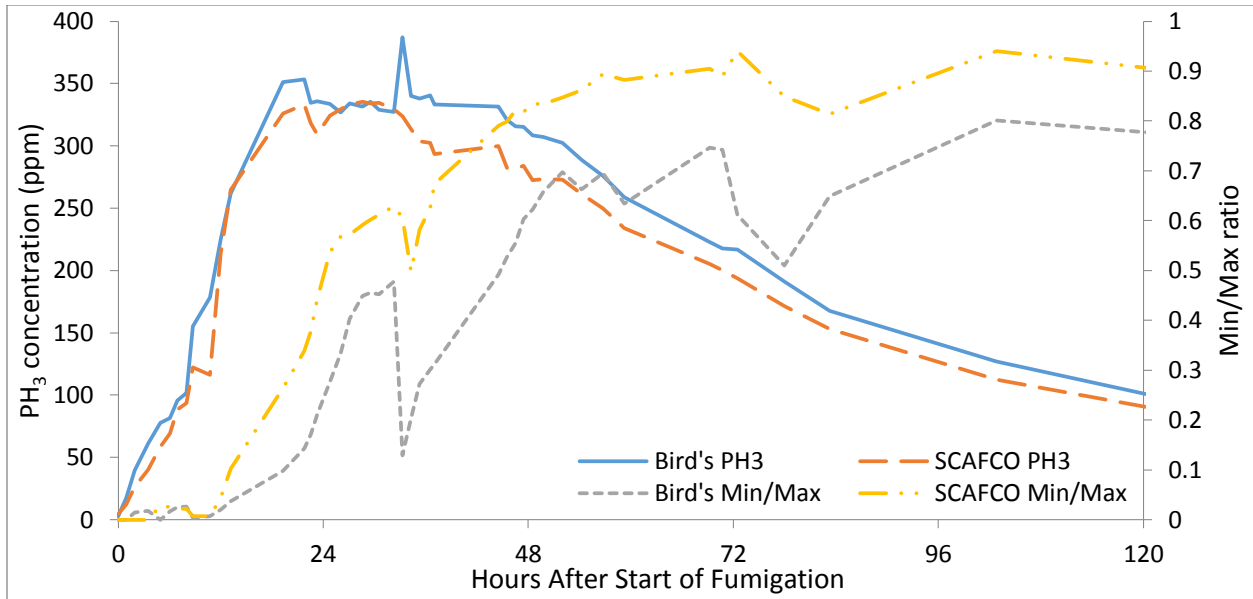
Banks and Annis (1984) developed criteria for judging a successful fumigation. Namely, (1) that the grain be insect-free after fumigation, (2) the average maximum concentration be greater than 50% of the prescribed dosage, (3) the average concentration at the end of the fumigation be greater than the minimum effective dose to kill all insects, and (4) the ratio of minimum to maximum concentration within the storage structure exceed 0.25 before 25% of the fumigation period. Criteria 1-3 reflect the ability of the structure to maintain lethal gas concentration, and criterion 4 refers to the amount of gas dispersion within the structure. For trials 1 and 5, the bioassay results (criterion 1) were used to judge efficacy of the fumigations. The average maximum concentration (criterion 2) was calculated by taking the mean of the maximum fumigant concentration in the silos at each sampling time. For criterion 3, the endpoint of the fumigations was considered to be 125 h. However, the actual endpoint of all the fumigations except trial 1 extended past 125 h to determine how long the fumigant would be retained. The minimum effective dose for both PH<sub>3</sub> and SF depend on the duration of fumigation. For PH<sub>3</sub>, Banks and Annis used a rate of 0.01 g/m<sup>3</sup> or approximately 7 ppm, and for SF the minimum effective dose was considered to be 2,840 ppm, the concentration allowable for a 125 h fumigation given the 1,500 g h/m<sup>3</sup> maximum allowable CTP. The rate of gas dispersion (criterion 4) was calculated by dividing the minimum concentration in the silo by the maximum concentration at every sampling time. This ratio indicated the level of concentration uniformity within the silos.

## Results and Discussion

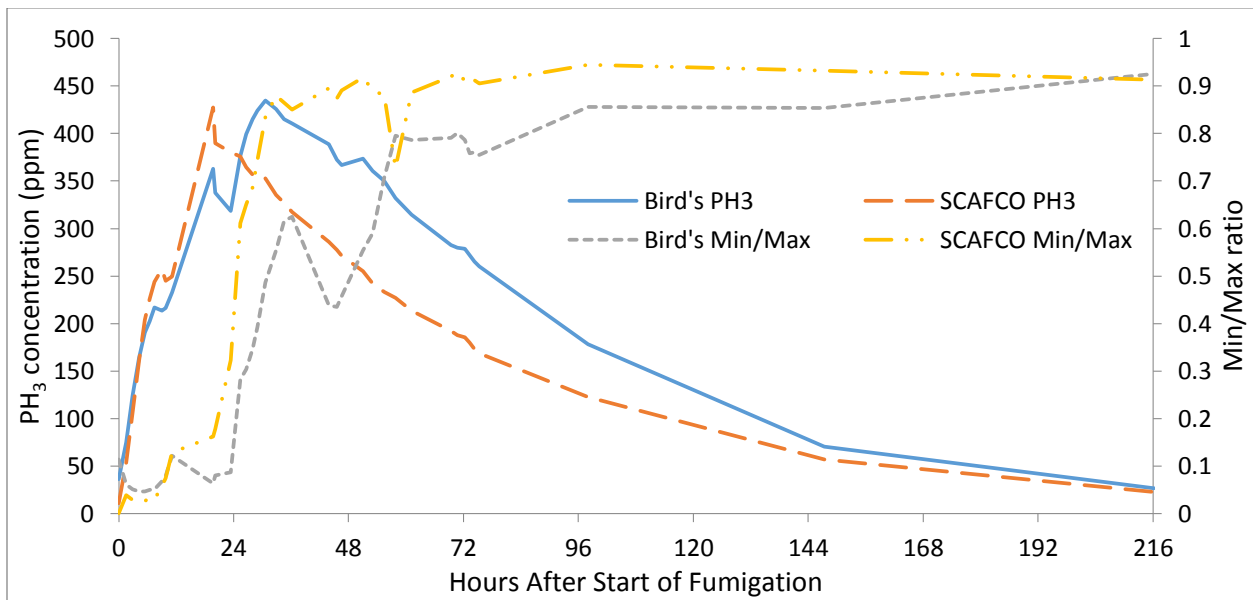
The average phosphine concentrations for both silos in trials 1 and 2 are shown in Figures 4.11 and 4.12, respectively. In trial 1, both silos reached a peak concentration after 32 hours. The SCAFCO silo reached a peak concentration of 354 ppm, and the Bird's silo reached a peak of 404 ppm. In trial 2, the SCAFCO silo reached a peak concentration of 490 ppm after 20 hours, compared to a peak of 498 ppm after 32 hours in the Bird's silo.

In trial 2 (using pellets), the concentration peaked earlier in the SCAFCO silo, but also began to drop more rapidly than in the Bird's silo. This may have been due in part to the higher partial vapor pressure of phosphine in the silo because of the more rapid production of  $\text{PH}_3$  using pellets versus tablets. At higher partial vapor pressures, gas loss rates increase per unit of time. Noyes and Phillips (2007) reported the same pattern of higher peaks obtained more quickly from using phosphine pellets during two fumigations at a Peavey Company Facility at the Tulsa Port of Catoosa in 2000 and 2002.

In trials 1 and 2, with respect to Banks and Annis' criterion 2, in both silos for both trials, the average maximum concentrations were over 50% of the expected amount (300 ppm). The concentration at the end of 125 h was approximately 100 ppm for both silos; well above the 7 ppm proposed by Banks and Annis. The min/max ratios were higher in the SCAFCO silo indicating that the distribution of  $\text{PH}_3$  was more rapid than in the Bird's silo. However, in both trials the silos reached the 0.25 threshold prior to 25% of the total exposure time as recommended by Banks and Annis (1984). The SCAFCO silo reached 0.25 after 19 hours and 22 hours for trials 1 and 2, respectively, and the Bird's reached 0.25 after 24 hours in both trials.



**Figure 4.11** Average fumigant concentrations in the Bird's and SCAFCO silos during August 24 – August 29, 2015 while fumigating approximately 43 MT of corn at 14% m.c. and 18°C. Approximately 30 phosphine tablets were released to reach a target concentration of 0.17 g/m<sup>3</sup> (300 ppm).

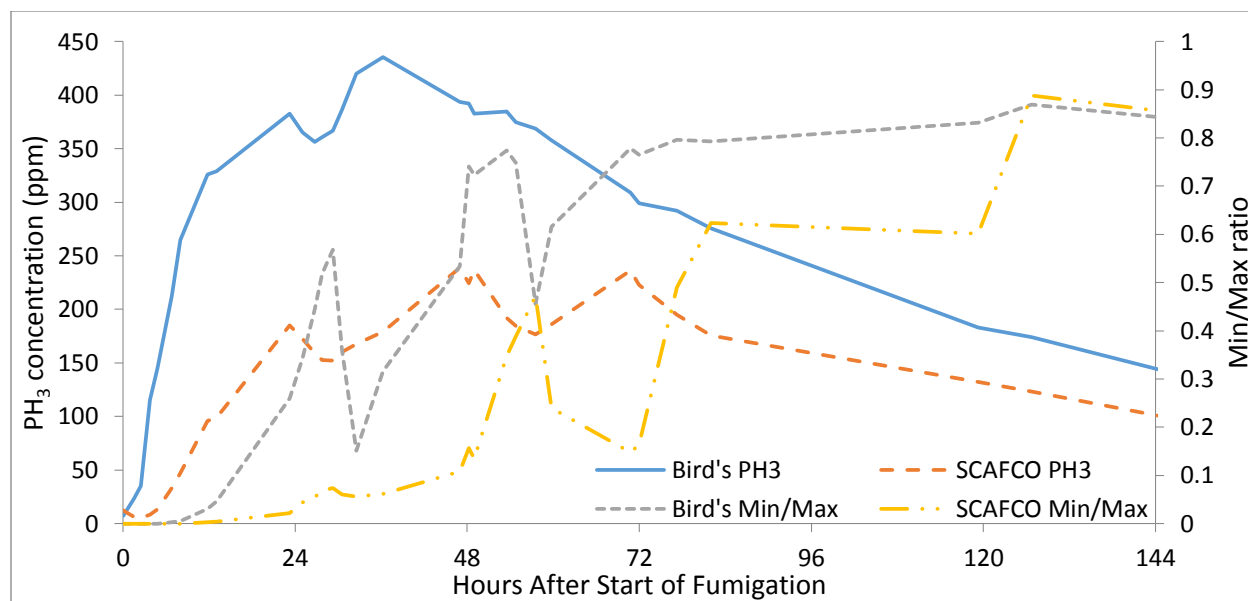


**Figure 4.12** Average fumigant concentrations in the Bird's and SCAFCO silos during August 31 – Sept. 9, 2015 while fumigating approximately 43 MT of corn at 14% m.c. and 18°C. Approximately 30 phosphine tablets were released to reach a target concentration of 0.17 g/m<sup>3</sup> (300 ppm).



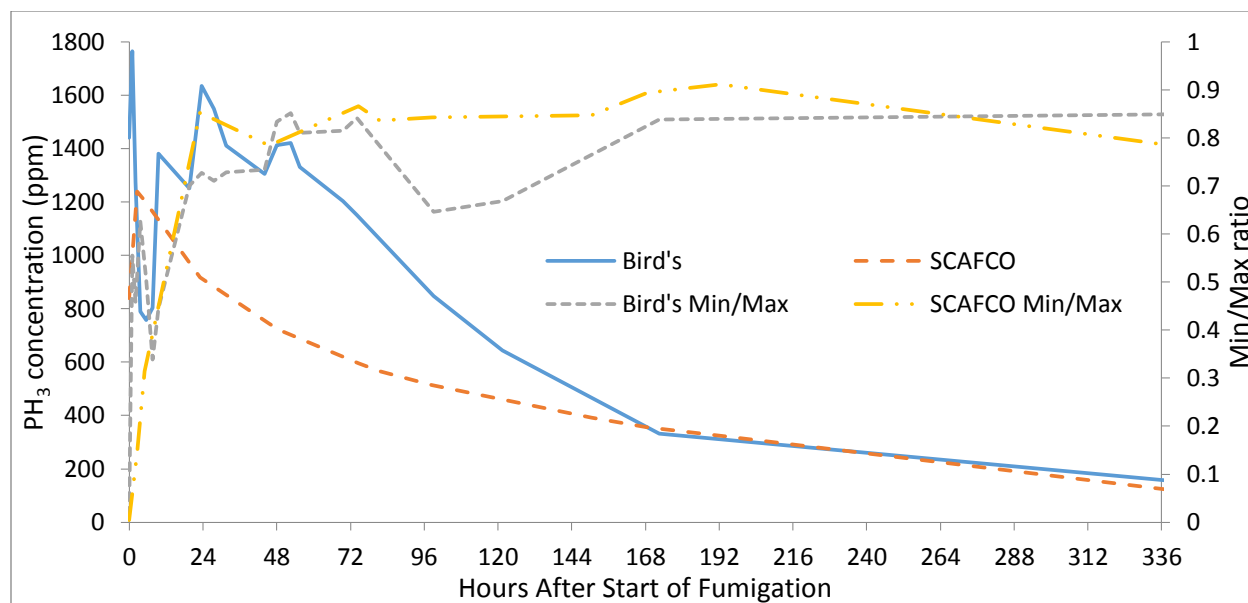
Figure 4.13 shows the average  $\text{PH}_3$  concentrations in the silos for trial 3. The ThermoSiphon in the SCAFCO silo was turned off, preventing recirculation in the silo and forcing  $\text{PH}_3$  to diffuse upward through the gap around the butterfly valve and into the grain mass. The average concentration of the 23 monitoring points inside the SCAFCO silo did not reach above 250 ppm, but in the Bird's silo, the fumigation concentration curve is similar to the curve in trial 1, reaching a peak of 435 ppm after about 36 h. In the SCAFCO silo, three concentration peaks at around 24, 48, and 72 h are seen. These measurements were the first readings taken in the morning, and the high concentration reflected by the peaks was located in the bottom of the silo, close to the source of gas from the  $\text{PH}_3$  tablets. These peaks likely reflect the "pooling" of  $\text{PH}_3$  in the bottom of the silo as the temperature-driven convection currents slowed during the evening and night. It is possible that the tablets were still generating  $\text{PH}_3$  after 48 h. The concentration for both silos after 125 h was still above 100 ppm. The min/max ratio in the SCAFCO silo did not reach 0.25 until after more than 48 h, while in the Bird's silo the min/max ratio reached 0.25 in less than 24 h. The low troughs in the Bird's min/max ratio at around 30 and 52 h, and in the SCAFCO min/max ratio at around 72 h were due to a high concentration of  $\text{PH}_3$  in the bottom of the silo in the evening when the thermosiphon reversed and  $\text{PH}_3$  moved upwards from the reaction chamber.

Figure 4.14 shows the gas concentrations for both silos during the VAPOR $\text{PH}_3\text{OS}^{\text{®}}$  fumigations. After the initial application, the Bird's silo maintained a concentration of approximately 1,400 ppm for over 55 hours before beginning to decrease. The SCAFCO silo maintained the initial concentration for approximately 5 hours, after which the  $\text{PH}_3$  began to decrease relatively steadily. The average concentrations were 1,373 and 876 ppm for the Bird's and SCAFCO silos, respectively. This was well above 350 ppm, which was 50% of the expected amount.



**Figure 4.13** Average fumigant concentrations in the Bird's and SCAFCO silos during September 18-24 while fumigating approximately 43 MT of corn at 14% m.c. and 20° C. Approximately 30 phosphine tablets were released to reach a target concentration of 0.17 g/m<sup>3</sup> (300 ppm).

The concentrations after 125 h for the Bird's and SCAFCO silos were approximately 640 and 425 ppm, respectively. The application of cylinderized gas distributed the fumigant inside both silos very quickly because the entire dose of PH<sub>3</sub> was applied at one time rather than generated over 24 h as a result of gas release from the tablets or pellets. Also, the applied dose (700 ppm) was more than twice the amount generated by tablets and pellets (300 ppm). The entire volume of PH<sub>3</sub> dispersed within the silo and reached a min/max ratio above 0.25 within an hour for the Bird's silo and less than 5 hours for the SCAFCO silo. The ratio was above 0.75 in under a day for both silos, and the distribution of PH<sub>3</sub> was uniform throughout the fumigations (above 0.6 and .75 for the Bird's and SCAFCO silos, respectively).



**Figure 4.14** Average PH<sub>3</sub> concentrations using VAPORPH<sub>3</sub>OS® cylinderized PH<sub>3</sub> in the Bird's silo during trial 4 (April 1 – 8, 2016) and SCAFCO silo during trial 5 (April 25 – May 9, 2016), fumigating approximately 43 MT of corn at 16% m.c. and 20° C. Approximately 205 grams of PH<sub>3</sub> was applied to achieve a target concentration of 1.5 g/m<sup>3</sup> (700 ppm).

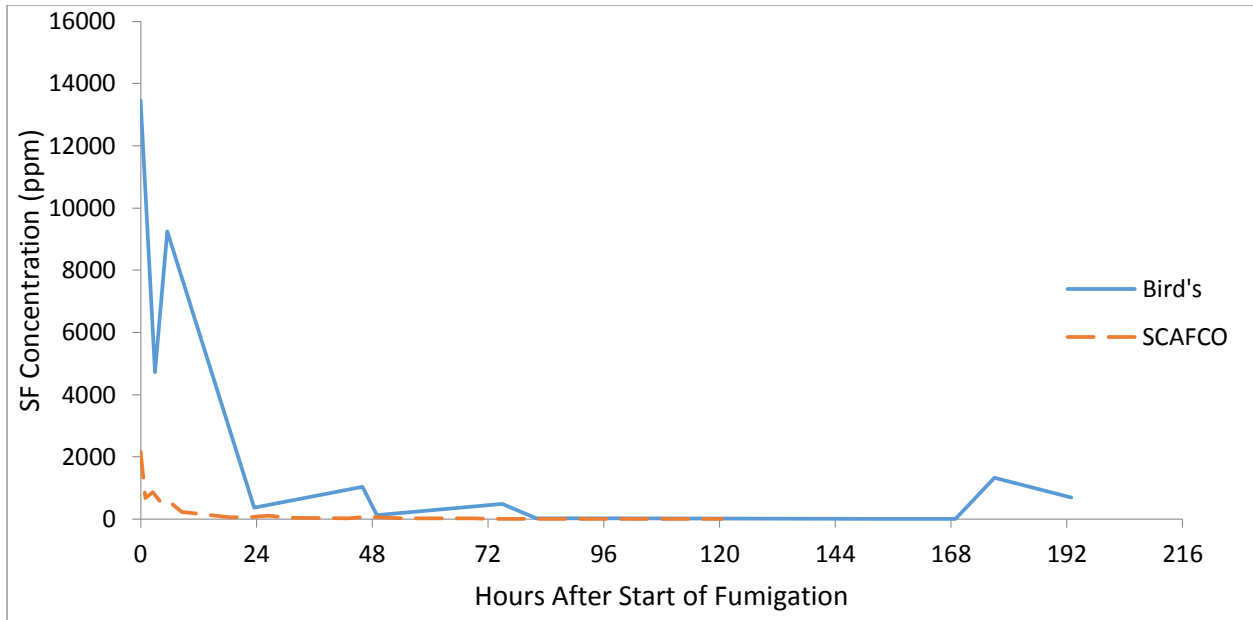
The concentration curves in this trial were similar to the curves for trial 3 using PH<sub>3</sub> pellets. For trials 1 and 3, in both silos the concentration depleted at similar rates after reaching the peak concentration. However, the Bird's silo had a higher peak concentration (1,765 ppm compared to 1238 ppm for the SCAFCO silo) and began to deplete later (52 hours compared to 5 hours). For this reason the SCAFCO silo actually had a smaller leakage rate due to how the leakage rate was calculated (Equation 4.1).

Figure 4.15 shows the average gas concentrations of both silos during the SF fumigations. It would appear that in the SCAFCO silo, virtually all of the fumigant had left the silo within 24 hours. For the Bird's silo the concentration measurements dropped to between 10 and 33 ppm after approximately 80 hours. Problems related to reading SF concentrations were encountered

during the SF fumigations. At every time step there were readings of zero ppm. Therefore, the min/max ratio was “0” throughout the fumigation.

Fumigant loss may be partially due to sorption of SF by the corn. Sorption into various commodities by PH<sub>3</sub> (Reed and Pan 1999, Berck 1968, and Dumas 1980, Xiaoping et al. 2004), and more recently SF (Hwaidi et al. 2015, Scheffrahn et al. 1989, and Sriranjini 2008) has been studied. Data are lacking on the sorption rates of SF in corn, but is available in wheat (Hwaidi et al. 2015), and other various food products (Scheffrahn et al. 1989, Sriranjini 2008). Experiments with hard white and durum wheat found sorption rates of between 1.25 and 1.85% per hour at 25° C and 15% m.c., with higher sorption rates at higher temperatures and moisture contents (Hwaidi et al. 2015). At 25° C, the daily sorption rate of PH<sub>3</sub> is similar for wheat and corn (about 10% of total PH<sub>3</sub> applied) (Dumas 1980). It may be possible that similar sorption rates for SF were in effect given the higher m.c. of the corn (16 – 17%) used in the present trial. If so, between 54 and 84 ppm/h, or a total of 1,643 ppm of SF would have been lost through sorption in the first 24 h. This helps to explain 48% of the loss of SF measured during the trials.

It is suspected that there were false zero readings during the SF fumigations because at a given location in the grain mass, the instrument would record a zero reading, then record a positive SF concentration again at a later point in time. It is likely that the fumigation curves in Figure 4.15 depict lower SF concentrations than were actually present in the silo. There are several possible explanations for this.



**Figure 4.15** Average SF concentrations using ProFume® cylinderized SF in the Bird’s silo during trial 5 (April 25 – May 3, 2016) and SCAFCO silo during trial 4 (April 1 – 5, 2016), fumigating approximately 43 MT of corn at 16% m.c. and 20° C. Approximately 1.36 kilograms of SF was applied as prescribed by the Fumiguide software.

For one, the equipment used to measure SF concentration utilized an onboard pump which drew gas through the monitoring lines. This was a low velocity pump and may not have had enough suction to overcome the negative pressure inside the silo and monitoring lines, i.e.,

$$P_{silo} + \Delta P_{lines} \leq P_{pump} \quad (4.2)$$

Where  $P_{pump}$  is the pressure of the onboard SF instrument pump,  $P_{lines}$  is the pressure in the monitoring lines, and  $P_{silo}$  is the pressure inside the silo. When the silo was opened, the pressure in the silo increased and the pump was able to draw SF into the monitoring lines.

Another possibility may be that the concentrations inside the silo prior to venting were outside the working range of the Spectros SF monitoring instrument, and therefore the instrument

returned faulty readings. However, the Spectros SF-ExplorIR instrument does have a working range of 0-30,000 ppm, which well encompasses the applied level of SF (4,540 ppm).

When the top and bottom covers of the Bird's silo were opened to vent the remaining gas after seven days, SF measurements throughout the silo jumped from 0 to between 200 and 1800 ppm (see spike after 168 hours). Professional fumigators have indicated that during some SF fumigations of grain, locations where SF has been measured will begin to measure zero concentration, but when the commodity is moved or fresh air is introduced during venting, the monitoring lines begin to record SF again (Dolan Jamison and Chris Newman, personal communication). Seven days after the start of trial 5, the SF concentrations all read zero throughout the grain mass. However, when the top and bottom covers were opened, SF was detected again throughout the grain mass, ranging from 24 to 1893 ppm, with an average of 958 ppm. Upon opening the covers, the pressure in the silo equilibrated with the outside. This would have facilitated the ability of the pump to draw gas from the silo to the instrument. Hwaidi et al. (2015) found that after initial aeration, no significant SF desorption occurred from whole wheat in jars, but every indication is that the wheat was not moved or agitated during these measurements. Sulfuryl fluoride desorption experiments with agitated or aerated grain should be considered for future research.

The pressure decay tests for the Bird's and SCAFCO silos prior to these trials yielded half-life times of 40 seconds and 20 seconds, respectively. While these do not meet the Australian standard AS2628 for sealed silos, they were comparable to half-life pressure decay times for the previous PH<sub>3</sub> fumigations that were successful. Thus, it is not likely the silos were excessively leaky.

Table 1 shows the CTPs, HLTs, average maximum concentrations, and leakage rates for the silos for all five fumigation trials. The HLTs and leakage rates for the SF fumigations were calculated from 0 to 24 h and 24-125 h because the leakage rates were very high during the first 24 h compared to the rest of the fumigation. The substantial differences of these values compared to what was observed in the PH<sub>3</sub> fumigations were likely due to a high degree of measurement error for the reasons explained above.

Reed and Pan (1999) found that PH<sub>3</sub> sorption rates are lower for subsequent fumigations on the same grain. Because the same corn was retained and used during all five fumigation trials, this may help explain why there were longer half loss times and lower leakage rates in trial 3 than trials 1 and 2.

Schneider et al. (2001) discussed the importance of knowing the HLT of a structure prior to fumigation in order to maximize fumigation cost effectiveness. Longer HLTs are indicative of well-sealed structures that are not as susceptible to wind effects and the stack effect. In the case of the Bird's silo in trials 1 and 3, both trials used the same number of aluminum phosphide tablets, but trial 3 had a lower leakage rate, longer HLT, and higher peak concentration. The wind speeds, average daily temperature, and solar radiation all have a large effect on gas loss from silos (Navarro 1998). For trials 1 and 3, the average temperature and wind speeds throughout the trials were relatively close (21.7 and 20.9 °C and 1.7 and 1.8 m/s, respectively). The average daily solar radiation during trial 1 was 215 W/min, respectively, compared to 165 W/min during trial 3. The greater amount of solar radiation during trial 1 may have caused the headspace air volume in the silos to expand and cause greater leakage than during trial 3.

**Table 4.1** Concentration time products, half-loss times, average maximum concentrations, and leakage rates in the Bird's and SCAFCO silos after 125 hours for five fumigation trials.

	CTP after 125 h (ppm-h)	Half-Loss Time (h)	Average Max Concentration (ppm)	Leakage Rate after 125 h (ppm/h)
<u>Trial 1</u>				
Bird's (PH <sub>3</sub> )	26,785	44	501	3.4
SCAFCO (PH <sub>3</sub> )	24,436	44	324	2.9
<u>Trial 2</u>				
Bird's (PH <sub>3</sub> )	38,561	48	467	4.1
SCAFCO (PH <sub>3</sub> )	30,547	32	343	3.9
<u>Trial 3</u>				
Bird's (PH <sub>3</sub> )	40,986	106	688	2.9
SCAFCO (PH <sub>3</sub> )	22,491	130	556	1.5
<u>Trial 4/5</u>				
Bird's (PH <sub>3</sub> )	163,701	98	1,373	9.4
SCAFCO (PH <sub>3</sub> )	102,942	67	876	6.2
<u>Trial 5/4</u>				
Bird's (SF)	160,213	<1	15,071	545.3*, 3.4**
SCAFCO (SF)	9,488	<1	3,021	89.0*, 1.0**

\*From 0-24 hours

\*\* From 24-125 hours

### Gas concentrations by horizontal levels and vertical sections

Uniform fumigant concentration is important to ensure the target concentration at every point in the silo is held for sufficient time to kill all insects at all life stages. When fumigating a leaky silo with poor gas circulation, a manager cannot be sure the targeted dosage is achieved at every



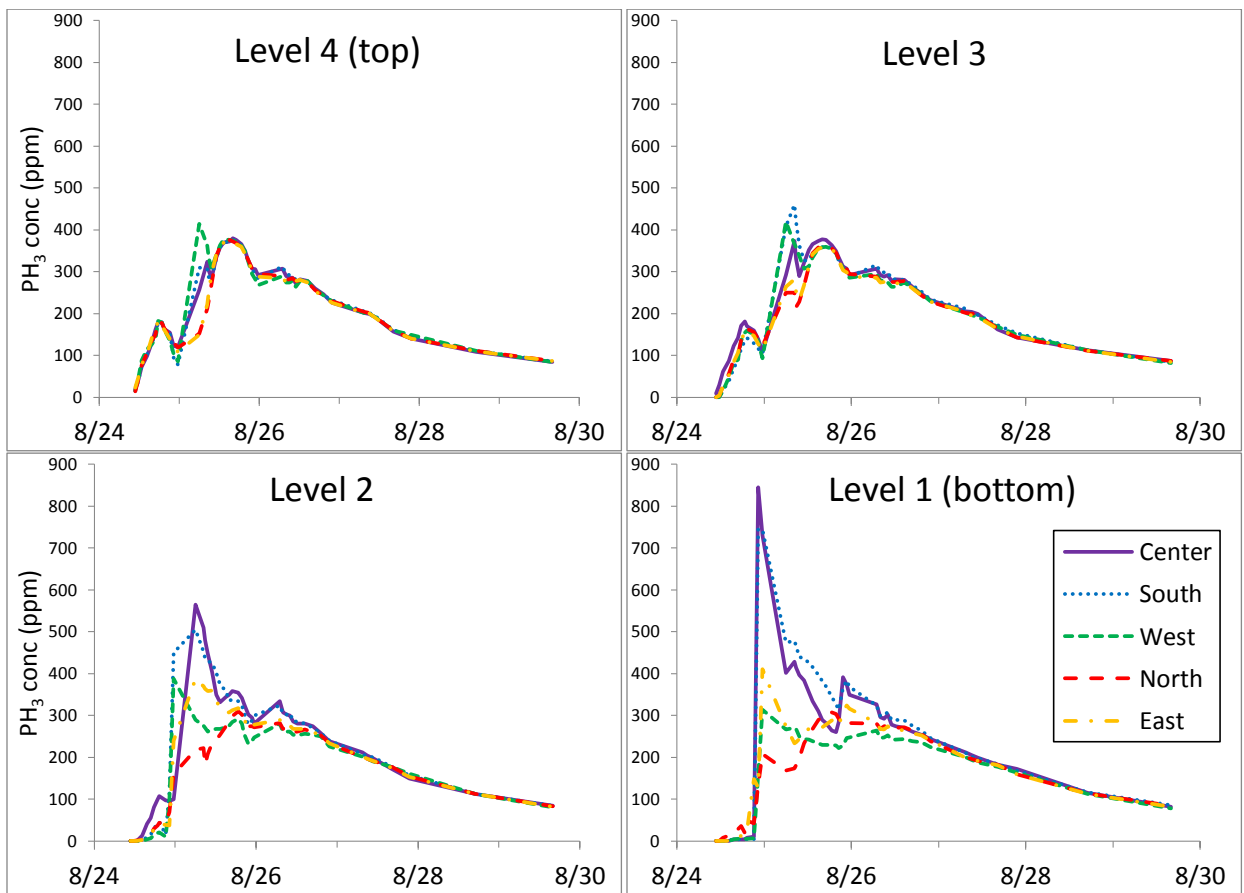
point in the silo. Monitoring the concentration at several points in the structure can help inform the manager whether the fumigation was a success or failure. Monitoring in the top of the grain mass is advised because of high insect densities found there (Flinn et al. 2004, Hagstrum 1989).

Figure 4.16 shows fumigant concentrations at each monitoring point at the four levels in the grain mass of the SCAFCO silo during trial 1. At the start of the trial, the phosphine concentrations were not uniform among the center, north, south, east and west vertical sections of the silos. This variation was most evident in the bottom half of the grain mass. The phosphine distribution in the top level was generally uniform. This pattern was observed in both silos for all three trials, except for the SCAFCO silo in trial 3 when the ThermoSiphon was turned off.

During  $\text{PH}_3$  generation from tablets and pellets the bottom levels of the grain mass generally had higher overall concentrations than the top level due to the proximity of the ground-level  $\text{PH}_3$  reaction chamber. After all the gas had evolved from the tablets or pellets, concentrations at different heights were considerably more uniform. In both silos, the min/max ratio was above 0.25 by the time the  $\text{PH}_3$  had evolved from the tablets or pellets except the SCAFCO silo for trial 3 (with ThermoSiphon off). As convection moved the fumigant up the ThermoSiphon, the gas diffused evenly throughout the headspace before being drawn down in the grain mass by the ThermoSiphon-driven recirculation effect of the convection currents in the grain mass.

Figure 4.17 shows the  $\text{PH}_3$  concentrations in the center, south, west, north and east sections at all four levels in the SCAFCO silo during trial 3 (September 18-23). Even though the average concentration in the silo, or the concentration at any one point in the silo may be sufficient to kill insects at all life stages, there may be areas of sub-lethal concentration. For example, during trial

3, the top level of the SCAFCO silo (with ThermoSiphon off) reached a CTP of only 19,158 ppm-h, while the Bird's silo (with ThermoSiphon on) reached 37,841 ppm-h, even though both silos had average concentrations sufficient to kill all insect life stages (22,492 and 40,986 ppm-h, respectively). The top layer of grain is usually where insect infestations are most dense (Flinn et al. 2004, Hagstrum 1989), so maintaining lethal concentrations especially in this area is critical to fumigation success.



**Figure 4.16.** Phosphine concentrations in the center, south, west, north and east sections at all four levels in the SCAFCO silo during trial 1 (August 24-27) with the ThermoSiphon turned on.

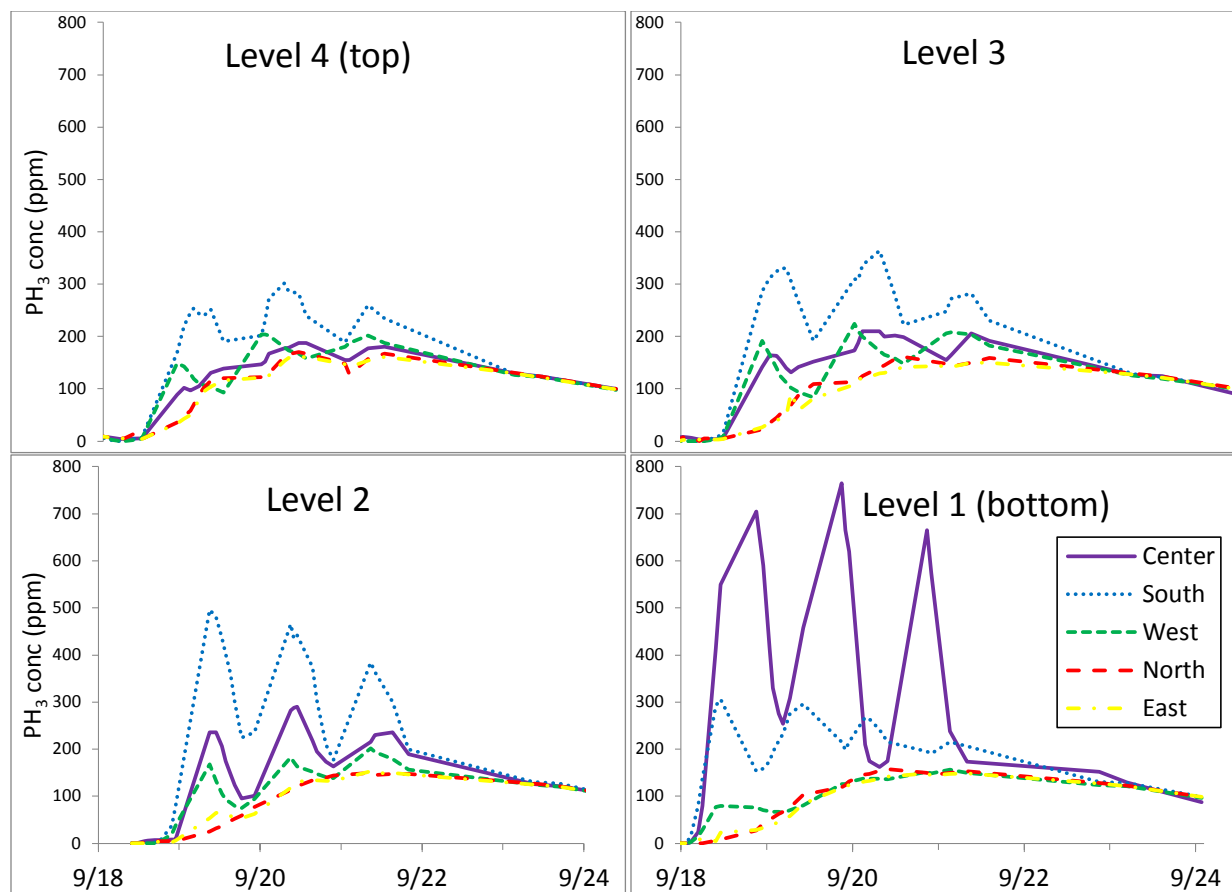
For the first three trials (with tablets or pellets), the center and south side of the grain mass had the highest overall concentrations. In the Bird's silo, the east side had the next highest

concentrations while in the SCAFCO silo the west side had the next highest concentrations. The lowest concentrations in the SCAFCO silo were on the east and north sides, while the lowest concentrations in the Bird's silo were typically on the west side. This is further discussed in the next section.

In trials 4 and 5 with cylinderized  $\text{PH}_3$ , for the first several hours of the fumigation, higher concentrations were found in the center (1,525 ppm compared to between 980 and 1,176 ppm for the other vertical locations (Figure 4.18). After about 4.5 h, the concentrations became much more uniform, all lines were between 893 and 862 ppm. The variation among the vertical monitoring lines reached uniformity much more quickly than in the trials with tablets and pellets. Among the center, north, south, east and west locations, the min/max ratios were 0.81 and 0.37 after one hour in the Bird's and SCAFCO silos, respectively.

In trials with  $\text{PH}_3$  tablets and pellets, the concentrations along the vertical monitoring lines became uniform after 36-48 h except in the case of trial 3 in the SCAFCO silo (without recirculation) in which the vertical sections became uniform after 120 hours (about 2.5 times longer).

Figure 4.19 shows the average gas concentrations at each height throughout the SCAFCO silo during trial 5 using VAPOR $\text{PH}_3\text{OS}^{\text{®}}$ . The distribution among these sampling points became uniform rapidly, the min/max ratio reaching more than 0.50 within three hours after the start of the fumigation. Similar results were seen in the Bird's silo with VAPOR $\text{PH}_3\text{OS}^{\text{®}}$ .

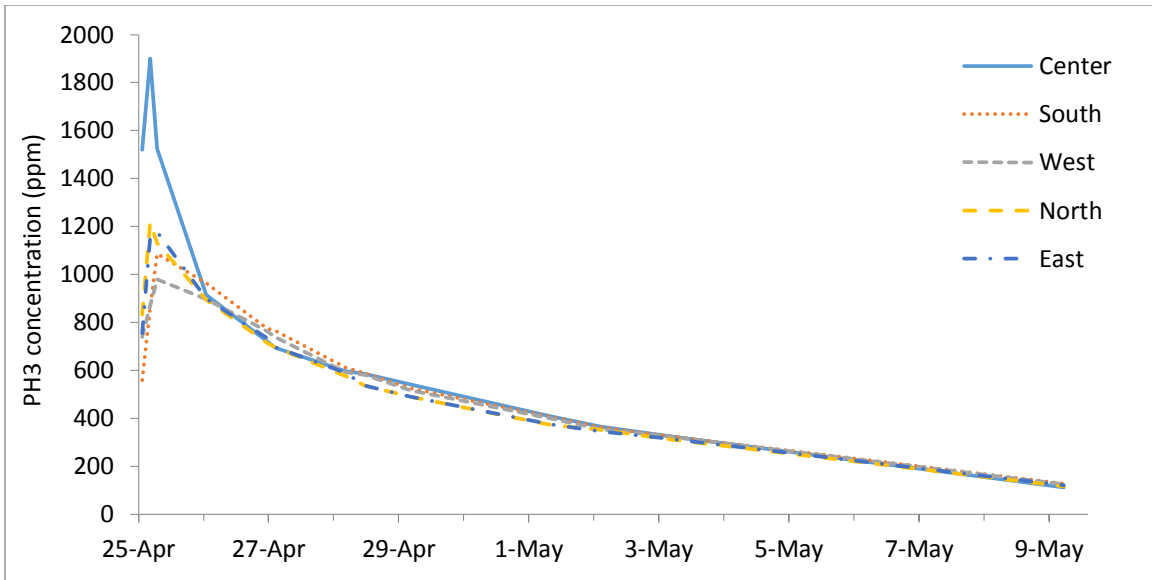


**Figure 4.17.** Phosphine concentrations in the center, south, west, north and east sections at all four levels in the SCAFCO silo during trial 3 (September 18-23) with the ThermoSiphon turned off.

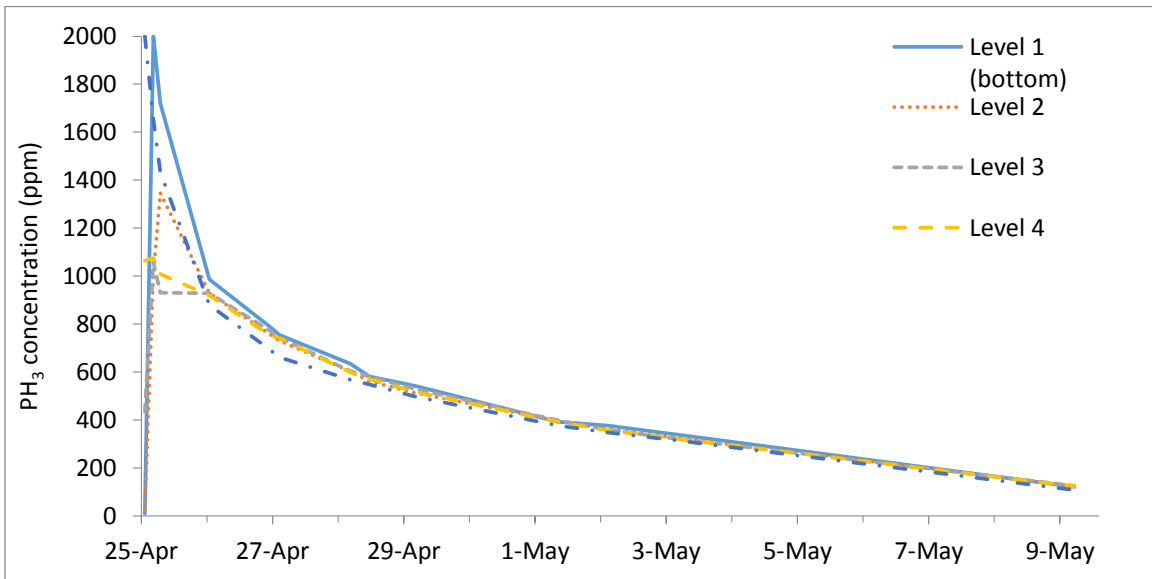
### Effect of diurnal fluctuations on gas movement

Figure 4.20 is a top view diagram of the silo location showing the areas of shaded versus exposed to the sun during mid-morning and mid-afternoon. As ambient conditions change, the air and grain temperature gradients inside the grain mass shift, causing the air movement inside the grain to change as well (Alagunsundaram et al. 1990). The areas of highest fumigant concentration corresponded to the sides of the silos that were exposed to the sun for the longest.

At the start of the fumigation,  $\text{PH}_3$  moved upwards through the ThermoSiphon and into the headspace. Variation in concentrations was seen mostly in the top levels as  $\text{PH}_3$  moved downwards through the grain mass.



**Figure 4.18.** Phosphine concentrations in the center, south, west, north, and east sections at all four levels in the SCAFCO silo during trial 5 (April 25 – May 9, 2016) using VAPORPH<sub>3</sub>OS®.



**Figure 4.19** Average PH<sub>3</sub> concentrations at the different heights in the SCAFCO silo during trial 5 (April 25-May 9 using VAPORPH<sub>3</sub>OS®).

After the sun had gone down, the ThermoSiphon stopped moving fumigant upward because there was no longer a solar effect to cause temperature gradients between the thermosiphon, headspace and grain mass sufficient for convection currents. However, the tablets or pellets were

still generating  $\text{PH}_3$  in the reaction chamber. As a result of the  $\text{PH}_3$  concentration gradient, the  $\text{PH}_3$  moved upwards from the grain mass in the bottom of the hopper into the upper level grain mass. This resulted in the bottom levels of both silos having much higher concentration than the top levels during the evening and night.

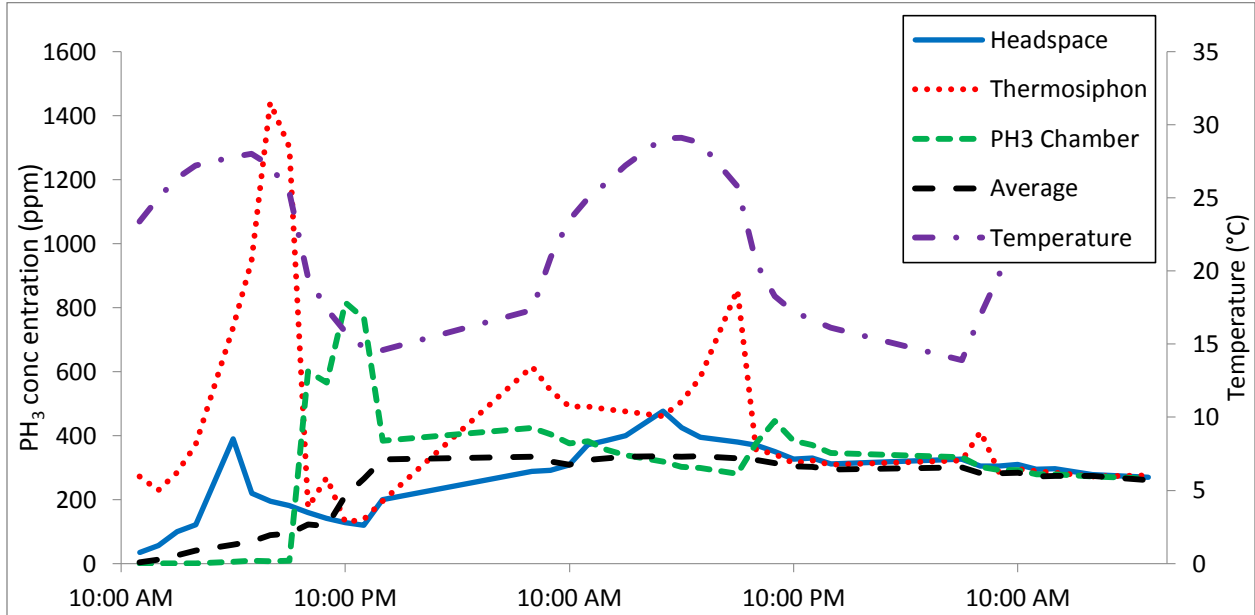


**Figure 4.20** Top view of the two silos while under fumigation. The sides of the silos with the lowest gas concentration were shaded throughout most, or all, of the day, and the sides with the highest gas concentration were exposed to more solar radiation unless the sky was overcast.

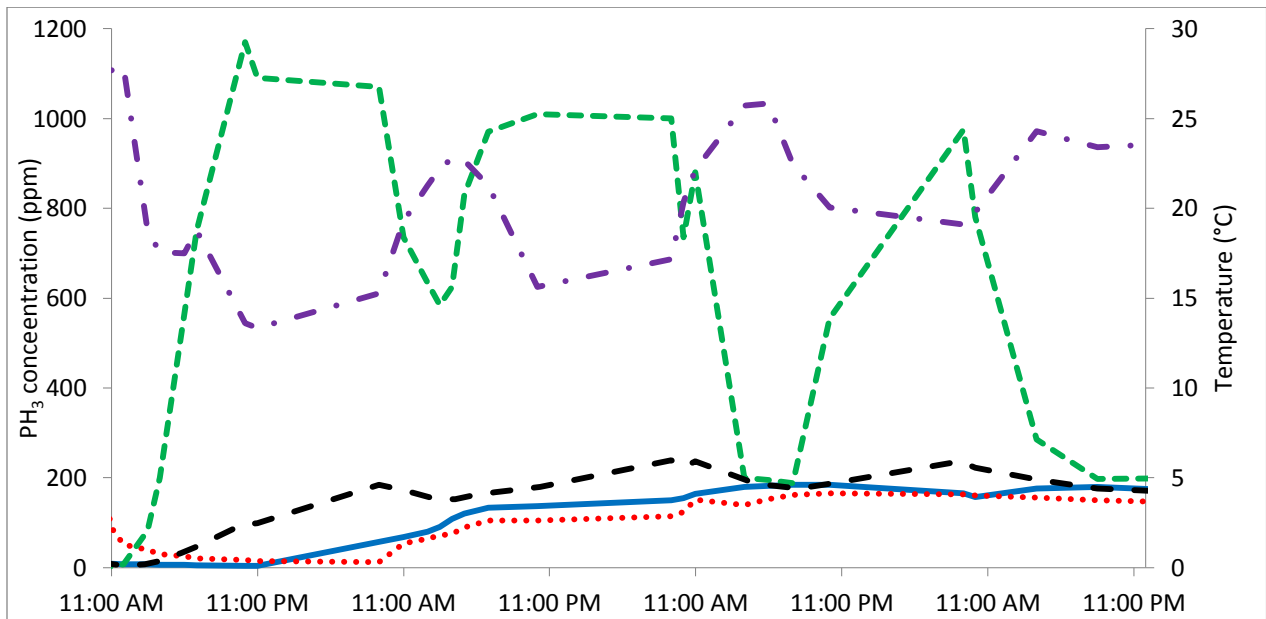
Except in trial 3, the SCAFCO silo achieved uniform gas concentration in the grain mass more quickly than the Bird's silo. This could be due to the fact that the SCAFCO silo had greater exposure to solar radiation during the hotter parts of the day (i.e., between approximately 7 am and 7 pm). This would have caused greater natural convection currents to move  $\text{PH}_3$  within the grain mass. The SCAFCO and Bird's silos had similar surface area to volume ratios (1:1.6 and 1:1.8, respectively). A high surface to volume ratio leads to more extreme temperature gradients within the grain mass (Montross 1999), which in turn causes higher convection velocities. In trial 3, there was no recirculation in the SCAFCO silo to facilitate  $\text{PH}_3$  distribution. As a result, it took more than 48 hours for the min/max ratio to reach 0.25, whereas in every other trial with pellets or tablets the min-to-max ratio reached 0.25 within approximately 24 hours.

Figures 4.21 and 4.22 illustrate further the beneficial effect of the ThermoSiphon in terms of  $\text{PH}_3$  concentrations in the headspace, in the ThermoSiphon, and directly above the  $\text{PH}_3$  reaction chamber, along with the ambient temperature during trials 1 and 3 in the SCAFCO silo. Peaks in the ThermoSiphon concentration were observed during the day when the sun was near its highest point (11:00 am to 2:00 pm) and the ambient temperature reached maximum. During the day, increased air temperatures due to solar radiation caused convection currents to move the air- $\text{PH}_3$  mixture upward in the thermosiphon, which reached concentrations as high as 1,440 ppm. The concentration in the headspace peaked at almost 400 ppm five hours after the start of the fumigation and the top level readings increased steadily to 160-170 ppm. Conversely, the concentrations were higher in the grain mass in the hopper bottom near the  $\text{PH}_3$  reaction chamber during the cooler evenings (approximately  $15^\circ\text{C}$ ). For the first eight hours of the fumigations, while the  $\text{PH}_3$  was being moved upwards through the ThermoSiphon, the  $\text{PH}_3$  concentrations near the  $\text{PH}_3$  reaction chamber were 6 ppm for the bottom level readings of the south and west lines, and 19 ppm for the north and east lines. After the sun set, the concentration in the ThermoSiphon dropped from 1,300 to 176 ppm, while it rose from 9 to 600 ppm near the  $\text{PH}_3$  reaction chamber as the gas was no longer being carried by the convection current created by the ThermoSiphon pipe. Instead, it travelled upwards through the gap around the butterfly valve and into the grain mass. In the evening, approximately twelve hours after the start of the fumigation, the concentration near the  $\text{PH}_3$  reaction chamber rose to 815 ppm and the bottom of the south monitoring line was 745 ppm. Concentrations at the bottom of the west, north and east lines were between 134 and 410 ppm. This may be due to slower convection currents within the grain mass on those sides, as they were shaded during the hotter period of the day and would not have warmed as much as the south side.





**Figure 4.21** Phosphine concentrations inside the headspace, ThermoSiphon, and near the reaction chamber in the SCAFCO silo during trial 1. The average PH<sub>3</sub> concentration and ambient temperature are also shown. The legend also applies to Figure 4.22.



**Figure 4.22** Phosphine concentrations inside the headspace, ThermoSiphon, and near the reaction chamber in the SCAFCO silo during trial 3 with the ThermoSiphon off. The average PH<sub>3</sub> concentration and ambient temperature are also shown.

When the ThermoSiphon was turned off in trial 3, the  $\text{PH}_3$  was forced to move upward through the grain mass. In the hopper bottom near the  $\text{PH}_3$  reaction chamber, concentrations were as high as 200 ppm five hours after the start of the fumigation, but no higher than 15 ppm anywhere else in the silo. The concentration in the lower part of the silo continued to rise during the evening, reaching 1,170 ppm near the reaction chamber. The variation was high in the bottom level readings, ranging from 5 ppm at the east and north lines to 77 ppm at the west line, 292 ppm at the south line, and 414 ppm at the center line. As ambient temperature rose, the concentration near the reaction chamber decreased, while the concentrations at the middle and upper levels in the grain mass increased. During the day, the effects due to solar radiation and higher ambient temperatures likely created convection currents within the grain mass that carried the  $\text{PH}_3$  away from the chamber. These currents were asymmetrical within the silo, with different velocities at different locations up and down the grain mass because of differences in temperatures that cause variations in air velocities. The variations in  $\text{PH}_3$  concentrations observed in trials 1 through 3 are evidence for this phenomenon.

At night, convection slowed which caused less gas movement inside the grain mass, and thus resulted in higher concentrations near the  $\text{PH}_3$  reaction chamber. There was a gradual increase in the concentrations in the headspace and ThermoSiphon after  $\text{PH}_3$  finally reached the top levels. In trial 3, fumigant reached the ThermoSiphon through its connection in the roof, but it was turned off for the duration of the fumigation. While  $\text{PH}_3$  eventually dispersed evenly within the grain mass, the center and south lines had higher average concentrations (166 and 191 ppm, respectively) than the east, north and west lines (67, 94, and 69 ppm, respectively) for the first 80 hours. After 24 hours, the min/max ratio among the monitoring points at various heights reached

nearly 0.50, ranging from 95.4 and 198 ppm. But the average concentration did not get above 270 ppm at any level.

### **Thermosiphon Recirculation**

The thermosiphon had an air velocity of 0.02 to 0.08 m/s during the day in sunny conditions and 0.01 to 0.02 m/s in partly cloudy conditions, with ambient temperatures ranging from 16.1 to 19.6 °C. During the evening under no sun the velocity dropped to 0.0 m/s. In the 90 mm thermosiphon pipe these air velocities are equal to an airflow rate of 0.46 to 1.83 m<sup>3</sup>/h, or 0.005 to 0.04 m<sup>3</sup>/h/MT. This is well below the range of recommended recirculation airflow rates of 0.42 to 0.6 m<sup>3</sup>/h/MT (Noyes et al. 1998). However, for recirculation of CO<sub>2</sub>, Banks and Annis (1981) recommended a recirculation rate equal to 0.1 volumes of airspace in the silo per day. The thermosiphon airflow rates were between 0.02 and 0.08 for approximately 5 h during the day when the sun warmed the air in the thermosiphon. During this time, assuming an average airflow rate of 0.05 m/s (1.145 m<sup>3</sup>/h), five hours of thermosiphon activity would move 5.7 m<sup>3</sup> of air. This is close to the 0.1 volumes of air in the Bird's and SCAFCO silos of 6.3 and 7.2 m<sup>3</sup>, respectively.

During the concentration readings outside the silos during trials 1 and 5, all readings were zero ppm. Even though the silos leaked somewhat as evidenced by the fumigation curves, the PH<sub>3</sub> leaking out was undetectable using the handheld Draeger X-am 5000 personal monitoring device. The device has a working range of 1 to 2000 ppm, while the maximum permissible exposure limit is 0.3 ppm. The 15 minute short term exposure limit is 1 ppm (NIOSH 2016). The leakage rate was either slow enough, or the leak was diluted enough in the air outside the silo to remain undetectable.

## Bioassays

Table 4.2 shows the results of the insect bioassays used during trial 1, including the corresponding PH<sub>3</sub> CTP to which the insects were exposed to at each location. All adult insects of both species were killed during the fumigation. The CTPs in the Bird's and SCAFCO silos (approximately 26,000 and 24,000 ppm-h, respectively) exceeded the target CTP of 21,600 ppm-h, and the fumigant was held for 5 days above 100 ppm.

Fumigation trial 1 was successful in controlling any eggs that were laid in the bioassays. After six weeks the only adult emergence was one dead adult *S. zeamais* found in a jar from the SCAFCO silo. The authors suspect this individual may have been overlooked in the initial mortality count following the fumigation trial. In the emergence count for the controls, 328 live and 3 dead adult *S. zeamais* were found, while 13 live and 12 dead *T. castaneum* were found. Rajendran (2000) reported a delay in hatching for eggs of susceptible *T. castaneum* exposed to 30 ppm of PH<sub>3</sub> for 120 h and a resistant strain exposed to 300 ppm for 72 hours. This highlights the importance of either maintaining lethal concentrations within the grain bulk long enough to kill more resistant life stages of eggs and pupae, or let the individuals in these stages develop into the more susceptible stages of larvae and adults (Winks 1987). It is generally understood that in PH<sub>3</sub> fumigations a longer exposure time rather than a higher concentration is more important in order to kill all life stages of insects (Price and Mills 1988, Darglish et al. 2002).

Table 4.3 shows some of the various dosage recommendations from the literature for PH<sub>3</sub> fumigations to kill 99-100% of all life stages of stored product insects. Concentrations of PH<sub>3</sub> as low as 10 ppm can be used if the gas is held for a sufficient amount of time.

**Table 4.2** Bioassay results of *S. zeamais* and *T. castaneum* during PH<sub>3</sub> fumigation trial 1, August 24-29, 2015.

	<i>S. zeamais</i>		<i>T. castaneum</i>		CTP at location (ppm-h)
	Mortality	Emergence (6 weeks)	Mortality	Emergence (6 weeks)	
<u>Bird's</u>					
Center	100%	0	100%	0	26,994
South	100%	0	100%	0	26,197
West	100%	0	100%	0	26,416
North	100%	0	100%	0	26,863
East	100%	0	100%	0	26,174
Total	-	0	-	0	
<u>SCAFCO</u>					
Center	100%	0	100%	0	24,124
South	100%	0	100%	0	24,316
West	100%	0	100%	0	24,663
North	100%	1	100%	0	23,438
East	100%	0	100%	0	23,283
Total	-	1	-	0	
<u>Control</u>					
Center	18%	76	13%	0	-
South	20%	52	8%	1	-
West	24%	77	10%	8	-
North	0%	37	8%	8	-
East	5%	89	14%	8	-
Total	-	331	-	25	

Table 4.4 shows the bioassay results for the fumigations with PH<sub>3</sub> and SF, respectively. After the PH<sub>3</sub> fumigation, all adult insects were killed. One *T. castaneum* adult was found while doing the mortality counts, but this individual was very active and probably flew into the area from another part of the lab, as these insects are highly mobile.

For *R. dominica*, all PH resistant and PH<sub>3</sub> susceptible adult insects were killed during the SF and PH<sub>3</sub> fumigations in the Bird's and SCAFCO silos, respectively. In the controls, 135 PH<sub>3</sub> susceptible (3 dead, 132 alive) and 134 PH<sub>3</sub> resistant (39 dead, 134 alive) individuals survived.

From the SF bioassays, two adults (1 dead, 1 alive) and three adults (all dead) emerged from the PH<sub>3</sub> susceptible and PH<sub>3</sub> resistant bioassays, respectively. From the PH<sub>3</sub> bioassays, seven adults (2 dead, 5 alive) and three adults (1 dead, 2 alive) emerged from the PH<sub>3</sub> susceptible and PH<sub>3</sub> resistant bioassays, respectively. In the controls, 521 adults (29 dead, 492 alive) and 255 adults (24 dead, 231 alive) emerged from the PH<sub>3</sub> susceptible and PH<sub>3</sub> resistant bioassays, respectively. For *T. castaneum*, only one adult survivor (under SF) in all the bioassays was found after the fumigations. In the controls, 125 (6 dead, 119 alive) and 237 (6 dead, 231 alive) adult insects were found in the PH<sub>3</sub> susceptible and PH<sub>3</sub> resistant populations, respectively. From the SF bioassays, 26 adults (1 dead, 25 alive) and 10 adults (all alive) emerged from the PH<sub>3</sub> susceptible and PH<sub>3</sub> resistant bioassays, respectively. In the SCAFCO silo under PH<sub>3</sub>, five adults (all alive) and two adults (both alive) emerged from the PH<sub>3</sub> susceptible and PH<sub>3</sub> resistant bioassays, respectively. In the controls, 324 adults (34 dead, 290 alive) and 331 adults (19 dead, 312 alive) emerged from the PH<sub>3</sub> susceptible and PH<sub>3</sub> resistant bioassays, respectively.

*T. castaneum* had greater emergence than *R. dominica*. For *T. castaneum*, PH<sub>3</sub> performed better for controlling emergence. It also performed better for controlling emergence of PH<sub>3</sub> susceptible *R. dominica*, but for PH<sub>3</sub> resistant *R. dominica* SF and PH<sub>3</sub> performed equally well. Phosphine susceptible insects had higher emergence for both species in all fumigations except for *R. dominica* under SF fumigation, but there only two adults emerged from the PH<sub>3</sub> susceptible bioassay and three emerged from the PH<sub>3</sub> resistant.

**Table 4.3** Minimum PH<sub>3</sub> concentrations and times required to achieve a near-complete kill for all life stages of stored-product insects<sup>1</sup>.

Concentration (ppm)	Time (h)	CTP (ppm-h)	Source
10,000	36	360,000	Annis 2001
1,200	48	57,600	
1,000	192	192,000	
200	240	48,000	
35	480	16,800	
10	720	7,200	
300 <sup>2</sup>	168 <sup>2</sup>	50,400	Anon 2013
200 <sup>3</sup>	240 <sup>3</sup>	48,000	
710	120	85,200	Adapted from Collins et al. 2004 <sup>4</sup>
210	240	50,400	
142	336	47,712	

<sup>1</sup>Excluding *Trogoderma* spp.

<sup>2</sup>When grain is above 25° C

<sup>3</sup>When grain is between 15-25° C

<sup>4</sup>For highly PH<sub>3</sub>-resistant *R. dominica*

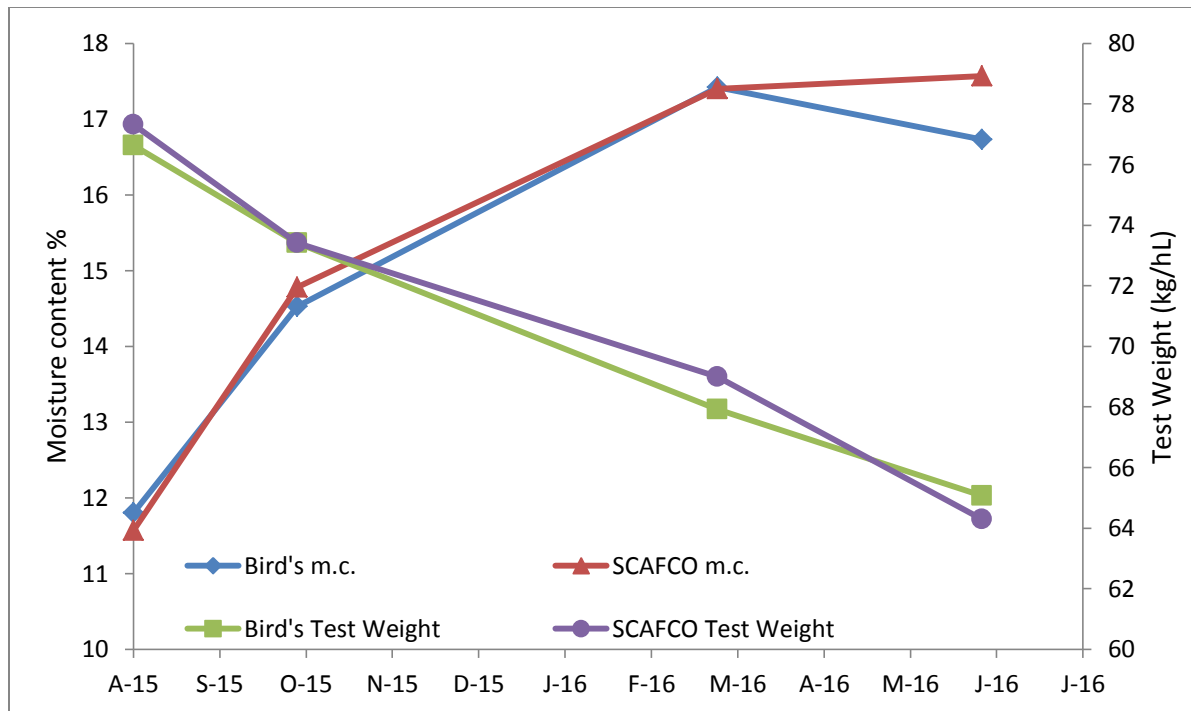
### Grain quality

Casada and Noyes (2001) recommended installing vents on the roof of sealed storage structures to prevent excessive moisture accumulation in the top of the grain due to moisture condensation. Vents were not installed on the silos used in this study, and the corn was kept in the silos for 10 months from August 2015 to June 2016. Figure 4.23 shows the moisture content of corn in the top 0.3 m of grain of both silos throughout the storage period. The average moisture content of the corn in that layer increased from approximately 11.5 to 17% in both silos over the duration of the storage period, and the test weights dropped substantially from approximately 77 to 65 kg/hL.

**Table 4.4.** Bioassay results of *R. dominica* and *T. castaneum* during fumigation trial 5, April 25-May 4, 2016

	<i>R. dominica</i>				<i>T. castaneum</i>				CTP at bioassay location (ppm-h)
	<u>PH<sub>3</sub> susceptible</u>		<u>PH<sub>3</sub> resistant</u>		<u>PH<sub>3</sub> susceptible</u>		<u>PH<sub>3</sub> resistant</u>		
	Mortality	Emergence	No. adults	Emergence	No. adults	Emergence	No. adults	Emergence	
<u>Bird's (SF)</u>									
Center	100%	0	100%	1	100%	6	100%	1	134 g-h/m <sup>3</sup>
South	100%	1	100%	1	97%	4	100%	0	164 g-h/m <sup>3</sup>
West	100%	0	100%	1	100%	5	100%	2	1,000 g-h/m <sup>3</sup>
North	100%	1	100%	0	100%	6	100%	1	374 g-h/m <sup>3</sup>
East	100%	0	100%	0	100%	5	100%	6	382 g-h/m <sup>3</sup>
Total	-	2	-	3	-	26	-	10	-
<u>SCAFCO (PH<sub>3</sub>)</u>									
Center	100%	0	100%	0	100%	0	100%	0	148,117
South	100%	1	100%	0	100%	0	100%	0	150,441
West	100%	4	100%	0	100%	1	100%	0	147,151
North	100%	2	100%	3	100%	4	100%	2	140,880
East	100%	0	100%	0	100%	0	100%	0	143,164
Total	-	7	-	3	-	5	-	2	-
<u>Control</u>									
Center	4%	110	27%	58	20%	96	4%	36	-
South	0%	134	18%	58	7%	122	0%	13	-
West	4%	71	33%	43	3%	65	2%	26	-
North	0%	76	15%	37	0%	98	2%	145	-
East	4%	130	19%	59	6%	43	4%	111	-
Total	-	521	-	255	-	424	-	331	-





**Figure 4.23 Moisture content and test weight of corn in the top 0.3 m layer kept in the sealed silos from August 2015 to June 2016.**

In March 2016, samples were taken further down in the grain, at 1 and 2 m depths. For the Bird's silo, the moisture contents were 14.9 and 13.8%, and the test weights were 72.8 and 74.1 kg/hL at 1 and 2 m depths, respectively. For the SCAFCO silo the moisture contents were 15.6 and 14.2%, and the test weights were 71.4 and 73.4 kg/hL at 1 and 2 m depths, respectively. When the corn was put into the silo, the moisture content and test weight in these layers were 12% and 77 kg/hL, respectively. This indicates a substantial moisture difference due to corn equilibration with humid headspace air and moisture condensation between the top layer and the rest of the grain mass in both silos. This difference can be seen up to 2 m under the grain surface, although to a lesser extent than the top 0.3 m of corn. Under the Bird's silo roof a strengthening ring was installed. A ring of moldy corn was observed directly underneath this ring in the exact same circumference and position. This indicates headspace condensation dripping from the roof onto the top of the corn causing spoilage.

Table 9 shows the insects found in the probe traps placed in the silos in March 2016. More insects were found in the SCAFCO silo than the Bird's silo, possibly because of the higher moisture content in the top of the grain. Though mold was found in the top layer of corn in both

silos, none of the insects found are primarily known as mold feeders. The silos were kept sealed through the winter, and while some insects may have found their way into the silos from outside through miniscule cracks in the silo structure, it is more likely that eggs previously laid in the grain were not killed during the fumigations in the fall of 2015.

**Table 4.5.** Insects found in probe traps placed in both silos for 2 weeks in March 2016

	Bird's		SCAFCO	
	<u>Alive</u>	<u>Dead</u>	<u>Alive</u>	<u>Dead</u>
<i>T. castaneum</i>	7	6	17	11
<i>R. dominica</i>	0	0	0	19
<i>S. oryzae</i>	1	1	0	1
Total	8	7	17	40

## Conclusions

This project evaluated the feasibility of using an Australian and a U.S. sealed steel hopper silo with thermosiphon recirculation equipment to achieve successful fumigations, control stored-grain insect pests, and maintain stored grain quality. The specific conclusions from this research are:

- Pressure testing of sealed silos can be easily and inexpensively done using a simple U-shape manometer.
- Both silos failed to achieve the 3-5 minute half-life during pressure decay testing as required by the Australian sealing standard. For the Australian silo, half-life times ranged from 5 seconds to 2 minutes 43 seconds, compared to half-life times of 20 to 50 for the U.S. silo. However, the fumigation success criteria proposed by Banks and Annis (1984), CTPs, and bioassay results obtained during the fumigation trials indicate both silos were nevertheless fumigated successfully.

- Using pellets,  $\text{PH}_3$  was produced at a higher rate than tablets and reached higher peak concentrations. Using tablets in trial 1, it took 32 h for the U.S. and Australian silos to reach a peak concentration of 354 and 404 ppm, respectively. Using pellets in trial 2, it took 20 h for the U.S. silo to reach a peak of 490 ppm, and 30 h for the Australian silo to reach a peak of 498 ppm.
- The Australian-designed thermosiphon recirculation technology effectively distributes fumigant throughout the grain mass of a silo as a result of solar radiation and temperature effects. This increases overall fumigant uniformity and speed of dispersion within the silo, even on the shaded sides. For trials 1 through 3, it took about 24 h for the average concentration to peak with the thermosiphon compared to 48 h without the thermosiphon. In the U.S. silo in trial 1 with the thermosiphon, all 23 monitoring points in the silo reached 300 ppm after about 24 h, while in trial 3 without the thermosiphon, readings in the north and east (shaded) did not reach 160 ppm.
- Phosphine distributed throughout the silo without recirculation from the thermosiphon, however it was at a slower rate. During dispersion of the fumigant, the south side of the silo, which received the most sunlight, had consistently higher concentrations (average 187 ppm) than the north and east sides which did not receive as much sun (average 77 ppm).
- In trials 4 and 5, the concentration of SF fluctuated between zero and non-zero readings throughout the fumigations. This was most likely due to instrument and operator error, and insufficient pump suction pressure of the SF monitoring instrument. Similar results have been reported anecdotally by professional fumigators.

- No PH<sub>3</sub> was detected outside of the silos at any time during the fumigations. This indicated that any PH<sub>3</sub> leaking to the outside was immediately diluted in the air to undetectable levels.
- In the bioassay containing PH<sub>3</sub> susceptible *S. zeamais* and *T. castaneum*, all adult insects were killed, and it is likely there was no emergence after six weeks of incubation (one dead adult was found but it was probably overlooked during the initial mortality count).
- Both SF and PH<sub>3</sub> were 100% effective in killing PH<sub>3</sub> resistant and PH<sub>3</sub> susceptible *R. dominica* and *T. castaneum* adults. Sulfuryl fluoride and PH<sub>3</sub> worked equally well to kill immature stages of PH<sub>3</sub> resistant strains of *R. dominica*, with only 3 emerged adults. Phosphine worked better to control emergence for *T. castaneum*, with 3 adults emerged compared to 10 from the SF treatment. The PH<sub>3</sub> resistant and susceptible strains of *R. dominica* were controlled equally well with both SF and PH<sub>3</sub>. There was greater emergence from the PH<sub>3</sub> susceptible strains of *T. castaneum* (31 adults) compared to the PH<sub>3</sub> resistant strain (12 adults) for both SF and PH<sub>3</sub>.
- Grain quality was affected by being stored in a non-aerated and non-vented silo. The test weight of corn dropped from approximately 77 to 65 kg/hL. In the top 0.3 m, corn moisture rose from 11.5 to 17% in both silos due to moisture equilibration with humid headspace air and moisture condensation over the duration of the storage period.
- After storing the corn for 6-7 months, mold was found in the topmost layer of corn (10 cm) in both silos. Condensation collected on the underside of the roof and dripped onto the grain surface as evidenced by the observed ring of mold directly underneath the support ring attached to the roof of the Bird's silo.

## Chapter 5 - Summary and Conclusions

Permanently sealing a corrugated steel silo after-market is time consuming and labor intensive. The additional time spent in sealing, checking for and fixing leaks, and installing the additional fumigation equipment was 90 man-hours. An experienced crew may be able to reduce this to about 25 man-hours for a similar sized silo, but additional equipment and sealing materials still add substantial cost to an unsealed silo. The additional equipment to seal the U.S. silo cost \$33/MT, or roughly 13% of the initial cost of the silo.

The total cost of modifying and sealing a corrugated steel hopper silo during construction to prepare it for fumigation on a permanent basis was estimated to be \$67/MT, or 25.7% of the list price. For an experienced work crew that cost may be lowered to \$42/MT, or approximately 16.2% of the list price. A considerable amount of money may be saved in the short term by temporarily sealing the silo prior to each fumigation which costs only \$1.50/MT for a smaller (60 MT) silo and even less for larger silos (\$0.04/MT for a 5,000 MT silo).

A grain manager must decide whether the cost of permanently sealing a grain silo is worth the potential gains in fumigation success and possibly lower long-term labor costs. Other considerations would be the safety aspects, as a permanently sealed silo would prevent the need for workers to climb in, around, and on grain silos to seal them.

The roof- and hopper-wall junctions, top lid, and overlapping roof ribs were areas of greatest leakage in the U.S. silo. Places where more than two sheets overlapped had more leaks than where only two sheets joined.

A hybrid approach to preparing structures for fumigation may be to permanently seal areas with greater leakage potential such as the roof-wall junction, and use temporary sealing for vents, fans, and other areas that are not conducive to being permanently sealed. The remaining leakage points could be quickly sealed prior to fumigation.

Neither the U.S. nor Australian silo had half-life pressure decay times that reached the 3-5 minutes prescribed by Australian Standard 2628. The longest half-life times were 50 s and 163 s for the U.S. and Australian silos, respectively. The half-life times were shorter after loading the silos with grain, suggesting the pressures from grain loading may have broken some of the seals and opened paths for air to travel through the silo wall seams.

The half-life times also decreased after 7-8 months through hot summer and cold winter weather. It is possible that exposure to temperatures above 35° C and below 0° C as experienced in Kansas, along with high winds and solar radiation deteriorated some of the sealant exposed to the elements.

The thermosiphon aided the distribution of PH<sub>3</sub> within the grain mass. With the thermosiphon, the minimum to maximum concentration ratio in the Australian silo reached 0.25 in less than 24 hours, but it took over 48 hours to reach 0.25 in the U.S. silo. The average concentration of PH<sub>3</sub> in the U.S. silo also seemed to be adversely affected by the lack of recirculation. It was unclear what effect the thermosiphon had with SF due to measurement errors in recording SF concentrations.

From an insect control point of view, the fumigations for trials 1 and 5 may be considered successful. All adult insects were killed during the fumigations, including the PH<sub>3</sub> resistant individuals. Emergence after 6 weeks was greatly reduced by fumigating with SF and PH<sub>3</sub> compared to the controls.

An alternative approach is to perform fumigations that kill adult insect pests only but allow other, less susceptible life stages to live. This may be reasonable immediately prior to marketing of grain to eliminate detectable live insects and to save on fumigation costs. However, this may provide selection pressure for PH<sub>3</sub> resistant insects. Grain handlers and managers should keep in mind the need to preserve PH<sub>3</sub> as a viable fumigant for the future.

The grain quality deteriorated in both silos over the storage period. Without adequate venting in the headspace, temperature fluctuations cause moisture-laden air to condense on the roof and drip onto the grain surface. This caused the moisture content to increase and the test weight to decrease in the top grain layer. Mold was visible in the top 10 cm of grain and was especially noticeable on the grain surface directly under the strengthening ring installed on the underside of the Australian silo roof.

## **Chapter 6 - Future Work and Recommendations**

A comparative fumigation cost/benefit analysis should be undertaken on larger bolted steel silos and flat bunker storage structures temporarily sealed versus permanently sealed. Some work has been done at Oklahoma State University on the cost saved in sealing a concrete elevator and using closed loop fumigation versus fumigating a silo as-is. They realized savings of \$0.50-\$1.50 per bushel in fumigation costs (Jones et al., unpublished data). This will not only be important for end users, but manufacturers seeking to not only minimize the cost of producing sealed silos, but also to convince their customers that the increased cost of sealed storage can be made up in lower fumigation and operating costs, as well as higher grain quality and quantity.

Currently, bolted steel silos manufactured in the U.S. are not designed to be airtight. Designing, manufacturing and constructing a silo that is readily sealable should be further explored because it would help ensure fumigation success and the continued efficacy of  $\text{PH}_3$ . Roof aeration vents should be sealable from the ground level. Components such as access doors, and inlet and discharge chutes should be redesigned for airtightness. The silo base to foundation interface should be permanently sealed, and the silo sidewall to roof interface should be sealable for airtightness during fumigations.

A three or five minute half-life pressure decay time is difficult to achieve on a bolted steel silo. More research needs to be undertaken to determine whether what the Australian Standard 2628 calls for is an appropriate and reasonable target, or whether it could be less to economize sealing (Casada and Noyes 2001) while also ensuring a successful fumigation.



It was unclear whether or not the thermosiphon aided recirculation of SF, or whether SF was too heavy to be carried by the relatively low air velocities generated passively via solar radiation on the thermosiphon pipe during day time hours. This should be further investigated. Given that CO<sub>2</sub> is heavier than PH<sub>3</sub> (44.01 g/mol and 33.99 g/mol, respectively), it would be worthwhile to also investigate whether thermosiphon technology could be used to distribute CO<sub>2</sub> when treating organic grains. One of the largest barriers to adoption when treating with CO<sub>2</sub> is the cost associated with the recirculation equipment (Noyes et al. 2002).

Sealing a silo for fumigation and aerating or venting a silo to maintain grain quality have opposite goals, i.e., preventing air exchange between the inside and outside of the silo.

Incorporating the ability to aerate the grain and vent the headspace of the silo after performing a fumigation in a sealed silo would be important to include in the design of a silo if grain is to be kept for longer periods of time.

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# **Appendix A - Australian Standard for Sealed Silos**

## **(AS2628-2010)**

### Foreword

Phosphine fumigation is commonly used to control insect pests in grain. The grains industry should retain this product in order to deliver insect and residue-free grain. Alternatives to phosphine are more expensive, more difficult to use, and some are less acceptable to markets.

The future availability and effectiveness of phosphine as a grain treatment is under threat on two fronts: (a) Insect resistance to phosphine is being found more frequently—all stages of the resistant insects can survive fumigation in unsealed silos. (b) If phosphine's good-safety record is not upheld, it could be withdrawn from some uses, including on-farm use.

The continued use of phosphine is vital to growers and others in the grains industry. It is the fumigation treatment preferred by most markets and no other treatment is as cost effective and easy to apply; however, insects resistant to phosphine are being found with increasing frequency. Using phosphine in unsealed silos will not kill all insects and will only lead to further selection of resistant insect strains. The use of sealed silos for effective fumigation is a key issue if phosphine is to be kept as a useful and active product in the long term.

Fumigation in a sealed silo passing a pressure test keeps the phosphine concentration high for long enough to control all known resistant insects.

A silo sealed to the standards required of phosphine treatment has the additional advantages that it may help protect fumigated grain from reinfestation and that it is available for treatment by carbon dioxide as used for "organic grain".

Where air inflow is incorporated (aeration) for grain conditioning during storage, a screen mesh should be used on air inlets and outlets to retain the integrity of the silos' insect-proof seal.

### Appendix B - Fumigation Concentration Data

Date	Location																							Avg of all points in bin	Avg of all points in a line					Avg of all points at a height				
	Headspace	Bottom	Line 1					Line 2				Line 3				Line 4				Line 5					Line					Level (1 is bottom, 5 is top)				
			1	2	3	4	5	1	2	3	4	1	2	3	4	1	2	3	4	1	2	3	4		1	2	3	4	5	1	2	3	4	5
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23		1	2	3	4	5	1	2	3	4	5
8/24/15 10:45 AM	35	272	0	0	0	9	19	0	0	0	15	0	0	0	15	0	0	0	14	0	0	0	23	4.52381	7	3.75	3.75	3.5	5.75	0	0	0	1.8	17.2
8/24/15 11:40 AM	56	228	0	0	0	29	42	0	0	0	40	0	0	0	47	0	0	4	43	0	0	4	49	12.28571	17.75	10	11.75	11.75	13.25	0	0	0	7.4	44.2
8/24/15 12:40 PM	100	284	0	0	0	61	73	0	0	17	77	0	0	16	88	8	2	27	77	0	0	26	78	26.19048	33.5	23.5	26	28.5	26	0	1.6	0.4	29.4	78.6
8/24/15 2:15 PM	122	370	0	0	13	87	98	0	2	39	102	0	0	47	115	13	3	56	106	0	3	53	104	40.04762	49.5	35.75	40.5	44.5	40	0	2.6	4.2	56.4	105
8/24/15 3:40 PM	389	735	5	4	41	122	130	5	9	69	132	4	4	85	127	20	10	89	135	5	9	84	142	58.61905	74.25	53.75	55	63.5	60	5	7.6	14.6	89.8	133.2
8/24/15 4:47 PM	220	945	9	4	55	142	162	4	16	89	150	5	7	90	140	28	16	101	150	5	16	106	160	69.28571	90.75	64.75	60.5	73.75	71.75	9	9.2	22	105.6	152.4
8/24/15 5:40 PM	194	1440	7	4	81	171	180	4	30	116	181	5	14	135	183	36	29	137	182	13	28	135	185	88.38095	109	82.75	84.25	96	90.25	7	12.4	36.4	138.8	182.2
8/24/15 6:45 PM	181	1300	9	4	100	181	179	5	42	136	179	6	19	155	179	19	37	153	178	19	39	157	168	93.52381	116	90.5	89.75	96.75	95.75	9	10.6	47.4	156.4	176.6
8/24/15 7:20 PM	160	176	600	8	107	170	163	4	46	142	163	6	21	160	164	49	43	163	166	25	42	160	162	122.0952	112	88.75	87.75	105.25	97.25	600	18.4	51.8	159	163.6
8/24/15 9:13 PM	142	268	565	10	97	159	154	4	15	129	153	7	11	149	135	42	42	156	145	148	38	147	139	116.4286	105	75.25	75.5	96.25	118	565	42.2	40.6	148	145.2
8/24/15 10:20 PM	128	128	815	845	96	139	123	745	115	116	95	158	99	133	103	134	69	139	131	128	37	128	120	212.7619	300.75	267.75	123.25	118.25	103.25	815	402	83.2	131	114.4
8/24/15 11:30 PM	120	139	765	730	100	108	120	745	450	105	78	314	388	93	86	208	160	123	119	410	240	113	108	264.9048	264.5	344.5	220.25	152.5	217.75	765	481.4	267.6	108.4	102.2
8/25/15 6:00 AM	199	195	384	402	565	292	256	474	505	408	310	266	290	420	414	168	218	250	152	274	388	264	149	326.1429	378.75	424.25	347.5	197	268.75	384	316.8	393.2	326.8	256.2
8/25/15 8:20 AM	288	615	424	428	510	374	324	478	452	460	306	270	276	366	362	173	222	250	212	234	370	282	222	333.0952	409	424	318.5	214.25	277	424	316.6	366	346.4	285.2
8/25/15 8:50 AM	292	540	404	414	480	330	302	466	436	420	286	268	270	358	332	181	182	216	232	236	364	252	252	318.1429	381.5	402	307	202.75	276	404	313	346.4	315.2	280.8
8/25/15 9:36 AM	308	490	376	396	448	290	284	440	430	364	280	250	262	338	307	198	208	228	270	244	358	232	296	309.4762	354.5	378.5	289.25	226	282.5	376	305.6	341.2	290.4	287.4
8/25/15 11:00 AM	372	490	382	384	404	322	334	436	426	326	334	244	266	308	340	234	234	266	338	262	360	268	342	324.2857	361	380.5	289.5	268	308	382	312	338	298	337.6
8/25/15 12:15 PM	384	482	356	356	350	352	358	426	402	328	358	242	268	312	364	254	242	312	360	268	336	314	360	329.4286	354	378.5	296.5	292	319.5	356	309.2	319.6	323.6	360
8/25/15 1:20 PM	400	476	338	334	332	366	370	416	374	344	370	238	268	334	378	268	254	344	370	268	318	342	370	333.1429	350.5	376	304.5	309	324.5	338	304.8	309.2	346	371.6
8/25/15 3:00 PM	476	460	318	310	344	374	372	396	358	356	372	234	272	356	376	290	274	358	376	268	308	354	374	335.2381	350	370.5	309.5	324.5	326	318	299.6	311.2	359.6	374
8/25/15 4:00 PM	424	505	302	290	350	378	380	382	336	358	376	230	276	358	374	296	288	362	372	272	308	358	374	334.2857	349.5	363	309.5	329.5	328	302	294	311.6	362.8	375.2
8/25/15 5:00 PM	394	580	298	282	358	376	376	370	338	362	372	230	284	360	372	304	298	362	368	278	312	362	366	334.6667	348	360.5	311.5	333	329.5	298	292.8	318	364.4	370.8
8/25/15 6:40 PM	380	855	280	264	354	364	366	344	334	354	364	230	292	354	362	308	310	356	360	288	318	354	360	329.3333	337	349	309.5	333.5	330	280	286.8	321.6	356.4	362.4
8/25/15 7:45 PM	370	356	376	260	342	350	350	326	324	344	348	228	280	344	348	304	302	346	346	286	308	342	342	323.619	325.5	335.5	300	324.5	319.5	376	280.8	311.2	345.2	346.8
8/25/15 8:30 PM	350	342	446	284	328	334	330	314	306	326	328	222	256	328	328	292	284	330	330	276	292	326	326	313.619	319	318.5	283.5	309	305	446	277.6	293.2	328.8	328.4
8/25/15 9:45 PM	326	314	384	392	302	312	308	366	278	306	304	228	232	308	294	282	274	310	308	310	268	304	304	303.5238	328.5	313.5	265.5	293.5	296.5	384	315.6	270.8	308	303.6
8/25/15 10:45 PM	330	322	370	374	294	308	306	384	288	300	296	246	240	300	282	288	274	306	304	324	274	298	298	302.5714	320.5	317	267	293	298.5	370	323.2	274	302.4	297.2
8/25/15 11:30 PM	312	308	346	350	280	292	292	368	300	288	284	248	248	286	268	282	272	294	294	316	278	288	288	293.4286	303.5	310	262.5	285.5	292.5	346	312.8	275.6	289.6	285.2
8/26/15 7:00 AM	326	322	332	326	334	308	308	324	324	316	312	264	280	294	290	280	282	288	290	282	290	286	284	299.7143	319	319	282	285	285.5	332	295.2	302	298.4	296.8
8/26/15 8:00 AM	304	412	302	296	308	292	290	298	304	300	294	246	262	278	274	268	268	276	280	268	276	274	276	282.381	296.5	299	265	273	273.5	302	275.2	283.6	284	282.8
8/26/15 9:00 AM	304	284	292	292	302	288	286	302	302	298	292	248	262	280	274	274	272	278	282	266	278	276	276	281.9048	292	298.5	266	276.5	274	292	276.4	283.2	284	282
8/26/15 10:00 AM	310	296	292	302	300	292	286	308	306	300	290	252	262	274	278	276	268	280	284	274	276	278	284	283.9048	295	301	266.5	277	278	292	282.4	282.4	284.8	284.4
8/26/15 11:00 AM	294	288	278	278	286	278	272	290	286	282	280	242	254	264	264	268	260	276	278	266	268	270	278	272.2857	278.5	284.5	256	270.5	270.5	278	268.8	270.8	274	274.4
8/26/15 12:15 PM	296	284	278	274	280	282	282	290	286	282	280	242	252	264	278	270	262	276	278	262	268	272	280	273.2381	279.5	284.5	259	271.5	270.5	278	267.6	269.6	275.2	279.6
8/26/15 2:30 PM	278	272	272	270	280	280	278	288	280	280	278	244	256	274	278	272	266	278	276	266	268	276	274	273.0476	277	281.5	263	273	271	272	268	270	277.6	276.8
8/26/15 4:45 PM	270	276	266	262	274	264	260	276	270	268	258	238	254	262	258	264	260	262	256	258	260	260	254	261.1429	265	268	253	260.5	258	266	259.6	263.6	263.2	257.2
8/26/15 7:15 PM	258	264	258	254	258	248	246	264	258	252	244	236	250	248	244	256	252	244	242	252	252	244	240	249.619	251.5	254.5	244.5	248.5	247	258	252.4	254	247.2	243.2
8/26/15 9:45 PM	244	242	254	244	238	232	226	246	240	234	232	224	226	230	230	238	234	228	230	240	234	228	226	234	235	235	238	227.5	232.5	232	254	238.4		

Date	Location																						
	Headspace	Bottom	Line 1					Line 2				Line 3				Line 4				Line 5			
			1	2	3	4	5	1	2	3	4	1	2	3	4	1	2	3	4	1	2	3	4
8/24/15 10:45 AM	19	945	2	0	0	0	9	0	0	0	9	0	0	0	7	0	0	0	14	0	0	0	16
8/24/15 11:40 AM	80	1020	4	3	3	31	59	0	0	0	69	0	0	0	70	0	0	0	67	0	0	0	62
8/24/15 12:40 PM	118	955	4	3	5	102	126	3	3	16	108	3	2	21	128	2	3	21	136	3	2	18	122
8/24/15 2:15 PM	164	1040	3	4	29	149	160	4	4	62	143	4	4	77	162	6	4	75	165	5	4	63	155
8/24/15 3:40 PM	180	1670	10	5	83	172	175	5	3	115	170	8	5	128	165	7	5	126	178	0	5	103	160
8/24/15 4:47 PM	171	1628	15	9	93	170	179	7	5	133	155	14	8	131	161	7	6	130	167	3	5	130	184
8/24/15 5:40 PM	202	2000	9	7	119	192	202	8	6	144	200	20	13	152	197	11	11	156	198	5	9	155	194
8/24/15 6:45 PM	182	1910	190	10	121	187	180	10	6	144	180	24	15	155	183	15	15	159	181	5	13	166	179
8/24/15 7:30 PM	174	276	1340	10	122	180	172	11	8	144	171	26	16	152	175	19	18	164	173	5	15	165	174
8/24/15 9:30 PM	160	166	1420	177	92	165	160	37	10	105	147	30	20	106	150	20	24	151	153	484	20	149	125
8/24/15 10:42 PM	151	153	1290	655	69	146	145	117	30	76	130	32	30	71	135	248	26	142	145	890	106	117	114
8/24/15 11:55 PM	142	145	1150	970	60	118	118	208	121	53	123	42	76	47	126	334	181	137	138	975	306	95	120
8/25/15 6:00 AM	137	138	835	835	775	230	176	268	418	448	133	82	218	324	134	220	392	240	128	515	615	262	129
8/25/15 8:30 AM	250	1410	755	760	750	334	278	187	374	476	184	109	191	324	186	228	364	272	181	372	570	338	184
8/25/15 9:15 AM	294	1020	590	705	705	334	288	164	338	464	218	120	174	320	214	144	256	276	230	346	535	350	250
8/25/15 10:03 AM	334	855	406	615	655	328	318	162	300	450	308	138	162	318	298	152	232	288	320	352	530	388	332
8/25/15 11:30 AM	374	880	272	484	585	346	362	179	270	414	362	168	162	306	366	175	214	300	364	372	525	420	360
8/25/15 12:45 PM	400	1070	210	374	478	368	378	195	252	374	386	188	170	320	390	199	212	326	390	374	510	392	380
8/25/15 1:50 PM	422	960	192	314	406	402	408	212	252	374	400	214	192	372	426	222	224	368	422	352	476	376	408
8/25/15 3:20 PM	434	1050	192	268	382	420	426	222	252	390	420	220	206	392	424	230	236	390	424	320	428	388	
8/25/15 4:15 PM	430	1220	193	252	386	424	424	226	250	396	418	224	214	398	424	236	240	398	424	302	396	396	422
8/25/15 5:15 PM	432	1570	191	238	390	420	422	228	250	400	420	222	224	400	422	244	248	402	422	278	268	402	416
8/25/15 7:00 PM	422	2000	196	224	380	410	410	226	248	392	408	232	232	392	410	254	260	398	408	250	334	398	408
8/25/15 8:00 PM	400	404	1700	220	370	392	386	224	240	380	386	234	228	378	390	252	260	386	386	232	312	388	386
8/25/15 9:00 PM	376	374	995	202	348	370	366	216	228	356	364	222	218	354	364	254	250	368	364	306	284	366	344
8/25/15 10:00 PM	360	360	785	360	322	354	352	222	214	328	340	220	214	316	344	302	246	346	348	555	266	336	324
8/25/15 11:15 PM	354	352	710	515	304	332	336	240	214	306	328	214	214	288	334	334	258	330	342	630	296	312	312
8/25/15 11:45 PM	342	342	660	560	288	314	318	248	220	280	284	318	206	216	266	342	274	314	334	625	326	296	308
8/26/15 7:15 AM	322	322	454	454	464	316	308	248	302	344	306	228	252	294	304	308	338	318	308	366	436	316	302
8/26/15 8:15 AM	318	890	432	418	440	320	312	232	284	334	302	232	250	288	302	296	322	314	306	322	406	322	304
8/26/15 9:15 AM	330	530	338	392	422	324	308	234	280	332	304	242	250	292	310	304	324	320	314	310	392	328	310
8/26/15 10:10 AM	340	408	290	364	408	330	332	246	268	334	320	252	252	296	320	296	306	320	328	310	384	334	326
8/26/15 11:15 AM	334	386	260	330	386	326	328	246	264	326	324	240	252	292	326	294	300	316	328	306	372	332	326
8/26/15 12:30 PM	334	360	250	308	368	328	328	252	262	322	328	242	258	302	330	296	298	318	330	308	364	334	328
8/26/15 2:45 PM	328	336	240	286	328	326	326	260	266	320	322	240	264	318	324	300	298	322	322	298	344	324	322
8/26/15 5:00 PM	308	336	234	268	318	308	304	250	262	312	304	212	264	310	304	292	292	312	306	280	320	310	304
8/26/15 7:30 PM	294	340	220	256	302	292	292	236	254	296	288	214	256	294	288	290	282	294	288	264	308	296	286
8/26/15 10:00 PM	274	276	322	232	278	272	270	220	232	272	262	204	234	270	268	262	270	272	268	240	266	262	254
8/27/15 8:00 AM	248	272	242	232	224	226	216	226	185	206	232	182	196	214	240	236	234	236	244	238	236	204	226
8/27/15 9:30 AM	242	210	196	212	220	226	240	187	185	206	234	178	197	214	240	232	228	234	240	228	228	206	236
8/27/15 11:15 AM	234	195	181	202	216	232	232	143	197	214	234	228	224	230	232	226	224	230	230	218	220	212	230
8/27/15 4:45 PM	197	167	160	188	210	198	197	161	181	206	195	108	189	204	196	212	210	204	195	195	204	202	202
8/27/15 10:00 PM	175	175	129	164	184	173	174	143	159	179	168	122	162	180	172	186	188	178	173	168	181	164	168
8/28/15 5:35 PM	129	112	109	123	133	129	129	114	128	131	128	114	128	131	128	136	135	131	129	122	131	130	128
8/29/15 4:30 PM	101	85	82	87	91	99	100	88	88	92	100	97	95	98	100	96	96	97	100	77	77	91	99

Figure 6.2 PH<sub>3</sub> concentration data from trial 1 on August 24-29, 2015 in the Bird's silo.



Date	Location																						
	Headspace	Bottom	Line 1					Line 2				Line 3				Line 4				Line 5			
			1	2	3	4	5	1	2	3	4	1	2	3	4	1	2	3	4	1	2	3	4
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	
8/31/15 11:15 AM	17	1080	5	3	10	0	14	11	5	11	4	15	7	9	13	16	14	4	29	17	12	8	22
8/31/15 1:00 PM	750	960	10	7	8	101	143	17	12	13	146	14	8	17	182	22	12	33	163	16	11	27	160
8/31/15 2:30 PM	1130	1110	12	8	58	220	280	9	12	83	238	13	8	114	278	10	13	158	278	14	15	135	278
8/31/15 4:00 PM	378	1230	13	10	155	320	348	14	56	163	340	14	28	208	344	12	64	260	342	17	58	242	352
8/31/15 5:15 PM	390	1400	13	10	250	364	378	11	137	244	374	22	87	286	378	23	144	324	380	20	141	314	378
8/31/15 6:00 PM	386	1470	14	13	290	370	388	15	194	276	396	15	137	316	384	35	204	348	382	54	198	338	380
8/31/15 7:10 PM	414	1410	12	17	330	388	410	28	242	292	400	21	178	326	388	67	246	356	386	86	244	312	392
8/31/15 8:30 PM	426	2000	23	27	344	400	400	41	272	300	388	31	204	326	380	98	287	358	386	131	274	346	388
8/31/15 9:30 PM	380	2000	28	36	348	368	376	44	250	278	364	34	190	308	364	129	256	338	366	119	259	326	366
8/31/15 10:45 PM	332	2000	47	50	340	344	326	57	284	282	328	44	206	310	318	188	294	344	334	178	294	332	334
9/1/15 7:15 AM	294	2000	1200	1320	274	256	278	945	240	244	240	805	214	244	214	402	272	274	264	468	280	274	268
9/1/15 8:00 AM	294	1690	1120	1170	264	242	264	785	254	228	232	685	236	228	208	328	264	264	258	372	266	260	260
9/1/15 10:50 AM	456	700	775	640	286	434	448	378	250	268	442	346	250	260	446	318	268	380	444	280	266	362	444
9/1/15 1:15 PM	456	484	442	368	434	430	414	318	346	386	422	290	290	386	424	272	368	430	410	270	362	424	408
9/1/15 2:15 PM	424	408	346	310	418	380	390	296	398	388	384	272	350	386	384	296	404	396	380	298	402	394	380
9/1/15 3:30 PM	386	406	306	314	390	374	368	304	396	368	368	272	376	368	362	330	392	374	364	338	394	374	362
9/1/15 4:45 PM	370	420	310	340	374	358	356	326	382	360	356	284	374	354	352	344	376	358	352	354	378	360	350
9/1/15 6:30 PM	368	448	350	374	364	356	354	354	368	354	350	312	362	346	346	356	358	348	344	364	358	346	342
9/1/15 8:40 PM	348	505	352	366	344	336	328	348	340	336	328	322	336	328	324	342	334	328	322	346	334	330	322
9/1/15 10:10 PM	332	482	356	356	332	322	318	344	330	330	318	326	328	320	314	332	324	318	308	336	322	320	308
9/2/15 12:00 AM	322	438	348	346	320	308	306	336	320	320	308	322	320	312	306	324	316	308		296	326	314	296
9/2/15 7:30 AM	292	290	306	306	282	282	282	302	286	290	284	292	286	284	282	288	278	278	276	288	276	278	274
9/2/15 9:10 AM	282	248	296	302	274	274	264	292	276	280	274	280	280	278	274	278	278	272	266	280	270	274	268
9/2/15 10:20 AM	276	274	292	288	270	268	264	284	274	268	268	276	270	272	264	272	266	266	260	278	266	268	260
9/2/15 2:50 PM	258	248	268	256	256	250	248	268	256	258	248	262	260	254	246	260	254	252	246	260	254	250	246
9/2/15 4:45 PM	240	242	258	252	242	236	234	254	244	242	234	252	246	240	232	250	242	236	232	248	242	236	230
9/2/15 7:15 PM	236	220	250	246	234	230	230	246	234	234	228	244	232	230	226	240	230	226	220	238	228	228	226
9/2/15 9:30 PM	228	200	242	236	226	224	220	234	222	224	218	232	222	222	216	226	220	218	294	220	218	218	214
9/3/15 1:00 AM	216	214	230	224	214	210	210	222	216	210	212	220	214	214	210	216	212	210	204	214	210	208	204
9/2/15 9:00 AM	202	184	200	199	195	196	195	192	194	194	193	184	191	192	191	189	190	191	191	189	189	190	189
9/2/15 10:30 AM	192	175	197	191	191	190	188	189	189	189	188	183	188	188	185	186	187	187	186	186	186	186	185
9/2/15 12:00 PM	182	164	191	189	187	184	174	188	187	185	182	181	188	185	183	190	189	187	183	187	186	183	181
9/2/15 1:00 PM	175	171	188	182	183	179	170	183	183	176	171	180	183	181	178	185	184	182	175	182	183	179	171
9/2/15 2:00 PM	177	175	182	179	182	175	166	172	173	171	167	176	177	175	166	176	171	177	170	172	171	169	167
9/2/15 3:00 PM	168	163	180	176	173	165	163	171	169	168	164	168	169	170	163	169	168	171	165	171	168	166	165
9/4/15 1:50 PM	118	113	125	125	123	121	121	126	124	123	121	124	124	123	120	125	124	122	119	124	122	122	119
9/6/15 3:00 PM	58	56	59	58	59	56	56	58	57	57	55	57	57	56	56	58	57	56	56	57	57	55	55
9/9/15 11:30 AM	23	22	23	23	23	23	23	23	23	23	23	21	22	22	23	22	23	23	22	22	22	22	22

Figure 6.3 PH<sub>3</sub> concentration data from trial 2 on August 31-September 9, 2015 in the SCAFCO silo.

Date	Location																						
	Headspace	Bottom	Line 1					Line 2				Line 3				Line 4				Line 5			
			1	2	3	4	5	1	2	3	4	1	2	3	4	1	2	3	4	1	2	3	4
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	
8/31/15 12:01 PM	96	1660	25	18	20	25	73	18	19	16	92	21	28	20	94	20	24	16	92	11	11	14	97
8/31/15 1:30 PM	204	1740	25	20	23	155	196	22	20	39	180	21	25	48	210	19	23	48	214	13	14	41	210
8/31/15 2:55 PM	324	1970	24	20	73	256	282	19	19	154	278	18	23	169	294	20	28	171	286	15	15	154	294
8/31/15 4:15 PM	360	2000	24	23	165	306	324	18	23	258	344	23	30	262	334	19	32	260	342	16	28	254	348
8/31/15 5:30 PM	372	2000	23	24	232	354	392	21	37	294	368	19	52	298	374	22	63	300	376	18	58	296	384
8/31/15 6:30 PM	390	2000	30	28	262	370	378	22	55	316	368	24	75	274	384	30	93	324	394	20	86	320	398
8/31/15 7:30 PM	388	2000	20	36	288	382	388	33	77	334	382	30	100	340	382	36	124	348	388	22	114	340	396
8/31/15 9:00 PM	356	2000	28	46	306	368	356	49	100	340	356	54	126	346	360	51	159	360	354	25	143	348	
8/31/15 9:50 PM	342	2000	44	44	282	346	336	48	92	334	336	61	134	342	344	79	171	356	336	28	152	344	334
8/31/15 11:10 PM	308	2000	412	50	304	316	302	74	121	330	306	72	149	334	310	106	192	340	308	65	168	324	302
9/1/15 7:45 AM	264	2000	1960	452	238	222	244	199	159	234	244	123	165	248	250	376	230	256	254	1050	274	206	238
9/1/15 8:10 AM	292	2000	1520	442	230	214	246	191	160	228	254	121	165	242	252	352	230	248	260	1010	276	204	250
9/1/15 11:20 AM	515	2000	1320	368	224	218	242	164	140	234	254	114	166	322	250	316	224	226	248	785	248	204	424
9/1/15 1:25 PM	545	1570	214	224	450	515	525	185	226	398	505	151	248	505	535	238	276	498	535	406	266	486	515
9/1/15 2:40 PM	535	1290	194	238	494	530	530	196	294	515	515	161	332	525	530	246	364	520	525	302	348	505	525
9/1/15 4:00 PM	520	1606	197	286	510	520	525	222	362	520	525	182	396	520	515	280	426	515	515	272	410	510	515
9/1/15 5:00 PM	515	1480	208	324	510	520	515	248	400	515	505	204	424	510	510	306	452	510	510	276	438	510	515
9/1/15 6:40 PM	515	1830	246	378	500	500	505	294	436	500	498	246	446	498	494	354	468	498	500	306	460	498	500
9/1/15 8:50 PM	478	2000	266	398	480	474	466	324	438	476	464	284	436	474	462	378	462	474	462	336	456	468	458
9/1/15 10:30 PM	444	2000	346	404	468	444	432	334	430	458	430	288	422	452	422	384	454	456	428	360	448	438	420
9/2/15 12:10 AM	420	905	410	408	458	424	414	344	426	436	412	286	414	432	398	404	446	434	410	422	440	406	398
9/2/15 7:50 AM	380	386	610	386	382	370	374	344	374	358	364	274	350	352	348	444	390	376	370	625	380	340	356
9/2/15 9:30 AM	404	1560	416	372	368	362	378	330	362	350	374	252	340	342	358	416	378	370	378	580	370	340	380
9/2/15 10:35 AM	422	1190	352	364	362	382	396	322	356	350	394	240	328	340	388	394	362	362	394	525	360	344	392
9/2/15 3:00 PM	408	610	322	358	406	406	402	316	360	406	400	226	346	406	402	358	380	408	398	376	374	402	398
9/2/15 5:00 PM	276	560	314	362	390	378	374	314	374	382	370	230	362	382	372	352	384	380	370	348	382	380	372
9/2/15 7:25 PM	358	575	322	370	368	356	354	324	368	358	350	262	358	358	350	356	368	356	350	354	368	354	348
9/2/15 9:45 PM	340	855	308	352	342	336	334	314	344	336	332	280	334	332	322	342	346	338	330	344	344	332	328
9/3/15 1:10 AM	320	350	346	334	326	318	316	298	324	312	308	272	314	304	288	326	328	322	312	338	322	298	302
9/3/15 9:25 AM	300	298	272	292	290	294	296	262	278	274	294	242	268	264	288	298	292	292	292	306	280	276	290
9/3/15 10:45 AM	292	278	268	286	286	290	290	258	272	284	288	234	262	276	286	288	288	290	286	292	278	282	286
9/3/15 12:20 PM	282	292	268	284	286	282	276	258	274	276	278	236	264	280	278	300	296	300	290	296	276	282	278
9/3/15 1:20 PM	268	282	264	276	282	274	264	254	270	272	270	220	258	274	274	286	284	290	278	286	276	280	278
9/3/15 2:20 PM	260	262	262	268	272	264	256	240	268	268	266	216	258	272	264	274	284	276	272	280	274	268	268
9/3/15 3:20 PM	254	252	260	260	268	262	252	240	262	262	258	210	258	264	258	268	274	270	264	278	274	266	258
9/4/15 2:00 PM	177	172	176	186	182	178	176	172	183	179	175	160	179	179	176	187	184	179	176	183	184	178	175
9/6/15 3:20 PM	68	72	73	73	71	69	69	72	72	70	69	64	70	70	68	75	72	70	69	74	72	70	68
9/9/15 12:10 PM	27	27	26	26	27	27	27	25	26	27	27	25	26	27	27	27	27	27	27	27	26	27	27

Figure 6.4 PH<sub>3</sub> concentration data from trial 2 on August 31-September 9, 2015 in the Bird's silo.

Date	Location																						
	Headspace	Bottom	Line 1					Line 2				Line 3				Line 4				Line 5			
			1	2	3	4	5	1	2	3	4	1	2	3	4	1	2	3	4	1	2	3	4
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	
9/18/2015 10:00	11	240	0	0	0	3	9	0	0	0	2	0	0	0	3	0	0	0	9	0	0	0	8
9/18/2015 11:30	9	89	3	0	0	7	9	0	0	0	5	0	0	0	6	0	0	3	9	0	0	0	9
9/18/2015 12:30	8	51	8	0	2	8	8	0	0	0	6	0	0	0	7	0	0	7	8	0	0	2	8
9/18/2015 13:45	8	40	82	0	5	7	7	5	0	0	4	0	0	0	4	0	2	7	7	0	0	3	7
9/18/2015 14:45	7	30	200	3	6	6	6	15	0	0	0	3	0	0	2	0	3	7	7	3	0	3	6
9/18/2015 16:45	6	25	565	24	7	3	4	81	2	0	0	15	0	0	0	0	4	0	6	8	3	3	5
9/18/2015 18:00	5	21	750	80	7	3	4	138	3	0	2	29	2	0	0	0	5	5	5	9	4	3	5
9/18/2015 21:45	4	17	1170	414	8	4	5	292	38	9	5	77	19	4	3	6	5	5	17	5	3	3	
9/18/2015 23:00	4	15	1090	550	8	6	5	306	78	16	7	80	38	9	5	8	5	5	4	23	6	4	4
9/19/2015 9:00	57	12	1070	705	236	142	89	153	496	286	169	76	168	192	149	27	25	23	36	29	54	27	34
9/19/2015 11:00	68	54	735	590	236	164	102	159	480	312	220	70	133	165	143	42	32	35	48	33	65	35	43
9/19/2015 12:45	80	65	635	412	206	163	97	175	436	324	246	68	106	140	129	50	37	45	58	35	65	40	50
9/19/2015 13:45	91	72	585	330	175	155	100	194	402	330	254	67	93	124	119	55	42	52	69	40	60	43	60
9/19/2015 15:15	108	75	625	276	148	140	105	218	360	330	244	67	86	112	110	62	48	61	89	46	58	47	80
9/19/2015 16:30	121	89	830	254	124	132	115	232	300	306	240	65	79	102	107	67	52	69	102	50	56	93	95
9/19/2015 18:30	133	105	970	308	95	142	131	276	224	266	252	70	75	94	104	76	59	88	115	58	54	59	104
9/19/2015 22:15	137	105	1010	458	101	152	139	296	236	192	191	81	94	84	93	103	77	109	120	84	62	82	116
9/20/2015 9:00	150	115	1000	765	282	173	147	216	466	310	202	126	183	224	204	119	113	113	122	118	118	108	121
9/20/2015 10:00	155	124	735	665	288	188	152	199	434	308	230	126	172	210	204	124	115	117	126	122	123	120	122
9/20/2015 11:00	164	151	880	620	290	210	167	216	442	336	272	131	163	200	202	132	121	124	130	124	131	118	125
9/20/2015 15:30	180	139	200	210	218	210	177	268	374	364	302	138	152	172	179	146	134	141	161	132	135	130	153
9/20/2015 16:45	182	152	195	175	195	200	178	264	304	338	286	137	147	165	173	147	136	145	166	133	134	131	159
9/20/2015 19:30	184	162	188	162	173	202	188	242	210	278	282	137	141	158	167	154	140	157	170	137	133	137	160
9/20/2015 21:45	185	166	555	175	163	199	188	216	177	222	240	136	138	148	158	158	144	161	167	142	133	142	161
9/21/2015 8:45	165	163	975	665	216	155	155	195	384	248	189	152	202	206	180	150	150	148	148	149	153	144	147
9/21/2015 10:00	157	161	780	575	230	164	155	190	364	272	204	149	194	208	188	145	145	143	127	144	149	142	142
9/21/2015 15:15	176	156	286	238	236	206	177	216	300	282	260	156	179	204	202	150	147	150	157	148	150	150	154
9/21/2015 20:00	180	150	198	174	189	192	180	206	200	230	234	150	157	182	188	153	148	159	167	149	146	150	161
9/23/2015 9:00	138	125	206	152	126	126	130	130	130	129	129	124	125	125	126	128	127	130	131	127	125	127	130
9/23/2015 16:45	127	127	134	129	121	124	123	128	127	122	123	121	120	119	122	123	121	123	123	122	120	121	121
9/24/2015 13:45	104	95	85	87	98	87	100	99	93	98	99	97	96	98	98	99	98	99	100	99	97	98	99

Figure 6.5 PH<sub>3</sub> concentration data from trial 3 on September 18-24, 2015 in the SCAFCO silo.

Date	Location																						
	Headspace	Bottom	Line 1					Line 2				Line 3				Line 4				Line 5			
			1	2	3	4	5	1	2	3	4	1	2	3	4	1	2	3	4	1	2	3	4
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	
9/18/2015 10:15	47	1510	3	0	0	5	31	0	0	0	28	0	0	0	24	0	0	0	32	0	0	0	31
9/18/2015 11:45	86	785	3	0	0	50	87	0	0	0	84	0	0	3	90	0	0	2	84	0	0	0	81
9/18/2015 12:45	119	1500	4	2	14	90	119	0	0	13	115	0	0	23	114	0	0	17	105	0	0	6	114
9/18/2015 14:00	116	125	1670	5	34	91	110	0	0	20	99	0	0	41	108	2	0	34	106	14	0	10	81
9/18/2015 15:00	108	93	2000	8	43	70	99	14	0	20	78	0	3	45	101	44	4	39	97	310	4	10	55
9/18/2015 17:00	89	91	1710	214	39	22	65	284	8	12	66	14	5	38	87	468	11	36	85	1170	59	9	59
9/18/2015 18:15	78	79	2000	466	35	19	57	442	45	12	62	39	10	36	77	530	42	39	75	1280	218	13	57
9/18/2015 22:00	60	60	930	1060	54	44	47	730	378	57	55	111	103	32	57	585	336	40	57	1020	920	162	62
9/18/2015 23:15	59	59	895	895	109	112	75	625	500	130	59	118	170	42	52	460	444	53	55	815	920	316	67
9/19/2015 9:30	370	1260	565	745	650	476	400	240	366	356	306	193	208	236	304	224	342	258	312	366	680	498	316
9/19/2015 11:15	418	1060	318	530	570	460	414	234	298	344	350	264	216	258	338	272	288	276	368	336	630	525	380
9/19/2015 13:00	442	1100	254	378	474	428	426	254	270	338	390	300	240	284	380	314	272	296	404	312	540	515	414
9/19/2015 14:00	470	1020	254	320	416	420	450	276	272	334	434	314	264	304	434	342	280	312	446	302	468	490	446
9/19/2015 15:30	480	1340	268	296	374	444	472	292	280	332	458	344	292	322	466	360	292	328	468	294	398	456	466
9/19/2015 16:45	486	685	785	290	362	446	470	296	284	330	454	312	310	332	460	372	304	336	460	286	348	422	452
9/19/2015 18:45	458	460	1860	308	346	396	426	282	282	312	382	310	318	332	414	374	316	338	414	368	306	360	376
9/19/2015 22:30	392	392	905	912	322	304	344	380	368	288	354	310	318	330	376	408	404	338	370	685	785	308	334
9/20/2015 9:15	408	408	610	505	488	420	416	326	344	358	378	346	326	334	376	366	368	356	378	360	438	400	376
9/20/2015 10:30	424	605	424	462	464	426	418	344	346	362	388	372	344	352	392	380	376	368	396	366	448	414	392
9/20/2015 11:15	428	630	344	394	440	424	418	336	350	364	392	318	358	360	390	390	374	375	400	354	438	418	398
9/20/2015 15:45	432	595	328	354	394	416	424	364	362	370	416	343	376	382	418	408	386	386	418	344	376	396	414
9/20/2015 17:00	426	775	324	350	384	406	414	354	360	366	404	310	372	376	406	400	382	380	406	336	362	378	400
9/20/2015 19:45	408	408	645	336	368	368	386	318	342	354	374	294	350	368	386	388	374	372	385	338	346	330	322
9/20/2015 22:00	386	386	490	388	358	336	358	302	322	342	364	318	334	360	376	382	372	368	372	378	374	318	306
9/21/2015 9:00	330	330	344	324	332	328	322	276	286	294	314	268	290	306	318	322	322	324	318	272	312	308	308
9/21/2015 10:15	320	320	332	306	318	314	310	266	276	289	304	272	280	296	310	314	314	316	310	254	302	300	302
9/21/2015 15:30	320	288	254	278	306	308	310	276	284	286	302	250	292	296	308	314	312	312	312	252	288	294	302
9/21/2015 20:15	302	302	250	256	286	286	294	256	268	274	288	240	278	286	294	300	294	298	296	238	264	262	276
9/23/2015 9:30	192	171	163	171	190	183	190	174	170	174	187	177	183	191	191	196	195	195	192	174	184	181	188
9/23/2015 17:00	177	158	159	170	179	177	175	168	172	175	175	162	178	179	175	183	182	181	174	169	171	171	177
9/24/2015 14:00	145	129	127	134	138	137	144	134	132	133	144	126	140	143	145	148	147	147	145	124	136	133	143

Figure 6.6 PH<sub>3</sub> concentration data from trial 3 on September 18-24, 2015 in the Bird's silo.

Date	Location																						
	Headspace	Bottom	Line 1					Line 2				Line 3				Line 4				Line 5			
			1	2	3	4	5	1	2	3	4	1	2	3	4	1	2	3	4	1	2	3	4
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	
4/1/16 1:30 PM	0	57121.52	30098.83	851.8536	118.313	0	0	4685.195	0	0	0	2366.26	0	0	0	3076.138	0	0	0	4353.918	0	0	0
4/1/16 2:30 PM	47.3252	0	3312.764	0	0	0	0	3857.004	0	70.9878	0	0	0	0	0	2934.162	0	0	0	3857.004	0	0	0
4/1/16 4:00 PM	47.3252	6507.215	6696.516	47.3252	23.6626	23.6626	23.6626	3289.101	47.3252	354.939	141.9756	2531.898	94.6504	23.6626	0	1632.719	0	0	0	3005.15	0	94.6504	0
4/1/16 6:00 PM	118.313	3880.666	2176.959	0	0	0	0	1869.345	0	354.939	331.2764	1751.032	94.6504	307.6138	0	946.504	0	0	0	1893.008	0	283.9512	0
4/1/16 8:00 PM	141.9756	0	0	94.6504	473.252	709.878	591.565	1774.695	0	0	0	1490.744	0	236.626	307.6138	1490.744	236.626	260.2886	0	2366.26	0	0	94.6504
4/1/16 10:00 PM	260.2886	260.2886	165.6382	354.939	709.878	449.5894	496.9146	47.3252	212.9634	567.9024	544.2398	473.252	94.6504	94.6504	141.9756	0	0	0	0	118.313	189.3008	331.2764	0
4/2/16 8:00 AM	449.5894	425.9268	0	141.9756	141.9756	165.6382	189.3008	212.9634	165.6382	0	0	0	0	70.9878	0	0	0	0	0	0	0	0	0
4/2/16 12:00 PM	449.5894	165.6382	0	47.3252	47.3252	141.9756	141.9756	236.626	141.9756	189.3008	70.9878	165.6382	0	0	0	0	0	0	47.3252	0	0	0	0
4/2/16 4:00 PM	425.9268	402.2642	236.626	307.6138	331.2764	331.2764	378.6016	425.9268	0	0	0	0	0	0	0	0	70.9878	141.9756	165.6382	0	0	0	0
4/2/16 8:00 PM	378.6016	378.6016	260.2886	331.2764	307.6138	0	0	47.3252	47.3252	0	0	0	0	0	0	0	0	0	0	23.6626	0	23.6626	0
4/3/16 8:30 AM	307.6138	307.6138	165.6382	260.2886	23.6626	23.6626	47.3252	47.3252	0	0	0	0	0	0	0	0	0	0	23.6626	23.6626	0	0	0
4/3/16 12:30 PM	331.2764	283.9512	189.3008	236.626	236.626	283.9512	307.6138	0	0	0	0	0	0	0	0	0	0	0	141.9756	141.9756	94.6504	141.9756	0
4/3/16 5:00 PM	283.9512	283.9512	165.6382	212.9634	236.626	70.9878	70.9878	70.9878	23.6626	70.9878	0	0	0	0	0	0	23.6626	0	47.3252	47.3252	0	0	0
4/3/16 8:00 PM	260.2886	260.2886	118.313	236.626	0	0	47.3252	47.3252	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
4/4/16 10:00 AM	212.9634	212.9634	141.9756	47.3252	47.3252	70.9878	0	0	0	0	0	0	0	0	47.3252	0	0	0	94.6504	23.6626	0	0	0
4/4/16 2:30 PM	0	0	0	47.3252	0	23.6626	0	0	0	0	0	0	0	0	47.3252	0	0	0	0	0	0	0	0
4/5/16 3:30 PM	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
4/6/16 2:00 PM	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0

Figure 6.7 SF concentration data from trial 4 on April 1-6, 2016 in the SCAFCO silo.

Date	Location																						
	Headspace	Bottom	Line 1					Line 2				Line 3				Line 4				Line 5			
			1	2	3	4	5	1	2	3	4	1	2	3	4	1	2	3	4	1	2	3	4
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	
4/1/2016 12:30	197	75	472	2000	2000	2000	751	65	2000	2000	1490	28	2000	1430	2000	56	2000	2000	1380	595	2000	2000	2000
4/1/16 1:30 PM	1420	2000	2000	2000	2000	1570	1400	2000	2000	2000	1290	1570	2000	1140	1870	2000	2000	1970	1150	2000	2000	2000	1110
4/1/16 2:30 PM	1280	2000	1640	1470	1250	1100	1160	1780	1680	1630	975	1540	1330	870	1030	1460	1280	1160	870	1470	1370	815	845
4/1/16 4:00 PM	1130	905	800	765	765	715	695	1030	1020	905	680	910	855	645	660	865	775	695	665	975	840	655	675
4/1/16 6:00 PM	1250	1290	1080	990	820	755	770	945	1020	915	675	740	820	615	650	665	695	680	585	660	675	615	545
4/1/16 8:00 PM	1410	1490	1330	1300	1200	935	950	1070	1100	980	700	705	785	580	695	595	690	675	520	550	570	450	470
4/1/16 10:00 PM	1920	1790	1740	1800	1540	1270	1500	1770	1990	1720	1330	1420	1570	1290	1430	1210	1320	1210	995	1040	1050	915	885
4/2/16 8:00 AM	1460	1520	1470	1390	1330	1300	1340	1390	1390	1300	1260	1290	1290	1210	1230	1230	1220	1170	1120	1120	1100	1030	1070
4/2/16 12:00 PM	1970	1930	1870	1810	1770	1760	1720	1780	1770	1700	1650	1700	1630	1580	1560	1610	1590	1560	1500	1490	1460	1360	1470
4/2/16 4:00 PM	1780	1880	1830	1770	1710	1630	1570	1730	1710	1630	1500	1610	1560	1450	1470	1540	1480	1450	1380	1460	1390	1300	1340
4/2/16 8:00 PM	1750	1680	1610	1610	1550	1490	1470	1600	1620	1500	1380	1480	1480	1310	1350	1350	1340	1330	1230	1280	1250	1220	1180
4/3/16 8:30 AM	1640	1610	1540	1490	1450	1400	1380	1400	1380	1330	1290	1310	1300	1240	1250	1360	1250	1210	1170	1190	1180	1150	1130
4/3/16 12:30 PM	1640	1590	1560	1530	1510	1500	1480	1480	1480	1440	1410	1420	1400	1370	1370	1380	1370	1350	1330	1330	1330	1320	1300
4/3/16 5:00 PM	1570	1570	1550	1520	1500	1470	1450	1490	1470	1430	1410	1430	1390	1390	1380	1410	1400	1370	1350	1380	1370	1340	1320
4/3/16 8:00 PM	1530	1520	1480	1450	1420	1400	1380	1410	1390	1360	1330	1330	1310	1300	1290	1320	1300	1280	1250	1260	1250	1220	1200
4/4/16 10:00 AM	1390	1380	1350	1320	1300	1260	1240	1270	1250	1220	1190	1220	1210	1160	1170	1200	1170	1160	1120	1130	1130	1100	1110
4/4/16 2:30 PM	1290	1290	1260	1230	1200	1180	1160	1210	1200	1150	1120	1180	1180	1100	1120	1130	1120	1100	1170	1100	1090	1090	1060
4/5/16 3:30 PM	925	980	975	955	920	890	860	935	925	885	845	755	630	740	740	935	910	860	825	865	800	730	795
4/6/16 2:00 PM	655	725	715	690	665	635	620	725	730	680	615	670	660	610	590	715	715	690	600	580	525	488	595
4/8/16 5:00 PM	330	352	350	342	336	326	320	350	358	342	318	342	350	316	316	346	350	344	312	330	318	300	308
4/20/16 5:00 PM	25	28	26	25	25	24	25	28	27	26	24	27	27	24	26	27	27	27	26	26	25	25	24

Figure 6.8 PH<sub>3</sub> concentration data from trial 4 on April 1-20, 2016 in the Bird's silo.

Date	Location																						
	Headspace	Bottom	Line 1					Line 2				Line 3				Line 4				Line 5			
			1	2	3	4	5	1	2	3	4	1	2	3	4	1	2	3	4	1	2	3	4
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	
4/25/16 11:30 AM	2000	15	10	81	2000	2000	2000	17	20	202	2000	9	12	935	2000	17	53	1260	2000	11	83	925	2000
4/25/16 2:00 PM	1910	1940	2000	2000	2000	1890	1710	865	550	288	1740	404	468	955	1650	595	1230	1430	1610	1200	1040	815	1560
4/25/16 4:30 PM	1720	1560	1720	1670	1520	1470	1440	1400	715	805	1420	1140	540	790	1450	1190	930	970	1430	1330	945	1010	1420
4/26/16 10:30 AM	985	960	985	960	935	895	870	955	960	955	980	910	905	910	860	910	915	910	840	910	920	920	850
4/27/16 9:00 AM	690	765	785	770	750	700	665	785	810	790	710	765	795	785	705	735	750	740	635	720	740	738	740
4/27/16 12:00 PM	720	645	755	725	695	685	675	775	795	790	705	760	780	775	645	710	725	725	630	685	720	730	655
4/28/16 2:00 PM	625	434	635	620	605	590	570	615	620	625	585	600	605	600	560	590	590	580	550	590	600	600	570
4/28/16 9:00 PM	575	565	580	595	600	590	560	575	610	595	575	590	585	575	570	525	560	550	510	525	555	545	525
4/29/16 2:00 PM	535	520	540	560	550	530	520	525	550	530	500	498	525	535	510	486	505	490	476	490	505	494	472
5/1/16 6:00 PM	390	380	392	418	414	404	392	408	418	410	384	396	400	402	396	384	384	364	354	380	382	376	356
5/2/16 12:00 PM	374	382	376	372	368	360	358	370	370	358	350	358	358	352	352	350	348	370	336	350	348	346	338
5/3/16 12:00 PM	336	338	340	336	330	324	322	334	330	326	316	330	328	326	318	320	312	316	312	318	320	318	310
5/9/16 3:00 PM	114	116	120	116	112	108	107	128	133	132	113	128	134	133	106	118	120	119	105	121	125	125	109

Figure 6.9 PH<sub>3</sub> concentration data from trial 5 on April 25-May 9, 2016 in the SCAFCO silo.

Date	Location																						
	Headspace	Bottom	Line 1					Line 2				Line 3				Line 4				Line 5			
			1	2	3	4	5	1	2	3	4	1	2	3	4	1	2	3	4	1	2	3	4
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	
4/25/16 11:00 AM	425.9268	43775.81	43681.16	496.9146	0	0	0	48437.34	0	0	402.2642	56719.25	0	331.2764	0	88119.52	0	0	402.2642	41267.57	2176.959	47.3252	449.5894
4/25/16 2:00 PM	1798.358	28134.83	0	0	0	1159.467	0	47206.89	0	899.1788	0	44296.39	189.3008	1751.032	0	0	520.5772	1159.467	1798.358	0	0	0	0
4/25/16 4:30 PM	3312.764	0	35493.9	189.3008	0	757.2032	1656.382	37765.51	1467.081	2271.61	0	43065.93	875.5162	2602.886	0	48295.37	1348.768	1703.707	0	13440.36	591.565	1632.719	1112.142
4/26/16 10:30 AM	6791.166	0	0	0	686.2154	0	118.313	0	0	0	0	1514.406	0	0	615.2276	0	354.939	449.5894	0	0	1609.057	1017.492	1301.443
4/27/16 9:00 AM	5466.061	4211.943	3857.004	567.9024	2839.512	0	94.6504	0	0	544.2398	0	0	0	0	70.9878	1798.358	1845.683	1561.732	2389.923	0	2105.971	2034.984	2176.959
4/27/16 12:00 PM	5205.772	94.6504	0	0	307.6138	236.626	165.6382	0	0	520.5772	141.9756	0	0	165.6382	260.2886	0	0	0	733.5406	0	0	0	0
4/28/16 2:00 PM	4140.955	4188.28	0	0	47.3252	0	0	0	23.6626	0	0	70.9878	94.6504	0	1561.732	1703.707	1727.37	1703.707	1585.394	1632.719	0	0	0
4/28/16 9:00 PM	3786.016	3786.016	0	0	0	0	0	0	0	0	0	0	94.6504	0	141.9756	118.313	0	0	0	141.9756	212.9634	0	0
4/29/16 2:00 PM	0	0	23.6626	94.6504	70.9878	0	0	141.9756	189.3008	141.9756	0	23.6626	0	0	0	0	0	0	0	0	0	0	0
5/1/16 6:00 PM	2247.947	189.3008	0	0	0	0	141.9756	0	0	0	0	0	0	0	0	23.6626	0	0	0	0	47.3252	0	0
5/2/16 12:00 PM	2011.321	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
5/2/16 8:00 PM	0	0	1609.057	1443.419	1396.093	828.191	23.6626	1774.695	1893.008	1680.045	402.2642	1751.032	1822.02	496.9146	1656.382	1703.707	1845.683	1727.37	189.3008	1751.032	1845.683	1774.695	283.9512
5/3/2016 12:00	47.3252	0	993.8292	757.2032	638.8902	118.313	23.6626	1112.142	1135.805	757.2032	23.6626	1159.467	1230.455	0	686.2154	1064.817	1088.48	899.1788	0	946.504	1088.48	993.8292	0

Figure 6.10 SF concentration data from trial 5 on April 25-May 9, 2016 in the Bird's silo.