

Donahaye, E.J., Navarro, S. and Leesch J.G. [Eds.] (2001) *Proc. Int. Conf. Controlled Atmosphere and Fumigation in Stored Products*, Fresno, CA. 29 Oct. - 3 Nov. 2000, Executive Printing Services, Clovis, CA, U.S.A. pp. 149-153

## PRELIMINARY STUDY ON CHESTNUT INSECT DISINFESTATION WITH SULFURYL FLUORIDE

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### ABSTRACT

At harvest, chestnuts are often infested at a high rate (10-20%) with young larvae of the chestnut fruit tortrix, *Cydia splendana* (Hübner) and/or the chestnut weevil, *Curculio elephas* (Gyllenhal). These larvae are barely or not at all visible and do not cause any loss in quality if they are killed early on. The systematic fumigation of chestnuts with methyl bromide (MB) ( $50 \text{ g m}^{-3}$  for 12 h, at  $15^\circ\text{C}$ ) before their commercialization has become common practice. Because the use of MB must be discontinued by the end of 2004, alternatives have to be found. A good candidate could be sulfuryl fluoride, a gas that has been used for 40 years in the USA against the dried wood termite. It has not been able to compete with MB in post-harvest use essentially because the egg stage is much more tolerant than the other stages. However, in the case of chestnuts, this stage is not present and therefore the problem does not arise. Fumigations were carried out in 43 Altuglas chambers with a single dose of  $40 \text{ g m}^{-3}$ , variable gas exposure periods of 2, 4, 6, 8 and 10 hours at  $15^\circ\text{C}$  which corresponds to the normal temperature of chestnuts at the time of harvest. The loading rate was 64% (8 kg of fruit) or 80% (10 kg). Results showed that this dose at  $15^\circ\text{C}$  or higher for an exposure period of 8 h is completely effective. Furthermore, sulfuryl fluoride is much more efficient against the chestnut weevil than against the chestnut fruit tortrix which is contrary to the effect of MB. Finally, sorption was much lower with sulfuryl fluoride than with MB, which would explain its higher efficacy. No significant odor or particular taste was noted in the taste test, and a panel of 4 people rated the nuts treated with sulfuryl fluoride to be superior even to the control.

### INTRODUCTION

At their time of harvest, chestnuts are often heavily infested at levels of up to 20% or more, with an average of 5 to 10%, by young larvae of the chestnut fruit tortrix (CFT), *Cydia splendana* (Hübner) (Lepidoptera, Tortricidae) and/or with the chestnut weevil (CW), *Curculio elephas* (Gyllenhal). (Coleoptera, Curculionidae). These young larvae are either completely invisible, or hardly visible, and do not cause any loss in quality if they are killed early on. However, if they are allowed to develop, they consume a large part of the chestnut meat, and then exit the chestnut to

pupate. This phase occurs between the time of commercialization and that of consumption and consequently provides a negative marketing image. For this reason, the systematic fumigation of chestnuts with methyl bromide (MB) before their commercialization has become common practice. In 1978, it was demonstrated that CW were more tolerant than CFT to MB and a fumigation standard was proposed that took into consideration this fact and also the very large sorption of MB into the chestnut. This resulted in the following fumigation schedule: temperature  $\geq 15^{\circ}\text{C}$ , exposure time 12 hours, dose  $50 \text{ g/m}^3$ .

Since MB must be phased out of use by the end of 2004, alternatives have to be found. Sulfuryl fluoride ( $\text{SO}_2\text{F}_2$ ) could be a good candidate, as it can be utilized under almost the same conditions as MB, particularly with regard to its exposure time (Schneider and Hartsel, 1999). Although, there is one limitation in that the egg stage requires a high concentration  $\times$  time (Ct) product (Bell *et al.*, 1999), in the case of chestnuts, this stage is not present in the fruit, as the adults lay their eggs on the leaves or burs on the trees and it is the young larvae which penetrate the fruit. For this reason, Sulfuryl fluoride has been used for similar trials on walnuts against the Codling moth, which infests the nuts in the same manner (Leesch, 1999; Zettler and Gill, 1999).

## MATERIALS AND METHODS

### **Efficacy trials**

*Fumigation:* The fumigations were carried out in 43-L 'Altuglas' chambers. The gas was injected into the chambers with a Hamilton 1-L gastight syringe. Homogenization was carried out using a 10 L/min pump to circulate the gas for 15 minutes. An arbitrary dose rate of  $40 \text{ g/m}^3$  was chosen, and the chambers were loaded with 8 kg or 10 kg of fruit, corresponding to 64% and 80% of chamber volume. A fumigation temperature of  $15^{\circ}\text{C}$  was chosen since this corresponds to the commonly experienced temperatures of chestnuts at the time of harvest. Fumigations were carried out for the following exposure times: 2, 4, 6, 8 and 10 hours.

At the end of fumigations, the chambers were taken outdoors and opened to enable the fumigant to escape. Several minutes later, the different chestnut batches were placed in separate crates.

*Observations on mortality:* After 48 hours, chestnut inspection was carried out by cutting open and shelling the nuts. Counts of live and dead insects were made according to species.

### **Sorption trial**

*Fumigation:* These fumigations were carried out in 11-L glass jars. Gas was injected into the jars using a 100 ml Hamilton syringe. MB was used as the reference gas. Both gases were administered at the same dose of  $40 \text{ g/m}^3$ . Homogenization was obtained by re-circulating the gas using a 10 L/min pump for 15 minutes.

The fumigation parameters were as follows: exposure time: 6 h, treatment temperature: 15°C. The variable parameters were: loading rate for the first series and MB, 42.5% (2.5 kg of nuts); for the second series: 67% (4 kg of nuts). The nuts that were used in the sorption test, also served as part of a taste test 48 hours later and also provided material for residue analysis (20 to 30 chestnuts per series).

*Gas concentration measurement:* Gas concentrations were measured using a Varian 3300 gas chromatograph equipped with a thermal conductivity detector. Gas samples were removed from the jars with a 250  $\mu$ L syringe. Each recorded concentration was obtained from the average of three GC injections. The measurement periods were as follows: 30 min, 1 h, 1h 30 min, 2 h, 3 h, 4 h, and 6 h for each trial, and additional 8 h and 10 h for the longer exposure times of the efficacy trials. Gas measurements during the efficacy trials were carried out with a Micro GC 2000 Chrompack-Varian chromatograph. Unfortunately its reproducibility proved to be defective due to the malfunctioning of the automatic injection valve. Therefore these results were considered unreliable.

**Organoleptic evaluation of chestnuts fumigated with sulphuryl fluoride and MB**  
Four batches of 15 chestnuts were prepared:

- Batch A: nuts fumigated with sulfuryl fluoride at a loading rate of 67.5%;
- Batch B: nuts fumigated with MB at a loading rate of 42.5%;
- Batch C: control, un-fumigated nuts;
- Batch D: nuts fumigated with sulfuryl fluoride at a loading rate of 42.5%.

Each batch was put in a microwave and cooked for 2 minutes. For evaluation, each batch was allotted a grade on a scale of 1 - 4 by a jury of four people, a score of 4 being the best.

## RESULTS

### Biological Efficacy

Table 1 presents the efficacy of  $\text{SO}_2\text{F}_2$  against CFT and CW according to exposure time. For a 40  $\text{g}/\text{m}^3$  dose rate at a temperature equal to or higher than 15°C, eight h is sufficient to achieve total kill. It was also noted that sulfuryl fluoride is much more effective against CW than against CFT, whereas the opposite is true for MB.

In general it can be seen that sulfuryl fluoride is more effective than MB for the fumigation of chestnuts. This is because with MB 12 h are required, at 50  $\text{g}/\text{m}^3$ , to obtain complete kill of CW. This may be due either to a greater intrinsic toxicity of sulfuryl fluoride or to lower sorption. Unfortunately, we were unable to determine the Ct products that corresponded to each dose.

TABLE 1  
Mortality of the larvae of chestnut fruit tortrix (CFT) and chestnut weevil (CW) inside chestnuts, after exposure to sulfuryl fluoride at 40 g/m<sup>3</sup>

Exposure time (h)	Chestnut fruit tortrix		Chestnut weevil	
	Total number observed	Mortality %	Total number observed	Mortality %
Control	232	52	23	0
2	190	90	16	88
4	237	94	23	100
6	215	98	32	100
8	214	100	12	100
10	215	100	14	100

### Sorption

The levels of sorption of sulfuryl fluoride and MB at the end of 6 hours exposure, are given in Table 2

TABLE 2  
Comparison of sorption of sulfuryl fluoride and methyl bromide into chestnuts after 6 h exposure

Fumigant	Loading %	Ct obtained g·h/m <sup>3</sup>	Initial concentration (IC) (5 min after fumigation) g/m <sup>3</sup>	Final concentration (FC) g/m <sup>3</sup>	Sorption % (*)
Sulfuryl fluoride	67	145	40.4	17.4	58
Sulfuryl fluoride	42.5	225	39.7	29	27
Methyl bromide	42.5	44	40.1	5.8	86

(\*) Sorption % =  $\frac{IC-FC}{IC} \times 100$

From the Table it can be seen that sorption of sulfuryl fluoride is much lower than that of MB. This may explain the greater "efficacy" of sulfuryl fluoride if we compare the Ct products obtained at a loading percentage of 42.5%, namely: 225 g·h/m<sup>3</sup> for SO<sub>2</sub>F<sub>2</sub> compared to 44 g·h/m<sup>3</sup> for MB. Even at a 67% loading level, sorption of sulfuryl fluoride is not as high as MB at 42.5% and the Ct product is much higher: 125 g·h/m<sup>3</sup> compared to 44 g·h/m<sup>3</sup>.

### Organoleptic evaluation of chestnuts fumigated with sulphuryl fluoride and MB

The results of the taste evaluations are given in Table 3.

TABLE 3

Results of taste evaluations of chestnuts fumigated with sulfuryl fluoride at 40 g/m<sup>3</sup> or with methyl bromide at 50 g/m<sup>3</sup>

Batch No.	Overall grade
Batch D: Sulfuryl fluoride at a loading rate of 42.5 %.	15
Batch A: Sulfuryl fluoride at a loading rate of 67.5 %.	13
Batch C: Control	9
Batch B: Methyl bromide at a loading rate of 42.5 %.	8

The taste panel detected no significant off-odour or particular taste during their evaluations. Despite some heterogeneity between the chestnuts, the nuts treated with sulfuryl fluoride clearly came out ahead in the organoleptic evaluations.

### DISCUSSION

These findings show that the efficacy of sulfuryl fluoride is excellent for the fumigation of chestnuts and is superior to MB. According to the results obtained, it would appear that a dose of 40 g/m<sup>3</sup> for a duration of 8 h at a temperature  $\geq 15^{\circ}\text{C}$  is sufficient to control the two pests of chestnut, the chestnut fruit tortrix, and the chestnut weevil. The gas imparts no taste to the nuts and sulphuryl fluoride fumigated nuts were even preferred by the taste panel. These results will have to be confirmed on a larger scale. Fumigant residues have not yet been analyzed.

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