New phosphine grain fumigation technology in Cyprus using the SIROFLO® /ECO₂FUME® flow-through method

A. Varnava¹, J. Potsos¹, G. Russell² and R. Ryan³

Abstract

A quantity of 560 tons of barley was fumigated with 620 grams of phosphine [ECO₂FUME® : 2wt% phosphine in liquid carbon dioxide] using the SIROFLO® system which was installed at the Cyprus Grain Commission, Larnaca grain storage facility in 1995. The grain stored in a vertical metal silo was fumigated in-situ with a low concentration of phosphine/air mixture introduced through the aeration ducts into the grain mass. The grain, heavily infested with Tribolium spp., Staphilus spp., Rhyzopertha dominica and Oryzaephilus surinamensis at ~33 adults/kg, was exposed to a minimum phosphine concentration of 38 ppm for 16 days. Bio-assay traps-tubes using adults insects were inserted at different locations at a depth of 20 cm below the grain surface.

During the fumigation process phosphine concentration was measured daily at 25 locations in the grain mass and around the silo. After the fumigation the silo was unloaded and 60 grain samples were taken and no live insects were detected. In all six bio-assay traps no live insects were detected.

The use of SIROFLO® system and the ECO₂FUME® gas mixture resulted in:
1. a completely successful fumigation of the grain;
2. an easy and automatic phosphine application process;
3. treatment of grain in-situ (no need to move grain);
4. improved worker safety;
5. no chemical residues in the grain.

Introduction

SIROFLO®, a CSIRO (Commonwealth Scientific Industrial Research Organization, Australia) developed and patented technique, is a low positive pressure distribution system for fumigation of grain in situ with a 2wt% phosphine in liquid carbon dioxide which is patented by BOC Gases (British Oxygen Corporation, Gases, Australia). SIROFLO® fumigation technique was firstly implemented on a commercial basis in Australia in 1988 after a series of trials in vertical silos that began in 1985 (R. G. Winks, 1992; R. G. Winks and G. F. Russell, 1997). In 1995, after an agreement between CSIRO/BOC, Australia and the Cyprus Grain Commission, the SIROFLO® system was installed in Cyprus, in a complex of eight vertical, non-airtight, corrugated hopper bottom metal silos of 1000 cubic meters capacity each at Larnaca storage facilities of the CGC. The SIROFLO® system was installed in Cyprus a. for demonstration purposes as an advanced grain fumigation technology during the 5th International Conference on Controlled Atmosphere and Fumigation of Stored Products held in Cyprus in 1996, and b. for further improvements in the technology of grain protection in Cyprus by testing and implementing the most advanced available technology at this time. After a series of trials defining the serious advantages of this system, the Cyprus Grain Commission signed an agreement with BOC/CSIRO for the installation and implementation of SIROFLO® technology and the uninterrupted supply of the phosphine in liquid carbon dioxide in cylinders ECO₂FUME® (R. F. Ryan, 1996). The installation of SIROFLO® system in other grain storage facilities of Cyprus Grain Commission, which are not fully sealed, is now in process. Simultaneously, the storage facilities of Cyprus Grain Commission where the SIROFLO® system is installed are now an international demonstration site for the SIROFLO® and ECO₂FUME® advanced fumigation technology

Material and Methods

The SIROFLO® system in Cyprus is installed in a complex of eight vertical non-airtight metal corrugated hopper bottom silos each of 1000 cubic meters capacity each. A low concentration of phosphine gas [ECO₂FUME® : 2wt% phosphine in liquid carbon dioxide] from cylinders is mixed
with an air stream from a blower. The air-phosphine gas mixture is introduced into the base of the silos through the existing aeration ducts. The air stream produces a small amount of pressure that causes the phosphine gas to spread evenly through the entire grain mass in the silo. By using the gas pressure regulator and the flowmeter control it is possible to adjust the concentration of phosphine gas in the grain mass (Fig. 1 and 2). All eight silos are connected to the SIROFLO® system and there is the possibility to fumigate any number of silos simultaneously. Each bin silo has a diameter of 8 meters, a cross-section of 50 sq. meters and a height of 18 meters. The schematic diagram of the SIROFLO® system is shown in Fig. 3 and Fig. 4.

One bin connected to the SIROFLO® was filled in September 1996 with 560 tons of local barley (moisture content $9.8\cdot 10.5\%$, test weight $48.3 - 51.9$ kg/hl and initial grain temperature $25 - 33^\circ C$). Before receiving the barley into the silo, grain samples were withdrawn from each one of the 32 trucks using a mechanical probe-a-load sampler, taking a composite sample from five points of approximately 4.5 kg each. All samples were checked for insect infestation (number of adults and species) and the results are shown in Table 1.

![Image](image-url)  

Fig. 1. Adjust the concentration of phosphine gas in the grain mass by using the gas pressure regulator and the flowmeter control.
Fig. 2. All silos are connected to the SIROFLO® system.

Table 1. Initial Insect Infestation in grain samples taken from each truck during filling of the silo before fumigation with SIROFLO® /ECO2FUME® Flow-Through Method.

<table>
<thead>
<tr>
<th>Total weight of sampled grain, tons</th>
<th>Number of grain samples</th>
<th>Weight of each sample, kg</th>
<th>Total weight of grain checked for insects</th>
<th>Tribolium spp.</th>
<th>Sitophilus spp.</th>
<th>Rhyzopertha dominica</th>
<th>Oryzaephilus surinamensis</th>
<th>Insect Species and calculated average number of adult insects detected in 1 kg of grain sample</th>
<th>Total number of adults insects</th>
</tr>
</thead>
<tbody>
<tr>
<td>560</td>
<td>32</td>
<td>4.5</td>
<td>144</td>
<td>20</td>
<td>0.5</td>
<td>3.5</td>
<td>9.3</td>
<td>33.3</td>
<td></td>
</tr>
</tbody>
</table>
Fig. 3. Pipe routing diagram using SIROFLO® system operation for 8 silos at Larnaca silo complex in Cyprus.

Fig. 4. Schematic diagram of the SIROFLO® system connected to the vertical silos as shown in Fig. 1.
The SIROFLO® system was adjusted to work with an Airflow Pressure of 440 kPa and Phosphine Flow of 0.72 l/ min. For this fumigation it was used one cylinder of 32 kg of ECOzFUME® 2wt% phosphine in liquid carbon dioxide, which is equal to 620 grams of a.i. of phosphine. The fumigation process lasted 16 days. It is important to mention that the same quantity of phosphine could be used for fumigation of a full or partly full silo since the required amount of phosphine depends on the silo cross-section.

After the cylinder was emptied and the fumigation was completed, the blower was left to pump clear air through the grain mass for one day in order to blow out any phosphine residues.

The phosphine concentration during the trial was monitored in several points inside and outside the grain mass and in the aeration ducts using a Bedfont EC80 phosphine monitor. For air sucking and monitoring the phosphine concentration, special metal pipes with wire mesh at the edge and plastic tubes inside were inserted into the grain mass, at different depths and locations (10 cm, 30 cm and 300 cm from the grain surface) at the top of grain in the silo. Phosphine concentration was measured also at the grain surface level and at 30 cm above the grain surface. Similar tubings for sucking air and monitoring phosphine concentration were inserted into the grain at a height of 3 meters above the silo bottom, 2 meters from the silo walls. Other points for monitoring phosphine were a. on the space between the roof and silo wall, appr. 1.5 meter above the grain surface, b. inside the aeration duct (before entering the silo), c. inside the pipe (above the valve) and d. inside the pipe (below the valve). The phosphine concentration in different points during the fumigation process is shown in Table 2.

Six bio-assay tubes with mixed-aged insects of different species extracted from the received grain (mainly Tribolium spp., Sitophilus spp., Rhyzopertha dominica and Oryzaephilus surinamensis), containing milled wheat, crushed oat and wheat flour, were inserted 20 cm below the grain surface, at the top of the silo. Four of these bio-assay tubes with insects were placed on the peripheral of the bin, 1 meter from the wall and the other two tubes on the highest point of the grain (grain peak). Another two bio-assay tubes with insects and feed mixture were kept in the laboratory as a control samples (temperature: 19 – 26°C, rh: 45 – 68%). After the fumigation was completed, the mortality of insects was counted. The results of this bio-assay is shown in Table 3.

After fumigation was completed and the monitoring tubimg and bio-assay tubes were removed, the whole grain was moved into another empty silo taking during transportation 60 grain samples of approximately 1.5 kg each for insects detection. The results are shown in Table 4.

**Results and Discussions**

The results of grain insect infestation analysis and phosphine concentration measurements at different stages of the trial are shown in Tables 1, 2, 3 and 4.

The grain before fumigation was heavily infested by two primary and two secondary insect species with an average number of 33 adult insects per kg. All 32 grain samples withdrawn from 560 tons of grain were found to be infested. From unpublished data it is known that resistance of Tribolium castaneum to phosphine exists already in some places in Cyprus. The initial insect infestation of grain used in this trial is shown in Table 1.

Fumigation lasted 16 days and the phosphine concentration was measured daily at 25 locations in the grain mass and around the silo. The phosphine concentration is shown in Table 2.

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**Table 2.** Phosphine concentration in ppm at different points outside and inside grain mass in a silo and in aeration ducts during a 16-days fumigation with SIROFLO® /ECOzFUME® Flow-Through Method.

<table>
<thead>
<tr>
<th>Location of phosphine measurements</th>
<th>Days</th>
<th>30 cm above grain surface</th>
<th>0 cm below grain surface</th>
<th>10 cm above grain surface</th>
<th>30 cm below grain surface</th>
<th>3 m above silo bottom</th>
<th>Near gap</th>
<th>Inside duct, before entering silo wall</th>
<th>Inside pipe, before entering silo valve</th>
<th>Released phosphine mixture, kg</th>
<th>Released phosphine, a.i., gr</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>1-4</td>
<td>3-6</td>
<td>37-58</td>
<td>40-68</td>
<td>50-70</td>
<td>54-72</td>
<td>55-79</td>
<td>54-80</td>
<td>54-78</td>
<td>31</td>
</tr>
</tbody>
</table>

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**Table 2.** Phosphine concentration in ppm at different points outside and inside grain mass in a silo and in aeration ducts during a 16-days fumigation with SIROFLO® /ECOzFUME® Flow-Through Method.
Table 3. Mortality of mixed-aged adults insects in bio-assay tubes placed at 20 cm below grain surface in a silo after a 16-days fumigation with SIROFLO® /ECO₂FUME® Flow-Through Method.

<table>
<thead>
<tr>
<th>Number of bio-assay tube</th>
<th>Tribolium spp.</th>
<th>Staphylius spp.</th>
<th>Rhyzopertha dominica</th>
<th>Oryzaephilus surinamensis</th>
<th>Total insects</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>alive</td>
<td>dead</td>
<td>alive</td>
<td>dead</td>
<td>alive</td>
</tr>
<tr>
<td>1</td>
<td>0</td>
<td>50</td>
<td>0</td>
<td>50</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>0</td>
<td>50</td>
<td>0</td>
<td>50</td>
<td>0</td>
</tr>
<tr>
<td>3</td>
<td>0</td>
<td>50</td>
<td>0</td>
<td>50</td>
<td>0</td>
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<td>4</td>
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<td>50</td>
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<td>50</td>
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<td>5</td>
<td>0</td>
<td>50</td>
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<td>50</td>
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<td>6</td>
<td>0</td>
<td>50</td>
<td>0</td>
<td>50</td>
<td>0</td>
</tr>
<tr>
<td>Total insects in 6 tubes</td>
<td>0</td>
<td>300</td>
<td>0</td>
<td>100</td>
<td>0</td>
</tr>
</tbody>
</table>

Control: Bio-assay tubes # 7 and # 8 kept in laboratory conditions (19 – 26°C, 45 – 68 % r. h.)

Table 4. Mortality of adult insects in 60 grain samples representing 560 tons of Barley taken during transportation of grain after completing the fumigation with SIROFLO® /ECO₂FUME® Flow-Through Method.

<table>
<thead>
<tr>
<th>Total weight of sampled grain, tons</th>
<th>Number of grain samples</th>
<th>Weight of each sample, kg</th>
<th>Total weight of grain checked for insects, kg</th>
<th>Number of alive insects detected in 60 grain samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>560</td>
<td>60</td>
<td>1.5</td>
<td>90</td>
<td>0 0 0 0</td>
</tr>
</tbody>
</table>

It is very important that the phosphine concentration inside the grain mass during all 16 days of fumigation was maintained constant and higher than 37 ppm reaching at deeper grain layers 70 – 80 ppm. This concentration was sufficient to kill adult stages of all the above mentioned insect species (Tables 3 and 4). In contrary to the high phosphine concentration inside the grain mass and inside the ducts before entering the silo, the phosphine concentration on grain surface and above the grain surface at the top of grain under the silo roof was low (1 – 6 ppm) during the whole period of the 16 days.

All adult insects in bio-assay tubes inserted at 20 cm below the grain surface were killed at the end of 16-days fumigation. The phosphine concentration range at this layer was 37 – 60 ppm. The results are shown in Table 3.

In all 60 grain samples taken after fumigation no alive adult insects were detected. The results are shown in Table 4.

The uniformity and accuracy in grain sampling, the large number of taken samples, the sufficient quantity of grain checked for infestation and the absence of adult alive insects in the mass of grain after fumigation show that the fumigation with SIROFLO® /ECO₂FUME® Flow-Through Method provided a grain actually free from alive adult insects.

Conclusions

1. The use of SIROFLO® system and the ECO₂FUME® gas mixture resulted in:
   1. a completely successful fumigation of the grain
   2. an easy and automatic phosphine application process
   3. treatment of grain in-situ (no need to move grain and therefore no machine-equipment wear, no electric-power consumption, no need in extra emptied silo space)
   4. improved worker safety
   5. no chemical residues in the grain

2. The use of SIROFLO® /ECO₂FUME® Flow-Through Method and their modifications will be a serious improvement in the grain storage protection, grain
industry and grain hygiene.
3. A further investigation is needed in order to increase the phosphine concentration in the space between the grain surface and the silo roof.

Acknowledgments

Thanks to Dr. Robert Winks from CSIRO, Australia, for his untiring and decisive support to introduce and install the SIROFLO® system and implement this technology in Cyprus during the International CAF Conference and afterward and his valuable contribution on many details regarding the system and this trial. Thanks also to BOC Gases, Australia and CSIRO, Australia for the excellent cooperation regarding the introduction and implementation of SIROFLO® and ECOzFUME® novel technologies in Cyprus. We thanks Mr. C. Patsalides, present Chairman, Mr. A. Tryfonides, ex-Chairman and Mr. C. Archimandrides, Manager of Cyprus Grain Commission for their support for the further installation and use of the SIROFLO®/ECOzFUME® in Cyprus. Thanks also to all personnel of Inspection Department, Technical Department and Larnaca Silo Distric Office of the Cyprus Grain Commission for their valuable assistance to install and operate the system, take samples and carry out all analyses and measurements of this trial.

References