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PRECISION FUMIGATION OF A SILO WITH PROFUME ™

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ABSTRACT

Options for the application and distribution of sulfuryl fluoride in grain silos were investigated. A vaporiser constructed to convert liquid sulfuryl fluoride to gas enabled the application of more than 4 kg of gas to the silo within a few minutes. However, without recirculation, the time taken for the gas to equilibrate throughout the silo was several days.

The use of recirculation allowed for a controlled addition of liquid ProFume™ into a recirculating air stream at a location where liquid entering the stream would be vaporised prior to reaching the grain mass, negating the need for a vaporiser. Recirculation was achieved by pumping air through an external pipe connecting to the lid of the silo with an aeration duct in the base of the silo. Optimal rates of addition and the time to achieve an even distribution of gas in the silo were determined for addition of ProFume™ to either the headspace (with downward recirculation) or into the base (with upward recirculation).

The suggested method for application resulting from these trials requires the addition of ProFume™ at a rate sufficient to achieve the desired end concentration (or slightly higher) in the inlet pipe. Gas should be added to the largest airspace whether that is the headspace, aeration duct or plenum, and recirculation should continue until the concentration in the outlet pipe stabilises. If the concentration of ProFume™ applied is not close to the desired concentration, the time to equilibration will be affected by the degree to which the application is higher or lower than the end concentration, the volume of the headspace and the number of air exchanges per hour achieved by recirculation.

INTRODUCTION

ProFume™ gas fumigant (99.8% sulfuryl fluoride) is under development as an alternative to methyl bromide for use in flour mills, food processing facilities and grain stores. Laboratory studies have shown that control of all life stages of stored product insects can be achieved (Drinkall *et al.,* 1996; Bell and Savvidou, 1999; Dow AgroSciences, unpublished data).

Sulfuryl fluoride is applied to open structures such as flour mills and food processing facilities as a liquid under pressure, through introduction tubing. The liquid immediately converts to gas when released from the tubing, utilising the heat in the air. However, given the limited headspace and air movement within grain stores a liquid application of this nature is not optimal for preventing liquid sulfuryl fluoride from contacting the grain and achieving rapid equilibrium within the storage facility. Therefore, a different approach was investigated to apply ProFume™ to grain stores.

If ProFume™ is to be considered as an alternative to methyl bromide, the total time taken to complete the fumigation of grain stores should be similar to that required for methyl bromide fumigations. While the time taken to kill insects at a given concentration cannot be altered unless the temperature is raised, the time taken to apply the gas, achieve an even distribution and aerate can be optimised.

This paper looks at the development of a system for applying gaseous ProFume™ to a grain store and achieving an even distribution of gas within the silo in as short a time as possible.

MATERIALS AND METHODS

Silo (Grain Store)

The silo used in these trials was a 50 tonne free-standing Kotzur sealed silo constructed by Modern Engineering Constructions (www.kotzur.com/silos.html) fitted with a wall mounted pressure relief ("breather") valve to prevent buckling of the structure due to changes in atmospheric pressure. A pressure test showed the silo to be well sealed with a pressure halving time of more than 90 s. In calm weather this equated to a gas half loss time of 4.9 days and under windy conditions to a gas half loss time of 2.9 days. The dimensions of the silo are given in .

TABLE 1

. Dimensions and capacity of the silo used for these trials.

Capacity	Capacity Wheat	Maximum load	Height centre	Diameter	Cone angle
58 m^3	47 t	55 t	6.7 m	3.8 m	35°

To enable the measurement of gas concentrations at different locations in the silo, a cable was suspended from the centre top of the bin with 6 gas sampling lines attached at 1 m intervals. One sampling line was inserted into the base of the silo and a further eight sampling lines into the walls of the silo, 4 each at 2 m and 4 m from the bottom of the bin, hereafter referred to as the lower wall sample lines and upper wall sampling lines, respectively.

The silo was filled to approximately 80% capacity with wheat, leaving a headspace of approximately 12 m^3. The total gaseous space in the silo, including the interstitial spaces, was calculated to be approximately 31 m^3.

TABLE 2.
Sampling locations in the silo.

Gas sampling lines		Temperature measurement points	
Number	Location	Number	Location
1 (Core 1)	Centre, top (headspace)	t 1	Centre, 6 m (bottom cone)
2 (Core 2)	Centre, 1 m	t 2	Centre, 5 m
3 (Core 3)	Centre, 2 m	t 3	Centre, 4 m
4 (Core 4)	Centre, 3 m	t 4	Centre, 3 m
5 (Core 5)	Centre, 4 m	t 5	Centre, 2 m
6 (Core 6)	Centre, 5 m	t 6	Centre, 1 m (headspace)
7 (Core 7)	Centre, 6 m (bottom cone)	t 7	Silo inlet
8	Standard (or ambient)	t 8	Silo outlet
9	Edge, lower SE	t 9	Fumiscope
10	Edge, lower NE		
11	Edge, lower SW		
12	Edge, lower NW		
13	Edge, upper SE		
14	Edge, upper NE		
15	Edge, upper SW		
16	Edge, upper NW		

Data acquisition and control

A computer running Windows 95 was used to control the gas sampling system and record the data. It was fitted with an Advantech (www.advantech.com) PCL812 combination card and a PCL740 digital-output card. The control system was designed in and run by Advantech Genie, a windows-based graphical data acquisition and control tool. At the end of each experiment, data files were processed using Microsoft Excel.

Gas sampling

Gas sampling was undertaken with an automated, computer-controlled system. Gas sampling lines (1/8th inch i.d. nylon tubing) ran from sampling locations in the grain silo to a bank of 16 solenoid valves arranged in a manifold. A pump sucked gas samples through the tubes and directed them to a 3-way solenoid valve that either vented to atmosphere (so that samples could be rapidly drawn through the tubes), or through a flow controller to the gas sensor for analysis.

Gas analysis

Gas analysis was done using a Fumiscope (Model 4.2, Key Chemical Equip., Clearwater, FL. USA) thermal conductivity meter calibrated for sulfuryl fluoride and modified to enable automated recording of concentrations in synchrony with the sampling system. The Fumiscope, which has its own internal pump, was left on for the period of each experiment. It sucked sample gas from a T-junction that was connected to the gas sampling system and to a short (approximately 50 cm) vent tube. When gas samples were being pumped from the silo, the Fumiscope drew in sample gas; otherwise it drew fresh air through the vent tube.

As the response of the Fumiscope's thermal conductivity sensor is temperature-dependant, a precision temperature sensor (LM35, National Semiconductor; www.national.com) was attached to the sensor so that results could be corrected for temperature. To account for the drift in the zero reading of the Fumiscope, the sampling system was set to record a zero-reading prior to each gas sample. In some experiments, when rapid consecutive readings were required, there was not enough time between samples to record zero readings, so corrections were made using the zero reading taken before commencing fumigation. A gas standard (sulfuryl fluoride diluted in air), contained in a 100 L Tedlar gas-tight bag (SKC, www.tedlarbag.com) was attached to one gas sampling line.

Temperature

T-type thermocouples were placed next to the gas sample tubes at the 7 locations down the centre of the silo, as well as outside the silo and in the recirculation pipes. The thermocouple wires were led out of the bin through the gas injection cowl situated underneath the shedder plate and attached to a thermocouple analog-to-digital conversion module (ADAM 4018, advantech.com). The module was sampled by the computer in the instrument hut.

Introduction of ProFume™ into silo

The silo used for these experiments is raised off the ground and has a conical base. Modifications of the silo were made to facilitate the introduction of gas into either the top or the base of the silo. PVC pipes and reinforced flexible hose (approx 70 mm diameter) were used to connect the top (lid of the silo) and the base of the silo either directly or through a fan (when active recirculation was required). The direction of the recirculation was reversible by changing the inlet and outlet connections to the fan. To facilitate the movement of gas into and out of the base of the silo, a void was created across the cone by installing a “shedder plate” (www.smallaire.com.au). The shedder plate resembles an upturned “v” section of steel. When the silo is filled with grain a void (air space) remains underneath the shedder plate. The bottom gas inlet was inserted into the void under the shedder plate so that recirculating gas was either drawn from or passed into that void, rather than directly into the grain bulk.

Initial concerns over the possibility of a “fog out” due to lowering the temperature by rapid addition of liquid ProFume™ into the small headspace of the silo led to the use of a heat exchanger to ensure that gas and not liquid entered the silo and that the temperature of the gas entering the silo was close to ambient. In experiments that used recirculation, liquid ProFume™ was metered into the recirculation pipe, so that complete vaporisation occurred in the air stream before it entered the silo, negating the need for a heat exchanger.

Gaseous ProFume™ introduction

A heat exchanger was constructed from a 15 m length coil of copper tubing (13 mm OD) in a 200 L drum of water. As the gas’s boiling point is low (-55.2°C) the vaporiser was designed to work with water at ambient temperature, thereby avoiding the need to include heating coils or burners. The inlet to the heat exchanger was connected to a cylinder of sulfuryl fluoride with flexible pressure hose. The outlet was connected to the silo with clear plastic hose, which allowed any liquid exiting the vaporiser to be observed. Even with quite cold water (7.9°C), this vaporiser was found to be adequate; 4.4 kg of sulfuryl fluoride was vaporised within 3 min, with a drop in the water temperature of 1ºC in accordance with the calculated value, and no liquid in the outlet hose.

Liquid ProFume™ introduction

Introduction of liquid ProFume™ from the cylinder was controlled using a Whitey SS-22RS2 stainless steel metering valve. The valve was inserted in line in a 5 m length of _ inch tubing running from the ProFume™ cylinder to the recirculation pipe. Using this valve, we were able to control the flow rate of ProFume™ to as low as 2.8 kg h^{-1}. For commercial application, orifice plates or lengths of capillary tubing would be a cheaper option once a desired flow rate was determined.

The air speed of the recirculation pump was measured using a TSI Hotwire anemometer. The average flow through a pipe of radius 16 mm was 16.9 m s^{-1}, which equates to an air flow of 49 m^3 h^{-1}. The concentration in the inlet pipe (_ g m^{-3}) resulting from an addition of _ kg ProFume™/hour into this air flow, would therefore follow the relationship: _ =1000_ / 49. Thus an application rate of 2.8 kg h^{-1} would result in a concentration of 57 g m^{-3} entering the silo from the inlet pipe.

In practice, the observed concentration at the inlet was slightly lower than that predicted by the rate of addition and the air flow. This could be due to the position of the sampling line and possible plume/mixing behaviour within the inlet pipe.

RESULTS AND DISCUSSION

Gaseous introduction with natural convection

Initial trials undertaken to assess the application of ProFume™ to a silo without recirculation, relied on natural convection and diffusion to distribute the gas. In each of these trials 4 kg of ProFume™ as gas, was added to the silo through the heat exchanger under its own pressure, over several minutes.

Gas added to headspace

The concentrations observed in the core of the silo where gas was added to the headspace and not recirculated are shown in **Error! Reference source not found.**. After approximately 48 h the concentrations through the grain mass had not equilibrated and varied from approximately 125 g m^{-3} in the headspace to less than 90 g m^{-3} in the lower sections of the silo. The continual loss of concentration at sample location 6, which is in the void under the shedder plate, may indicate that there was some leakage at that point.

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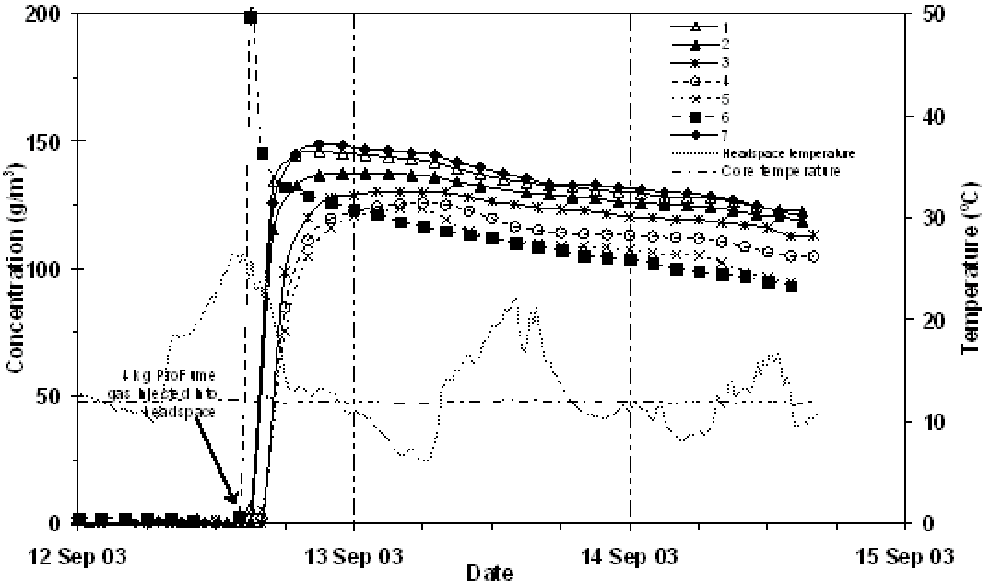

Figure 1. Concentration of sulfuryl fluoride during a trial of passive addition of ProFume™ to the headspace.

Gas added to Base

When gas was added to the base of the silo (into the shedder plate void) the distribution was very uneven (Figure 2). Thirty six hours after application the gas concentrations varied wildly from more than 1000 g m^{-3} in the base and headspace to less than 10 g m^{-3} in the centre of the grain bulk. A short period of fan-forced recirculation reduced the variation and a second period of recirculation equilibrated the gas through out the silo.

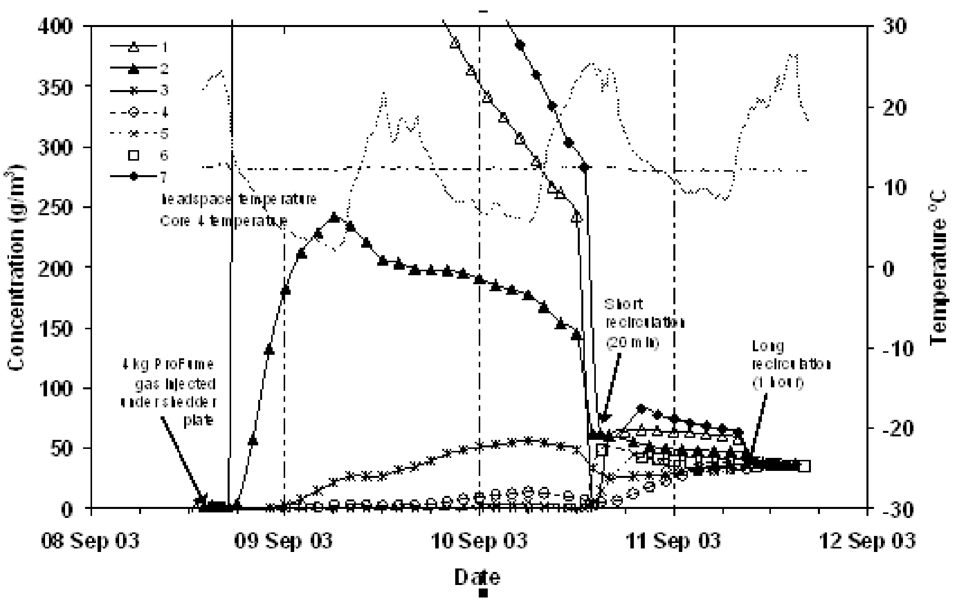

Figure 2. Concentration of sulfuryl fluoride during passive addition of ProFume™ added under the shedder plate – no recirculation for 40 h followed by a short period of recirculation (20 min), no recirculation for a further 24 h and a final 1 h of recirculation.

Comparing these two trials shows that the volume of the headspace (12 m^3) had a buffering effect on the addition of gas. Addition to the headspace resulted in better mixing and distribution of gas, whereas when gas was added into the small volume of the shedder plate void (less than 0.5 m^3) there seems to have been little mixing and distribution, although, surprisingly a large proportion of gas moved from the void under the shedder plate to the headspace with very little mixing, resulting in high headspace concentrations. This may be explained by the gas following the route of least resistance, from the shedder plate void, up the wall to the headspace. This has not been proven but seems to be a likely scenario. Given that the gas was concentrated in the headspace and in the shedder plate void, diffusion appears to have taken place predominately from the headspace into the upper sections of the grain mass and not from the base of the silo. The concentration at sampling locations 2 (2 m from the top of the silo and within the top of the grain mass) and 3 (3 m from the top of the silo and at least 2 m within the gain mass) rose well above those in the lower sections of the silo over the following day. This is likely to be a result of convection within the silo, due to diurnal heating and cooling of the headspace, which interacted with the stable temperature of the grain mass.

The effect of recirculation can be seen in rapid equilibration of concentrations that occurred when the recirculation fan was turned on firstly for 20 min and later for 1 h (Figure 2).

Active addition of liquid ProFume™

In most trials undertaken with recirculation, 2.4 kg of ProFume™ was added to the recirculating air flow as a liquid. This application equated to a final concentration in the silo of approximately 80 g m^{-3}.

Prior to undertaking trials where ProFume™ was actively added to an air stream, the effect of adding liquid ProFume™ into a recirculating air flow was assessed. Liquid ProFume™ was released at a rate of 6.4 kg h^{-1} (inlet concentration of 130 g m^{-3}) into the recirculating air flow and the temperature in the recirculation pipe 15 cm downstream was recorded . The fan warmed the air passing through it (0 – 4 min) and then addition of liquid ProFume™ cooled the air (4 – 28 min), but when combined, the temperature remained above ambient, which was approximately 20ºC. The full warming effect of the fan on the recirculating air was observed when the addition of ProFume™ ceased; the temperature in the inlet pipe rose to almost 40ºC prior to the pump being turned off.

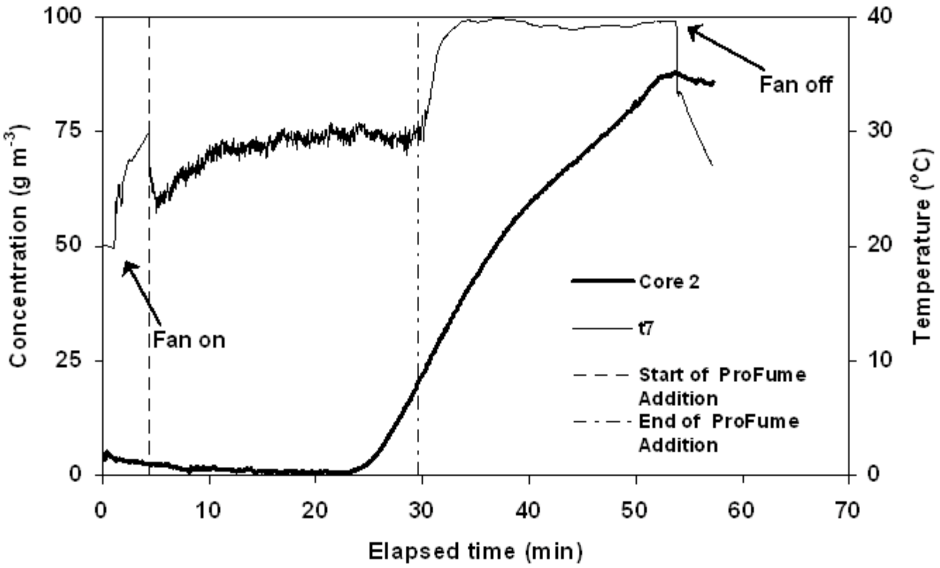

Figure 3. ProFume™ concentration at Location 2 (in the grain mass at least 1 m below the grain peak) and temperature profile in the inlet pipe during liquid application.

Active addition of ProFume™ into the headspace

When ProFume™ was added to the headspace, the pump was used to recirculate the air within the silo in a downward direction from the headspace through the grain, withdrawing it from under the shedder plate. ProFume™ was added to the recirculating flow, after the pump, as the recirculating air was being pushed up the external pipe to re-enter the headspace.

a single sampling location (Core 2) was continuously monitored during the application and equilibration of gas, at the rate of 6.2 kg h^{-1}. Core 2 was chosen because it had been found (data not shown) to be the last sampling location in the silo to detect gas during downward recirculation. Gas was not detected at this location until 22 min after the commencement of ProFume™ addition. ProFume™ addition took 26 min but a further 24 min elapsed (50 min in total) before the gas level at Core 2 was stable. Following this introduction, the concentration was measured at all 15 locations throughout the silo and was found to be relatively even

TABLE 3

Concentrations observed at all locations throughout the silo after cessation of recirculation

Location (Core)	Concentration ($g\ m^{-3}$)	Location (Walls)	Concentration ($g\ m^{-3}$)
1(Head space)	83.4	9	73.4

2	81.8	10	73.5
3	74.7	11	74.4
4	72.0	12	74.0
5	75.0	13	72.3
6	74.7	14	72.0
7 (Bottom cone)	80.1	15	72.4
8 (Standard conc.)	75.0	16	72.4

In further experiments, the rate of application was altered to assess whether there was an optimal concentration at which to apply the gas. Rates of application varied from 3.9 to 20 kg h^{-1}, which equates to inlet concentrations of between 80 and 400 g m^{-3}. Two examples of different rates of application are given in **Error! Reference source not found.** (4.2 kg h^{-1}) and **Error! Reference source not found.** (20 kg h^{-1}). A summary of the time taken to achieve an even distribution of gas throughout the silo with different rates of application is given in .

TABLE 4.
Time taken for the equilibration of gas concentrations in the silo, following application of ProFume™ at varying rates.

Application rate (kg h^{-1})	Inlet concentration (g m^{-3})	Time to equilibration (h)
3.9	80	1.1
4.2	86	1.1
6.2	126	0.9
6.2	126	0.9
6.4	131	0.9
11	224	0.9
20	408	0.8

When the gas was added to the air stream to produce a concentration which was to be the ultimate concentration in the silo (4 kg h^{-1}), a secondary front began before gas addition was complete. This illustrates that the recirculation of gas in the silo is not uniform and that sections of the silo are recirculated at a greater rate than others. Therefore, adding gas at a rate slightly higher than the end concentration would be required if the aim was to add the required amount of ProFume™ while passing a single concentration front through most of the bulk..

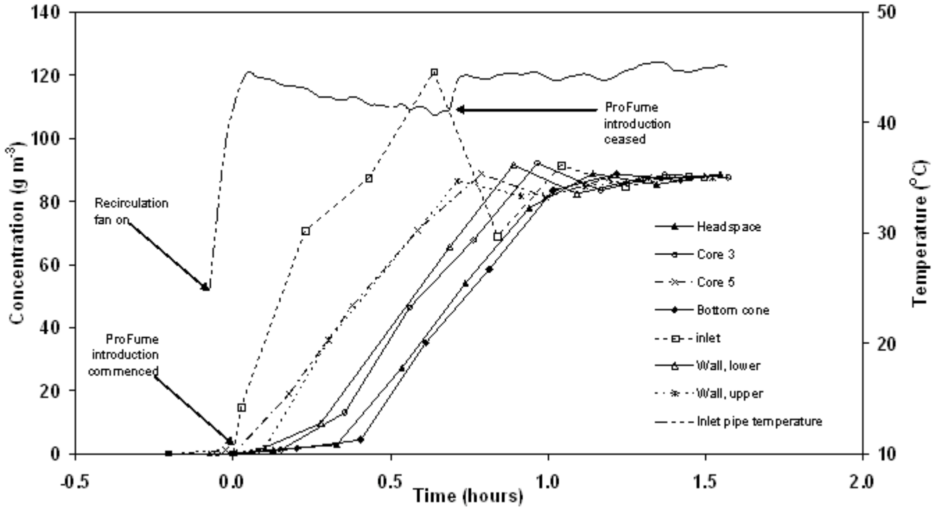

Figure 4. Concentration of sulfuryl fluoride during trial of active addition of ProFume™ to the headspace at the rate of 4.2 kg h^{-1}.

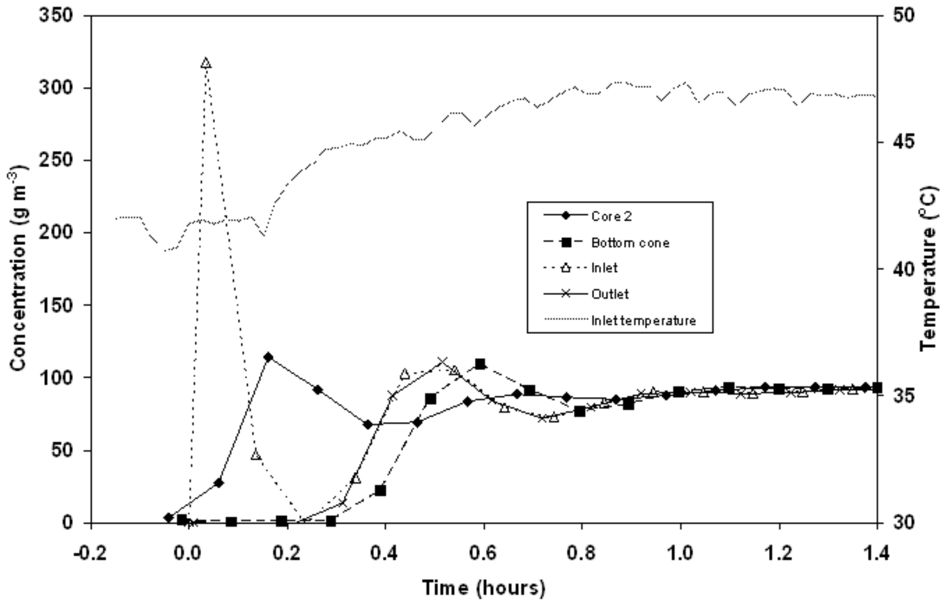

Figure 5. Concentration of sulfuryl fluoride during trial of active addition of ProFume™ to the headspace at the rate of 20 kg h^{-1}

Active addition of ProFume™ into a smaller headspace

In previous experiments, the headspace was approximately 12 m^3 and provided a buffer for the mixing and distribution of gas during recirculation. To test the effect of headspace volume, the silo was filled to near capacity, resulting in a headspace which was substantially smaller (probably less than 3 m^3). Gas was applied by downward recirculation to achieve a final concentration of approximately 15 g m^{-3} at the rate of 2.8 kg h^{-1} (equating to an inlet concentration of approximately 57 g m^{-3}). The resultant concentration profiles showed that the smaller headspace resulted in marked pulsing of a high concentration of gas through the silo. The time for equilibration was longer than in previous trials, taking a little over 3 h, compared with 1 h in previous trials. This result is very similar to that observed by Horn *et al.,* (2003) during a phosphine fumigation of a silo filled with wheat. When they compared that fumigation with a silo that was half full, they noted that in the larger headspace, there was greater dilution during each recirculation and therefore faster the equilibration of gas distribution.

Active addition of ProFume™ into the base of the silo

Addition to the base of the silo was tested using the pump to recirculate the air within the silo in an upward direction. Air was drawn from the headspace through the external pipe and pushed into the small void under the shedder plate. Liquid ProFume™ was added to the external pipe 2.5 m upstream of the pump. Under upward recirculation Core 5 was identified (data not shown) as the last sample location where gas was detected.

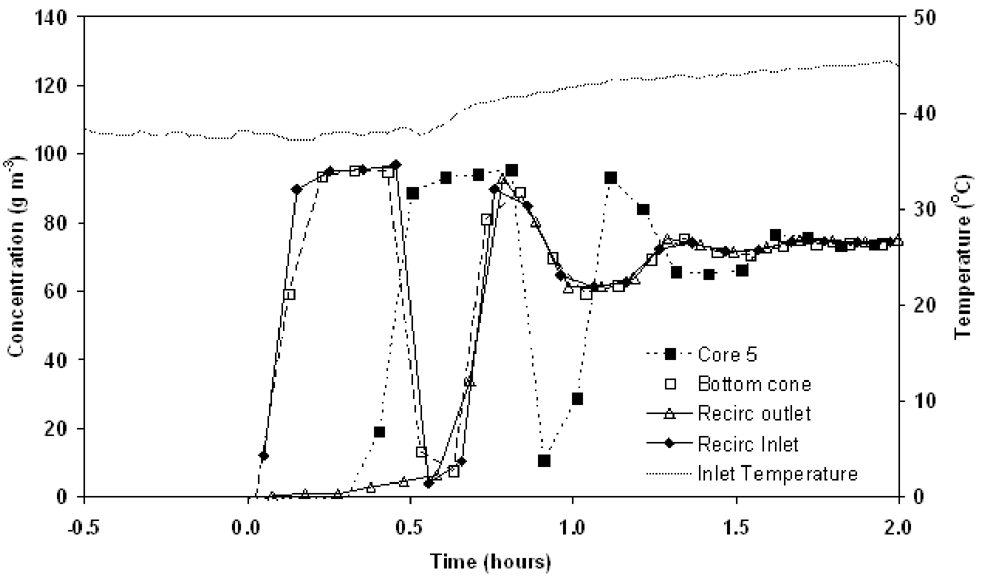

Figure 6 Active addition of ProFume™ into a reduced headspace at the rate of 2.9 kg h^{-1}.

Three rates of application were tested, the lowest rate being 3.0 kg h^{-1} This was a slow application that pushed a single front of approximately 50 g m^{-3} through the silo in approximately 40 min and then "piggybacked" a second front on the first so that the concentration in the inlet stepped up to approximately 100 g m^{-3}. The addition of ProFume™ was complete before the second front passed through the entire silo. The resulting uneven distribution of gas required almost an hour of recirculation to equilibrate.

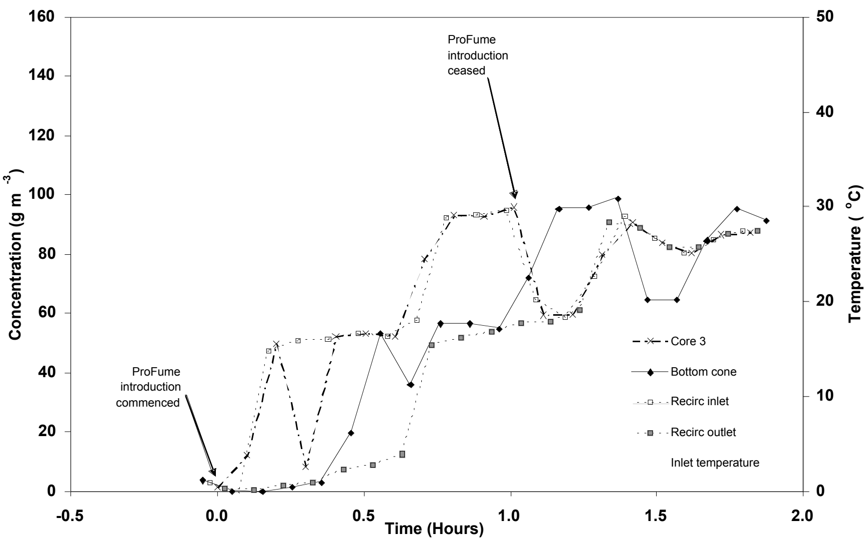

Figure 7. ProFume™ concentrations observed during active addition to the base at the rate of 3.0 kg h^{-1}.

The addition of ProFume™ at the rate of 4.3 kg h^{-1} approximated the addition of gas at the desired final concentration. In this instance only a single concentration front passed through the silo and a fairly even distribution, well within the limits set out by Banks and Annis (1984), was achieved within 75 min.

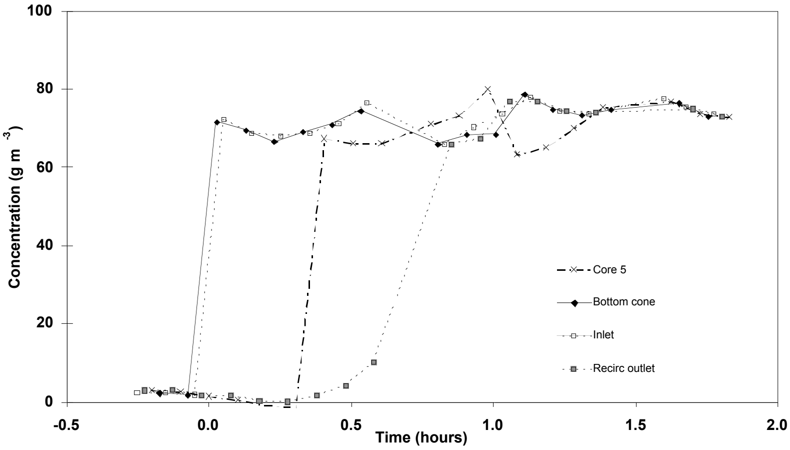

Figure 8. ProFume™ concentrations observed during active addition to the base at the rate of 4.3 kg h^{-1}

Increasing the application rate further, to 6.7 kg h^{-1} (inlet concentration of 136 g m^{-3}) created a front of high concentration (Figure 9) which was passed through only about a third of the silo before the input of ProFume™ was completed. In this instance, addition and equilibration of the gas throughout the silo took 1.5 h.

CONCLUSION

Given that a fumigation is not considered to have commenced until such time as there is an even distribution of gas, or at least until all parts of the fumigation enclosure are above a threshold concentration, the time taken for gas to distribute through a grain mass by diffusion and natural convection can increase the time of a fumigation process and can result in significant costs in time.

Of several options investigated for the application and distribution of sulfuryl fluoride in grain silos, the controlled addition of ProFume™ into a recirculating air stream would appear to be the most time-efficient. The optimal application relies on the rate of addition being matched to the recirculating air flow such that the resulting inlet concentration slightly exceeds the final expected concentration. Given that scenario, the concentration at all locations within the bin would be expected to climb steadily until equilibrium is reached However, if the rate of application is either too high or too low, a “plug” of higher concentration will recirculate through the silo and will equilibrate at a rate dependant on the free space in the silo (faster with a larger headspace) and the speed of recirculation.

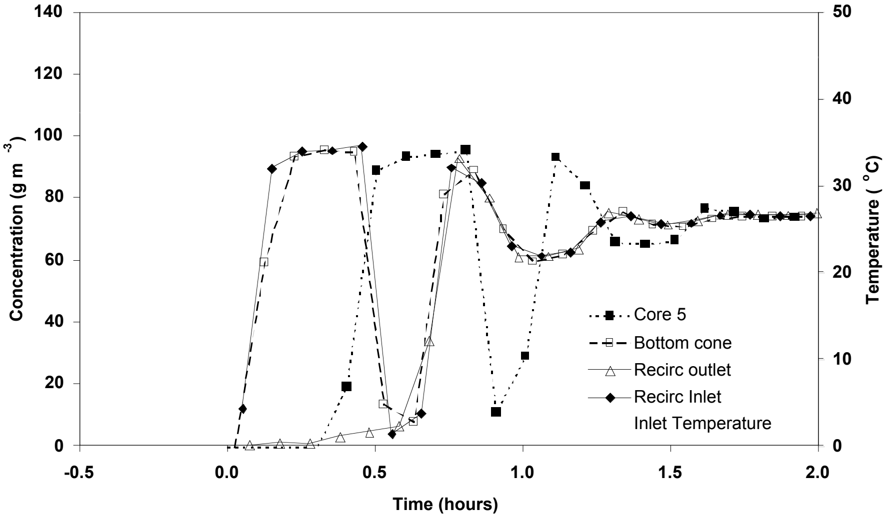

Figure 9. ProFume™ concentrations observed during active addition to the base at the rate of 6.7 kg h^{-1}.

Although matching the rate of addition with the time to pass one concentration front through the silo would be ideal, each fumigation involves a unique recirculation profile. Parameters such as silo geometry, recirculation fan, fill rate, commodity type, and size and location of leaks are some of the factors that impinge on each fumigation. As a result the near-ideal behaviour observed in and is unlikely to occur. A more likely result is that a plug of high concentration will form and will move around the silo, diluting and diffusing during recirculation until equilibrium is reached. Assuming that an uneven application is likely, steps that may minimise the time taken to reach equilibrium include; adding the gas to the larger airspace, whether that is the headspace, aeration duct or plenum (if the silo is fitted with one), leaving a small buffer in the system by not filling the silo beyond a given percentage (e.g. 90%) and increasing the recirculation rate by increasing the capacity of the fan.

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