

## NOVEL FUMIGANTS AND HEAT TREATMENTS FOR FLOUR MILLS

Paul G. Fields

Cereal Research Centre, Agriculture and Agri-Food Canada, 195 Dafoe Rd, R3T 2M9,  
Winnipeg, Manitoba, Canada

\* Corresponding author's e-mail: [paul.fields@agr.gc.ca](mailto:paul.fields@agr.gc.ca)

### ABSTRACT

The efficacy of methyl bromide fumigations in mills (6 mills) was compared to heat treatments (3 mills), sulfuryl fluoride (ProFume®) (4 mills) and phosphine (ECO<sub>2</sub>FUME®), heat and carbon dioxide combination treatment (2 mills). The efficacy of treatments was estimated in three ways: *Tribolium castaneum* adults and eggs in vials during treatment, pheromone traps and rebolt sifter tailings counts.

Almost all treatments were effective in killing 100% of adult *T. castaneum* put out in vials. There was more survival of eggs. In the sulfuryl fluoride treatments, egg mortality ranged from 35 to 99.6%. The other treatments had egg mortalities over 98%.

Insect populations in the mills were estimated using pheromone traps. In methyl bromide treatments, the range of times that it took insect populations to rebound to pre-treatment levels was between 3 weeks or over 30 weeks. For sulfuryl fluoride the rebound took from as little as 1 week to never rebounding within the 18-week study. Phosphine combination treatment saw populations rebound within seven to 29 weeks. In all three heat treatments, none of the populations returned to the original levels by 19 weeks post-treatment.

Adult and larva flour beetles were monitoring in the tailings from rebolt sifters. For the most of the mills, there is a good correlation between insects found in the pheromone traps and insects found in the rebolt sifter tailings. However, on several occasions pheromone traps were not a good predictor of insect numbers in the rebolt sifter tailings. In methyl bromide treatments, the rebound of insect populations to pre-treatment levels occurred between 13 and over 31 weeks. For sulfuryl fluoride, the rebound occurred between 9 and over 18 weeks. The phosphine combination treatment saw populations rebound within 1 to 33 weeks. In all three heat treatments, the mills either did not sample rebolt sifter tailings or no insects were found in the tailings.

**Key words:** Sulfuryl fluoride, phosphine, carbon dioxide, heat, red flour beetle, *Tribolium castaneum*

### INTRODUCTION

Methyl bromide (MB) is a very effective broad spectrum fumigant. It is used around the world to control a wide variety of pests (pathogens, nematodes, weeds and insects) in diverse substrates (soil, food, museum artefacts, buildings, equipment, aircraft). It has been used in flour mills since the 1930's, and it has become the major tool to control insects in food processing facilities, such as flour mills, pasta production plants and breakfast cereal plants.

In 1992, methyl bromide was recognized as a significant ozone depletor and its

consumption was frozen at 1991 levels starting in 1995 (Banks, 2002; Fields and White, 2002). In 1997 Parties of the Montreal Protocol on Substances that Deplete the Ozone Layer agreed to phase out methyl bromide starting in 2005 with interim reductions along the way. These dates are ten years later for developing countries. Given that methyl bromide is such a widely used fumigant, in 1997 Parties also agreed to allow critical and emergency use exemptions for very specific uses of methyl bromide. Users must demonstrate that there are no technically and economic alternatives to methyl bromide, and they are actively trying to find alternatives to methyl bromide. This Critical Use Exemption (CUE) has been granted for flour mills in Canada, USA, Europe and Australia. This project examined the efficacy of IPM (Integrated Pest Management), heat, sulfuryl fluoride, phosphine combined with heat and carbon dioxide, and compares them with methyl bromide fumigations.

Heat treatments have been used as early as the 16<sup>th</sup> century to control stored-product insects (Fields, 1992). Heating flour mills and food processing facilities to control insects in the USA and Canada has been used since 1910 and continues to this day (Fields and White, 2002). Steam, propane and electric energy sources have been used to power the heaters. In general, the building is heated to at least 50°C for 24 hours. Extra fans are used to distribute the heat within the structure. This project worked with two heat providers, Temp-Air and Armstrong-Hunt. Temp-Air uses the same heaters that are designed for temporary heating of construction sites and sporting events and modified for heat treatments. They have been doing heat treatments to control insect pests since 1999 (Johnson and Danley, 2003). Currently they heat treat approximately twenty-five locations a year, with most of these locations receiving two heat treatments a year. The size of the facilities range from 17,000 to 1,300,000 m<sup>3</sup> and include flour mills, food processing facilities and malting plants. Heaters use either propane, as is the case with these trials, or natural gas. Armstrong-Hunt has been manufacturing heavy-duty steam heaters for insect pest management since 1990. Currently they have provided portable equipment and permanently installations in over twenty-five locations. The plant sizes range from 300 to 113,000 m<sup>3</sup>, including flour and corn mills, pasta plants, food processing plants and pharmaceutical plants.

Sulfuryl fluoride (SF or SO<sub>2</sub>F<sub>2</sub>) has been proposed as a replacement for methyl bromide in the fumigation of flour mills and other structures (Bell et al., 1996; Banks, 2002,). Sulfuryl fluoride was originally registered for termite control in 1961, under the trade name Vikane®. Since 1995, Dow AgroSciences has been expanding the use pattern of sulfuryl fluoride for use in flour mills, under the trade name ProFume® (Schneider and Hartsell, 1999). Currently it is registered in USA, France, Switzerland, Germany, Italy, Belgium, United Kingdom, Mexico and Australia.

Phosphine has been used extensively as a fumigant in bulk grain. However, it is rarely used in empty flour mills, because it can cause corrosion of copper and other metals and requires more time than methyl bromide to control insects. Using phosphine in combination with carbon dioxide and heat mitigate these problems (Mueller, 1993). This phosphine combination treatment has been used successful in the USA to control insects in food processing facilities in 24 h without corrosion. It requires extra sealing. For example, some electrical boxes are sealed, a pressurized hose attached and a small amount of air bled into the line to prevent phosphine from entering into equipment where corrosion is a concern. ECO<sub>2</sub>FUME®, manufactured by Cytex Canada Inc. is an effective way to deliver the phosphine for this combination treatment for two reasons, good control of the phosphine concentration and rapid release of gas. ECO<sub>2</sub>FUME is 2% phosphine in 98% carbon dioxide held under pressure in gas cylinders.

## MATERIALS AND METHODS

### Treatments

There were two trials with propane-fired heaters (Temp-Air) and one trial with portable steam heaters (Armstrong International Inc.), four trials with sulfuryl fluoride (ProFume®), two trials with phosphine (ECO<sub>2</sub>FUME®), heat and carbon dioxide combination treatment and six trials with methyl bromide (Table 1). For full details of the treatments see Harrison (2007).

### Dome traps

Dome traps (Trece Inc.) that are specific for trapping flour mill insects were placed on the roll stand floor and the sifter floor, five to ten traps/floor and the insects removed and counted each week. The traps were in the mill 3-9 weeks before the control treatments and for at least 18 weeks post-treatment. The traps were baited with a pheromone for the confused and red flour beetles (*Tribolium confusum* Jacquelin du Val and *Tribolium castaneum* (Herbst)) and a vegetable oil attractant. The vast majority of insects caught in the traps were flour beetles, and those data are reported here. There were two measures of efficacy. The first was time taken to return to 100% pre-treatment level. The pre-treatment populations were estimated by the number of insects/trap/day (mean of weekly captures before treatment). The second was time taken until the insects were detected three consecutive weeks in both roll stand and sifter floors or in tailings. Mill 4 and Mill 6 were followed for more than one treatment, the initial pre-treatment population estimates were used for all treatments. There was no estimate of insect populations in the immediate area outside the mills, as was done in other studies (Campbell et al., 2002).

### Rebolt sifter tailings

In Mills 1 and 3, no insects were seen in the rebolt sifter tailings. In Mill 4, 1 kg samples were taken 5 d/week and the number of live and dead adults was counted. In Mill 5, all insects were counted for rebolt sifter tailings three times each 24 h. In Mill 6, 2-kg samples were taken from the rebolt sifter tailings barrel in the mill and in the packing plant. An estimate of the total flour in the tailings barrel is made to calculate the total number of insects in the barrel. Both of these rebolt sifters pull flour from the same bins. In Mill 8, tailings samples were taken immediately after every load and inspected. Samples for the entire week were summed. Insect counts were divided by the number of days of sampling and the daily average calculated for the pre-treatment period, and all numbers divided by this to express the insect count data as a percentage of pre-treatment levels.

### Bioassays

*Tribolium castaneum* (Steinbach strain), was used as a test insect. They were reared on white wheat flour with 5% brewer's yeast at 30°C, 60% r.h. Twenty unaged adults of unknown sex were placed in 16 g of culture medium in plastic vials, 4-8 d before the treatment, and held at 20-30°C, 60% r.h. So at the time of the treatment there were 20 adults per vial and an unknown number of immatures, most would be eggs. Eight vials were used as controls. They were treated as the insects exposed to the treatment, but they were not held in the mill during the treatment. Twenty-five vials were placed thorough-out the mill a few hours before the treatment and retrieved a few hours after the end of the treatment. About half of the vials were placed in the middle of the mill and half of them near windows or doors. In some of the treatments, a group of vials were placed at one location, and pulled at regular intervals during the treatment. Dataloggers (Hobo dataloggers, Onset Computers Inc.) were placed with each vial, and the temperature recorded every 15 minutes.

Table 1. Summary of treatments to control insects in Canadian flour mills, heat treatments with external propane fired heaters, heat treatments with steam-powered portable heaters, sulfuryl fluoride (SF), phosphine combined with carbon dioxide and heat, methyl bromide (MB).

| Treatment             | Mill | Plant     | Start Date<br>(month/year) | Mill<br>Preparation<br>Time (h) | Duration of<br>Treatment<br>(h) | Duration of<br>Post-treatment<br>(h) | Plant Shut Down<br>Time (h) |
|-----------------------|------|-----------|----------------------------|---------------------------------|---------------------------------|--------------------------------------|-----------------------------|
| Heat, propane         | 1    | mill      | 08/06                      | 25.5                            | 29.3                            | 2.25                                 | 64                          |
| Heat, steam           | 3    | mill      | 06/06                      | 5                               | 24                              | 2                                    | 33.5                        |
| Heat, propane         | 7    | mix plant | 09/06                      | 19                              | 28.3                            | 12.3                                 | 79.5                        |
| SF                    | 4A   | old mill  | 12/04                      | 36                              | 27                              | 6                                    | 96                          |
| SF                    | 4B   | new mill  | 12/04                      | 34                              | 29                              | 6                                    | 96                          |
| SF                    | 4A   | old mill  | 10/05                      | 15                              | 29.5                            | 28.5                                 | 96                          |
| SF                    | 4B   | new mill  | 10/05                      | 13.5                            | 13.5                            | 46                                   | 96                          |
| SF                    | 5    | mill      | 08/06                      | 24.5                            | 22                              | 12.5                                 | 67                          |
| SF                    | 8    | mill      | 06/06                      | 30.8                            | 24.3                            | 6                                    | 104                         |
| PH <sub>3</sub> combo | 6B   | packing   | 11/05                      | 6.5                             | 26.5                            | 12                                   | 53.75                       |
| PH <sub>3</sub> combo | 6A   | mill      | 07/06                      | 51                              | 26.5                            | 4                                    | 92                          |
| MB                    | 4A   | old mill  | 11/03                      | 33                              | 33.5                            | 9.5                                  | 94.5                        |
| MB                    | 4B   | new mill  | 11/03                      | 33                              | 33.5                            | 9.5                                  | 94.5                        |
| MB                    | 4A   | old mill  | 06/04                      | 10                              | 32                              | 7                                    | 72                          |
| MB                    | 4B   | new mill  | 06/04                      | 10                              | 32                              | 7                                    | 72                          |
| MB                    | 4A   | old mill  | 05/05                      | 10                              | 32                              | 7                                    | 72                          |
| MB                    | 4B   | new mill  | 05/05                      | 10                              | 32                              | 7                                    | 72                          |
| MB                    | 6A   | mill      | 11/05                      | 40.8                            | 28.3                            | 12                                   | 101                         |
| MB                    | 6B   | packing   | 07/06                      | 23.5                            | 25                              | 5                                    | 60                          |
| MB                    | 6A   | mill      | 11/06                      | 37                              | 24                              | 3.5                                  | 74                          |
| MB                    | 6B   | packing   | 11/06                      | 21                              | 24                              | 3.5                                  | 56                          |
| MB                    | 7    | mill      | 06/06                      | 6                               | 25                              | 11                                   | 46                          |

## RESULTS

A summary of the conditions for the treatments is given in Table 1, and a summary of the efficacy is given in Table 2.

### **Insect bioassays**

There were three treatments with heat, two (Mills 1 and 7) that used external propane-fired heaters (Temp-Air), and one (Mill 3) that use internal portable-steam-powered heaters (Armstrong International Inc.). For Mill 1 (propane heaters), the average time above 50°C was 24.4 h and average maximum temperature was 65°C. For Mill 7 (propane heaters), the average time above 50°C was 26.4 h and average maximum temperature was 57.8°C. For Mill 3 (steam heaters) the average time above 50°C was 24.9 h and average maximum temperature was 56.5°C.

All insects, both adults and eggs, were dead in the two propane-fired heat treatments in Mill 1 and 7. In Mill 1, in the timed sequence, all insects were dead by 10 h after the heating began and the final temperatures had reached 57°C. In Mill 7, in the timed sequence, all insects were dead by 5.5 h after the heating began and the temperature had reached 53°C.

In Mill 3, the steam heat treatment, most bioassay insects were all dead except at four of the twenty-five locations. In the timed sequence, all insects were dead by 21 h after the heating began and the temperatures had reached 53-57°C.

The only damage caused by the heat treatments was in Mill 1, one sprinkler head was activated because during the start-up one of the fabric ducting was not properly anchored and hot air was directed at sprinkler head. The duct was rerouted and anchored to overcome the problem. Mill 3 and 7 exhibited no damage to the building, processing equipment or electronics.

The Canadian sulfuryl fluoride label did not allow for the treatment of food products. So the mills were emptied of all food products such as; wheat, feed, milled products and additives. In Mills 5 and 8, silos within the mill containing finished flour were sealed and not exposed to sulfuryl fluoride. Sulfuryl fluoride has a range of times and doses that is adjusted in relation to the life stage and species targeted. *Tribolium castaneum*, the insect used in the bioassay and a common pest of flour mills in Canada, has a relatively high tolerance to sulfuryl fluoride. Eggs are the most tolerant life stage. In general, there are two doses, a high dose that will kill all life stages, and a lower dose that will kill all post-embryonic life stages and some but not all of the eggs. The sulfuryl fluoride doses used in these trials, range from the dose to kill all post-embryonic life stages (target ct Product (CTP) = 428 gh/m<sup>3</sup>; Mill 4, December 2004), to the dose to kill all life stages (target CTP = 645-965 gh/m<sup>3</sup>; Mill 4, October 2005; Mill 5, August 2006; Mill 8, June 2006, target CTP was adjusted for temperature).

For all sulfuryl fluoride fumigations, there was 100% mortality of the bioassay adults. In the first fumigation with SF in Mill 4 in December 2004, the average mortality of bioassay immatures (mostly eggs) was 35% in Mill 4A and 63% in Mill 4B, with a CTP of 457-490 gh/m<sup>3</sup>. This was expected, as the dose chosen was the low dose targeting post-embryonic stages. The other three later fumigations used the high dose with CTs from 832 to 1280 gh/m<sup>3</sup> and gave average control of bioassay immatures from 94.5 to 99.9%.

The average temperature in Mill 4 during the December 2004 fumigation was 19°C in Mill 4A and 23°C in Mill 4B, which is cooler than fumigations done with sulfuryl fluoride in the USA. As with most fumigants, higher temperature improves efficacy. There was a strong correlation between temperature and mortality (Fig. 1).

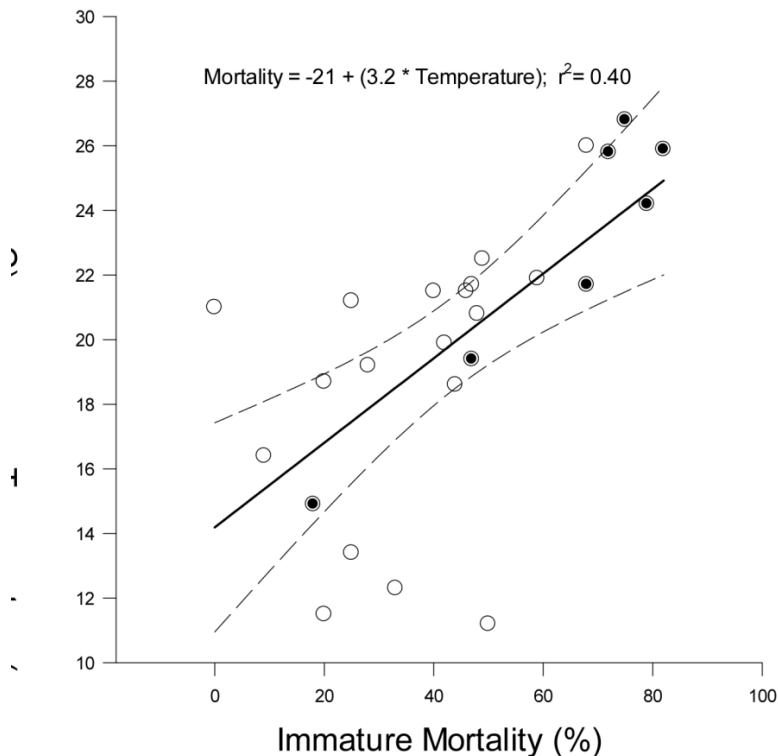


Fig. 1- The mortality of *T. castaneum* immatures (mostly eggs) during sulfuryl fluoride fumigations with CTP of 457-490 gh/m<sup>3</sup> in Mill 4A (○) and Mill 4B (●) on December 2004.

During the second sulfuryl fumigation an effort was made to raise the mill temperature by turning up the heat for the comfort heaters and using propane-fired heaters before the fumigation began. This increased the average temperature to 26°C. The other mills that used sulfuryl fluoride did the fumigations during the summer, did not use heaters, and had average temperatures of 24.3°C (Mill 5) and 29.5°C (Mill 8).

Some of the insect bioassays were removed from the fumigation after different durations. In Mill 5 for *T. castaneum*, all adults were dead after 2 h (118 gh/m<sup>3</sup>), all immatures were dead after 16 h (1150 gh/m<sup>3</sup>). In Mill 8 for *T. castaneum*, all adults were dead after 4 h (259 gh/m<sup>3</sup>), all immatures were dead after 12 h (803 gh/m<sup>3</sup>). In Mill 8 for *T. confusum*, all adults were dead after 4 h (259 gh/m<sup>3</sup>), all immatures were dead after 8 h (529 gh/m<sup>3</sup>).

The treatment with phosphine, heat and carbon dioxide was unique in the trials in that a methyl bromide treatment was done on the same date as the alternative treatment, but in a different part of the facility. The first fumigation was done in November 2005, the packing plant was fumigated with phosphine and the mill fumigated with methyl bromide. For the second fumigation, the packing plant was fumigated with methyl bromide and the mill with phosphine, heat and carbon dioxide.

All adults in the bioassays were killed with the phosphine combination treatment on both of the dates. The average immature mortality in the phosphine combination treatment was 98.6% for the 2005 fumigation and 99.5% for the 2006 fumigation.

The phosphine did cause corrosion on the copper strips placed in the packing plant. The copper plates were blackened (Harrison, 2007), whereas the controls and the methyl bromide fumigated strips did not. Some equipment that was sealed was exposed to phosphine gas. Each piece of equipment that was sealed had a line for delivering compressed air and a second line for gas sampling. In the 2005 fumigation, nine sealed boxes had phosphine from 25 to 115 ppm, due to a malfunction in the delivery of the compressed air. However, none of these pieces of equipment had problems after the fumigation. After the 2005 fumigation, one light at the top floor of the mix plant needed a ballast replaced. This may have been unrelated to the fumigation. After the 2006 fumigation there was no failure of the equipment at start-up.

There were several methyl bromide fumigations that were part of this project; in Mills 4, 6 and 7. All adults in the bioassay were killed in all the fumigations. For the immatures in the bioassay, occasionally there was a small amount of survival.

### **Rebound of insect populations**

It is difficult to estimate the populations of insects in flour mills. Flour beetles are small, cryptic and the populations are not uniformly distributed throughout the mill. Also, insect populations change rapidly, at 30°C, 70% r.h., *T. castaneum* populations increase 70-fold per month.

For the three mills that used heat treatments, none of the insects returned to the pre-treatment levels with the 20 weeks of pheromone sampling. Mills 1 and 3 had very low levels throughout the post-treatment sampling. Mill 7, which used propane-fired heaters, consistently had insects in the pheromone traps (insects found on both floors for three consecutive weeks) 2 weeks after heat treatment. Mills 1 and 3 never have had significant numbers of insects in rebolt sifter tailings before or after treatments. Mill 7 does not regularly check for insects in rebolt sifter tailings.

For the three mills that used sulfuryl fluoride there was a wide variation in rebound of insects after fumigation. The low concentration (457 gh/m<sup>3</sup>), combined with the cool temperatures during the fumigation (18.9°C) were probably contributing factors to the rebound in Mill 4A. Insects were regularly found in pheromone traps after 16 weeks, the rebound to pre-treatment levels (over 100%) occurred after 13 to 18 weeks. Insects were consistently found in the tailings after 21 weeks, and at very high levels (Table 2).

A second fumigation with sulfuryl fluoride at the high concentration (1096 gh/m<sup>3</sup>) and at higher temperatures (26.5°C), still saw insects regularly being caught in pheromone traps 11 weeks after the fumigation, although the time to rebound to pre-treatment levels occurred after 17 to 25 weeks. The major difference with the second fumigation in Mill 4A was that the insects in the rebolt sifter tailings remained low until 26 weeks after the fumigation.

Mill 8 used similar concentrations (836 gh/m<sup>3</sup>) as the 2005 fumigation in Mill 4A. However, populations quickly rebounded. Insects were regularly found in pheromone traps 5 weeks after fumigation, populations returned to pre-treatment levels after 3 to 8 weeks. Insects were regularly found in the rebolt sifter tailings 3 weeks after fumigation and returned to pre-treatment levels after 12 weeks. The insects in the roll stand floor were not consistently found before the fumigation, so the sifter floor and basement probably give a better indication of the population trends before and after the fumigation. Some flour remained in the mill silos. It was sealed off from the rest of the mill and was not exposed to sulfuryl fluoride.

Table 2. Summary of efficacy of treatments to control insects in Canadian flour mills, heat treatments with external propane-fired heaters, heat treatments with steam-powered portable heaters, sulfuryl fluoride (SF), phosphine combined with carbon dioxide and heat, methyl bromide (MB).

| Method                | Mill | Start Date (month/year) | Temp. Inside (°C) | Temp. Outside (°C) | Gas  | Half Loss Time (h) | Gas CT (gh/m <sup>3</sup> ) | Bioassay Adult Mortality (%) | Bioassay Immature Mortality (%) | Trap catches       |                | Tailings           |                |
|-----------------------|------|-------------------------|-------------------|--------------------|------|--------------------|-----------------------------|------------------------------|---------------------------------|--------------------|----------------|--------------------|----------------|
|                       |      |                         |                   |                    |      |                    |                             |                              |                                 | Rebound Time (wks) | To 100% Levels | Rebound Time (wks) | To 100% Levels |
| Heat, propane         | 1    | 08/06                   | 56.5              | 27.5               | -    | -                  | 100                         | 100                          | 20+                             | 20+                | 20+            | -                  | -              |
| Heat, steam           | 3    | 06/06                   | 49.7              | 23.7               | -    | -                  | 94                          | 99.6                         | 20+                             | 20+                | 20+            | -                  | -              |
| Heat, propane         | 7    | 09/06                   | 51.1              | 18.8               | -    | -                  | 100                         | 100                          | 2                               | 19+                | 19+            | -                  | -              |
| SF                    | 4A   | 12/04                   | 19.8              | 2.6                | 457  | 6.3                | 100                         | 35                           | 16                              | 13                 | 18             | 21                 | 9              |
| SF                    | 4B   | 12/04                   | 24.2              | 2.9                | 490  | 2.9                | 100                         | 63                           | -                               | -                  | -              | -                  | -              |
| SF                    | 4A   | 10/05                   | 27.6              | 7.0                | 1096 | 5.8                | 100                         | 96                           | 11                              | 17                 | 25             | 26                 | 26             |
| SF                    | 4B   | 10/05                   | 27.0              | 4.6                | 1180 | 5.3                | 100                         | 92                           | -                               | -                  | -              | -                  | -              |
| SF                    | 5    | 08/06                   | 29.5              | 20.8               | 1280 | 6.0                | 100                         | 99.7                         | 18+                             | 18+                | 1              | 23+                | 18+            |
| SF                    | 8    | 06/06                   | 31.7              | 24.3               | 832  | 4.7                | 100                         | 99.6                         | 5                               | 3                  | 8              | 3                  | 12             |
| PH <sub>3</sub> combo | 6B   | 11/05                   | 33.2              | 3.7                | -    | -                  | 100                         | 98.6                         | 29                              | 29                 | 29             | 23                 | 33             |
| PH <sub>3</sub> combo | 6A   | 07/06                   | 33.4              | 25.1               | -    | -                  | 100                         | 99                           | 10                              | 17+                | 7              | 5                  | 5              |
| MB                    | 4A   | 11/03                   | 16.4              | -5.1               | 365  | 5.7                | 100                         | -                            | 30+                             | 30+                | 30+            | 31+                | 31+            |
| MB                    | 4B   | 11/03                   | 24.2              | -5.1               | 108  | 1.2                | 100                         | -                            | 30+                             | 30+                | 30+            | -                  | -              |
| MB                    | 4A   | 06/04                   | -                 | -                  | 365  | 6.3                | -                           | -                            | 25+                             | 25+                | 25+            | 14                 | 16             |
| MB                    | 4B   | 06/04                   | -                 | -                  | 279  | 12.0               | -                           | -                            | 25+                             | 25+                | 25+            | -                  | -              |
| MB                    | 4A   | 05/05                   | 24.3              | 13.4               | 443  | 4.9                | 100                         | 99.8                         | 17                              | 22                 | 22+            | 14                 | 15             |
| MB                    | 4B   | 05/05                   | 28.9              | 13.4               | 273  | 5.3                | 100                         | 100                          | -                               | -                  | -              | -                  | -              |
| MB                    | 6A   | 11/05                   | 27.3              | 3.6                | -    | -                  | 100                         | 100                          | 25                              | 19                 | 29             | 22                 | 25             |
| MB                    | 6B   | 07/06                   | 40.0              | 25.1               | 210  | 4.3                | 100                         | 100                          | 9                               | 2                  | 3              | 4                  | 18+            |
| MB                    | 6A   | 11/06                   | 27.6              | 5.1                | -    | -                  | 100                         | 100                          | -                               | -                  | -              | -                  | -              |
| MB                    | 6B   | 11/06                   | 29.9              | 5.1                | -    | -                  | 100                         | 100                          | -                               | -                  | -              | -                  | -              |
| MB                    | 7    | 06/06                   | 24.7              | 13.2               | 242  | 3.5                | 100                         | 100                          | 25                              | 33+                | 33+            | -                  | -              |

<sup>1</sup> 3 consecutive weeks of insects detected in both roll stand and sifter floors or in tailings

<sup>2</sup> Weeks for populations to return to 100% pre-treatment levels.

- Initial numbers too low to estimate rebound, or samples not taken

+ Insects were not found 3 consecutive weeks in a row, or had not reached 100% of pre-treatment during study



One reason that the populations rebounded so quickly in Mill 8 after the fumigation could be that these unfumigated silos served as a source of insects that quickly reinfested the mill.

Mill 5 used the highest concentration tested in these trials (1280 gh/m<sup>3</sup>). Although insects were found in pheromone traps regularly on the sifter floor for 4 weeks right after the fumigation, these numbers quickly dropped and remained low the remaining 14 weeks post fumigation sampling. One suggestion is that the insects found in the traps were on the window sills that were covered with polyethylene and outside the fumigated area, but still inside the mill. Insects on the roll stand floor did not rebound within the 18 weeks of sampling after the fumigation. There were some insects in the rebolt sifter tailings right after the fumigation. This mill did not divide live and dead insects, and these insects were probably dead insects that were being flushed from the system after the fumigation.

In the December 2004 fumigation in the packing plant with the phosphine combination treatment, the insects were consistently found in the traps after 29 weeks, the same length time it took the insects to the pre-treatment levels. Insects in the rebolt sifter tailings were regularly found after 23 weeks, and reached pre-treatment levels after 33 weeks.

In the July 2006 phosphine fumigation in the mill, the insects were regularly found in the traps after 10 weeks and returned to pre-treatment levels in the sifter floor after 7 weeks and never returned to original levels in the roll stand floor. Insects in the rebolt sifter tailings were regularly found after 5 weeks, and reached pre-treatment levels at 5 weeks after treatment. Rebolt sifter tailings should be interpreted with caution because although one rebolt sifter is located in the mill and the other in the packing plant both pull flour from the same bins.

The efficacy of methyl bromide fumigations were assessed in Mills 4, 6 and 7. In Mill 4A the two methyl bromide fumigations were done at the upper level of the label rate Mill 4A (November 2003: 38-50 g/m<sup>3</sup>, 377 gh/m<sup>3</sup>; May 2005: 24-67 g/m<sup>3</sup>, 447 gh/m<sup>3</sup>) The label rate for methyl bromide is 16 to 48 g/m<sup>3</sup>. For the fumigation in November 2003 with methyl bromide, insect populations remained low both in the traps and in the rebolt sifter tailings for over 31 weeks. For the fumigation in June 2004, the insects were not consistently found in the traps in the 22-week sampling after the fumigation. Insect levels in the tailings rebounded after 16 weeks. For the fumigation in May 2005, the insects were consistently found in the traps after 17 weeks, and had returned to the pre-treatment levels after 22 weeks on the roll stand, but did not on the sifter floor. Insect levels in the tailings rebounded after 15 weeks. The new mill, Mill 4B, after the first fumigation with methyl bromide in November 2003, insects caught in traps remained at low levels for the rest of the study, so it is impossible to determine the efficacy of the subsequent fumigations.

The methyl bromide fumigations in Mill 6 were conducted at the same time as the phosphine combination fumigations. In the December 2005 fumigation in the mill plant with methyl bromide the insects were consistently found in the traps after 25 weeks (vs. 29 weeks with phosphine), the insects returned to pre-treatment levels in 29 weeks (vs. 29 weeks with phosphine). Insects were consistently found in rebolt sifter tailings after 22 weeks (vs. 23 weeks with phosphine), the insects returned to pre-treatment levels in 25 weeks (vs. 33 weeks with phosphine). In the July 2006 fumigation in the packing plant, the insects were regularly found in the traps after 9 weeks (vs. 10 weeks with phosphine) and returned to pre-treatment levels after 2-3 weeks (vs. 7 to over 17 weeks with phosphine). Insects were consistently found in rebolt sifter tailings after 4 weeks (vs. 5 weeks with phosphine), the insects never returned to pre-treatment levels in 18 weeks of sampling (vs. 5 weeks with phosphine).

Although the two buildings, mill and packing plant, are well isolated from a fumigation perspective, they are not from a product flow perspective. Flour and any insects in the flour

would flow from the mill to the packing plant, but little product flows from the packing plant to mill. The insect Dome trap that caught over 50% of the insects trapped in the packing plant were from one trap on the first floor, close to the barrels that hold the tailings from the rebolt sifter. Hence, a large insect population in the mill could increase the insects caught in traps in the first floor packing plant. Also for the rebolt sifter tailings, the two sifters pull flour from the same bins.

Finally, the fumigation with methyl bromide in Mill 7 saw insects consistently in the traps after 25 weeks, but populations had not reached pre-treatment levels when the study ended after 33 weeks.

## DISCUSSION

For several reasons it is difficult to make direct comparisons between treatments because they were carried out at different mills or the same mill treated at different times. Mills differ in age of the building, age of equipment, cleaning practices, location, which effects outside temperatures and humidity, grain milled and product produced. All these factors affect the pest pressure that a facility faces. A good example is Mill 4A and Mill 4B. Both mills were fumigated at the same time. Mill 4A has older equipment in an old building with wooden floors and insect populations are difficult to control. Mill 4B has new equipment in a new building and after the initial fumigation in December 2003, insects populations remained very low throughout 2.5 years of the study.

There is limited replication with regard to treatments. There were three heat treatments, done in two different ways. There were four sulfuryl fluoride treatments, one at the low dose and three at the high dose. There were two phosphine combination treatments. There were seven methyl bromide fumigations. Even treatments done in the same mill are done at different times, so pest pressures may be different from one treatment to another. For example, the pest pressure will be much greater in the summer than the winter. One would expect that all things being equal, the rebound of insect populations from a treatment in the fall would be slower than a treatment done in the spring. There were only four fumigations with sulfuryl fluoride, each with a different concentration or in a different mill. Sulfuryl fluoride does not yet have food tolerances in Canada, so some of the bins contained flour, but were isolated from the gas to prevent food contact. This may have been the reason that insects populations rebounded quickly in one mill that used sulfuryl fluoride. In the USA and other countries, food products can be treated with sulfuryl fluoride and do not need to be removed from the facilities. Whereas, methyl bromide can be used to treat flour remaining in the mill. The phosphine, heat and carbon dioxide had only two treatments, and there are problems using the twinned methyl bromide for comparisons.

Despite the limitations, this project has provided Canadian flour millers and pest control operators with many opportunities to test several alternatives to methyl bromide in their facilities. Sulfuryl fluoride, heat and phosphine combination treatment (phosphine, heat and carbon dioxide) can control insect populations in flour mills for over 18 weeks.

For any of the fumigants tested, methyl bromide, sulfuryl fluoride or phosphine, improved sealing in Canadian flour mills before fumigation, would allow for effective fumigations with less gas. Many flour mills in the USA are much better sealed than the Canadian mills we tested. The American mills have Half Loss Times twice that found in mills in this study. Higher temperatures would also improve the efficacy of any of the fumigants. For example, the CTP for sulfuryl fluoride needed to control *T. castaneum* eggs is 1768 gh/m<sup>3</sup>

Table 3. Stage specific toxicity for various fumigants.

| Fumigant          | Temp. (°C) | Time (h) | Insect   | CTP to kill 95% of the population (gh/m <sup>3</sup> ) <sup>1</sup> |        |             | Reference         |                                |
|-------------------|------------|----------|--|---|--------|-------------|-------------------|--------------------------------|
|                   |            |          |  | Egg   | Larvae | Pupae Adult |                   |                                |
| sulfuryl fluoride | 27         | 16       | <i>T. confusum</i>                                 | 1125  | -      | -           | Kenga, 1957       |                                |
|                   | 27         | 16       | <i>Sitophilus granarius</i> (L.)                   | 794   | 14     | 14          | Kenga, 1957       |                                |
|                   | 25         | 24       | <i>T. confusum</i>                                 | 498   | -      | -           | Bell et al., 1999 |                                |
|                   | 25         | 24       | <i>T. castaneum</i>                                | 1368  | -      | -           | Bell et al., 1999 |                                |
|                   | 25         | 24       | <i>T. castaneum</i>                                | 1768  | -      | -           | Bell et al., 1999 |                                |
|                   | 30         | 24       | <i>T. castaneum</i>                                | 1154  | -      | -           | Bell et al., 1999 |                                |
| methyl bromide    | 25         | -        | <i>T. confusum</i> <sup>2</sup>                    | -   | -      | 90          | 60                | Heseltine & Thompson, 1974     |
|                   | 15         | -        | <i>T. confusum</i> <sup>2</sup>                    | 96  | 255    | 255         | -                 | Bell et al., 1988 <sup>3</sup> |
| phosphine         | 25         | -        | <i>T. confusum</i> <sup>2</sup>                    | 65  | 98     | 251         | -                 | Bell et al., 1988 <sup>3</sup> |
|                   | 27         | 2        | rice weevil  | 37  | 10     | 44          | 27                | Krohne & Lindgren, 1958        |
|                   | 25         | 48       | <i>Plodia interpunctella</i> (Hübner) <sup>2</sup> | 77  | 1      | 1           | -                 | Bell, 1976                     |
|                   | 27         | 16       | <i>T. confusum</i>                                 | 2   | 0.1    | 0.3         | 0.3               | Lindgren & Vincent 1966        |

<sup>1</sup> g/m<sup>3</sup> = mg/L = ounces/1000 ft<sup>3</sup>

<sup>2</sup> complete kill

<sup>3</sup> methyl bromide at 4.9 g/L

at 25°C, but only 1154 gh/m<sup>3</sup> at 30°C (Table 3, Bell and Savvidou, 1999). Comfort heaters could be used during the summer to increase mill temperatures, or additional heaters could be used for fall fumigations.

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