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Evaluation of the use of sulphuryl fluoride (Profume) in the malting industry in the United Kingdom

by

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1. ABSTRACT

A literature review was undertaken to evaluate the potential of sulphuryl fluoride (SF) for use in the malting industry. Searches revealed a total of 915 references for SF, of which 59 were relevant to this review.

Sulphuryl fluoride is currently registered for use in structures such as flour mills and food manufacturing premises in the United Kingdom under the trade name Profume®. A number of studies have shown SF to be an effective replacement for methyl bromide (MB) in these situations. However, in the United Kingdom heating is required to maintain the temperature at 25°C to improve efficacy in order to make a 24 hour treatment possible.

There is no evidence that sorption of SF or corrosion due to SF should give any cause for concern. However, there is a need to determine the level of fluoride and sulphate residues likely to be encountered if SF is to be used on malt or barley.

Unfortunately there are no plans to extend the registration of Profume® to cover malt or barley and so, unless the manufacturers can be persuaded of a commercial advantage in extending the registration beyond their current plans, the use of SF in the malting industry would be limited to the fumigation of structures empty of barley and malt.

2. INTRODUCTION

The malting industry in the United Kingdom relies on phosphine for the control of storage pests. However, the misuse of phosphine has led to the development of phosphine resistant strains of insects in many countries (Champ and Dyte, 1976). With the withdrawal of methyl bromide (MB), and the consequent increasing reliance on phosphine, the level of resistance is likely to increase. The withdrawal of MB will also remove the method of first choice of dealing with phosphine resistant insects. There is therefore a need to investigate alternative fumigants.

One such alternative is sulphuryl fluoride (SF) which is a fumigant manufactured by Dow Agrosciences (Dow) that has been used for termite control under the trade name Vikane since the 1950s.

It has now been registered for use in flour mills in the United Kingdom under the trade name Profume®.

The purpose of this review is to evaluate the literature available on SF, with a view towards its use in the malting industry.

3. SCOPE OF THE REVIEW

In 1996 a literature review on the sorption, penetration, taint, residues, corrosion and toxicity to insects of SF was conducted at the Central Science Laboratory (Bell, 1996). The various literature searches undertaken for the review identified a total of 850 references for SF. Of these, 138 records were examined and 34 were referenced in the report.

This subsequent review for the malting industry has identified a further 65 records published since 1996, 25 of which were selected for more detailed evaluation together with the 34 records referenced in the previous review.

4. SULPHURYL FLUORIDE IN STRUCTURES

Sulphuryl fluoride has been used under the trade name Vikane since the 1950s. The major use has been Drywood termite control in the southern costal regions of the USA and has been widely used in the USA in beetle fumigations of art objects and other valuable materials (Schneider, 1993). It has been used against wood boring insects in Europe for more than 10 years (Binker, 1993).

The fumigant is supplied in cylinders as a pressurised liquid. Dosing of sealed structures is done through polythene tubes (shooting lines) directly from the cylinders with no need for pressurisation of the cylinder or vaporisation of the gas (Drinkall *et al*. 2003).

There have been a number of studies that have shown SF to be an effective replacement for MB in flour mills (Bell *et al*., 2004; Reichmuth *et al*., 2003 and Drinkall *et al*. 2003). In the United Kingdom heating is required to keep the temperature to 25^{o}C to improve efficacy in order to make a 24 hour treatment possible (Bell *et al*., 2004).

5. TOXICITY OF SULPHURYL FLUORIDE TO INSECTS

The majority of publications describe the efficacy of SF concentrate on the response of termites under laboratory or field test conditions. Most of the remainder deal with furniture and timber pests. Information exists for a very wide range of pest organisms including ticks (Roth, 1973), mites (Vaivanijkul and Haramoto, 1969), snails (Roth and Kennedy, 1973) and oak wilt fungus (Woodward and Schmidt, 1995).

Studies on different insect species agree that the egg is the most difficult stage to kill (Kenaga, 1957; 1961; Stewart, 1957; Outram, 1967a; Su *et al.*, 1989; Su and Scheffrahn, 1990; Williams and Sprenkel, 1990; Drinkall *et al.*, 1996; Bell *et al*. 2003). Bell *et al*. gave the concentration-time products (CTPs) necessary for 100% control of 10 pests of flour. In all cases the egg was the most tolerant stage with the exception of the flour mite (*Acarus siro*) where the mobile stages of mite proved of equal tolerance to eggs.

Outram (1967a) examined the factors affecting the tolerance of eggs of desert locust (*Schistocerca gregaria*) and mealworm (*Tenebrio molitor*) to SF. The amount of SF taken up by eggs exposed to 11 g m^{-3} for up to 64 hr was determined. Susceptible eggs took up and retained more fumigant per unit time than resistant eggs. Sulphuryl fluoride took more than 24 hours to penetrate to the interior of eggs showing higher levels of tolerance. Further studies identified the chorion (the outer layer of the egg) as a site of major uptake of gas and the probable agent of protection for the developing embryo (Outram, 1967b). Meikle *et al.* (1963) argued that inorganic fluoride is the primary poison. Death is caused by lack of available energy due to fluoride inhibiting glycolysis and the fatty acid metabolism.

Temperature has a striking effect on the efficacy of SF. Kenaga (1957) found that for the confused flour beetle (*Tribolium confusum*) the LD95 at 4°C was more than double that at 27°C. Bell *et al*. (1999) found that 1-2 day old eggs of Mediterranean flour moth (*Ephestia kuehniella*) were controlled by a CTP of 3000 g h m^{-3} at 15°C but required only 764 g h m^{-3} at 25°C, less than a third of the dose required at the higher temperature. They also found that the tolerance of *Tribolium castaneum* and *Trogoderma variable* at 30°C was only half that at 25°C.

There have been many studies on the efficacy of SF to storage pests. Table 1 gives a summary of the best of the available data from Drinkall *et al*. (1996), Reichmuth *et al*. (1999), Schnider and Heartsell (1999) and Bell *et al*. (2003).

Table 1. Concentration-Time Products (CTPs) giving 100% kill and Maximum CTPs where survival occurred in four studies on the efficacy of sulphuryl fluoride against storage pests.

Species	Max. CTP survived ($g\ h\ m^{-3}$)	CTP giving 100% kill ($g\ h\ m^{-3}$)	Temperature (^oC)	Reference
Flour mite (*Acarus siro*)	669		25	Bell *et al*., 2005
Turkish grain beetle (*Cryptolestes turcicus*)	470	784	25	Bell *et al*., 2005
Mediterranean flour moth (*Ephestia kuehniella*)	672	912	25	Bell *et al*., 2005
Mediterranean flour moth (*Ephestia kuehniella*)	960	1440	20	Reichmuth *et al*., 1999
Broad-horned flour beetle (*Gnatocerus cornutus*)	560		25	Bell *et al*., 2005
Booklouse (*Liposcelis bostrychophila*)	667	1000	25	Bell *et al*., 2005
Saw-tooth grain beetle (*Oryzaephilus surinamensis*)	893	957	20	Drinkall *et al*., 1996
Indian meal moth (*Plodia interpunctella*)	854		25	Schnider and Heartsell, 1999
Indian meal moth (*Plodia interpunctella*)	434	446	20	Drinkall *et al*., 1996
Australian spider beetle (*Ptinus tectus*)	320		25	Bell *et al*., 2005
Granary weevil (*Sitophilus granarius*)	784	966	25	Bell *et al*., 2005
Granary weevil (*Sitophilus granarius*)	1272	1339	20	Drinkall *et al*., 1996
Drugstore beetle (*Stegobium paniceum*)		434	20	Drinkall *et al*., 1996

Table 1. Continued.

Species	Max. CTP survived ($g\ h\ m^{-3}$)	CTP giving 100% kill ($g\ h\ m^{-3}$)	Temperature (^oC)	Reference
Yellow mealworm beetle (*Tenebrio molitor*)	320		25	Bell *et al.*, 2005
Rust-red flour beetle (*Tribolium castaneum*)	1178	1669	25	Bell *et al.*, 2005
Rust-red flour beetle (*Tribolium castaneum*)	1157		25	Schneider and Heartsell, 1999
Confused flour beetle (*Tribolium confusum*)	605	672	25	Bell *et al.*, 2005
Confused flour beetle (*Tribolium confusum*)	792	941	25	Schneider and Heartsell, 1999
Confused flour beetle (*Tribolium confusum*)	1339	1908	20	Drinkall *et al.*, 1996
Large cabinet beetle (*Trogoderma inclusum*)		434	20	Drinkall *et al.*, 1996
Warehouse beetle (*Trogoderma variabile*)	1195		25	Schneider and Heartsell, 1999

7. SORPTION OF SULPHURYL FLUORIDE ONTO COMMODITIES

Sulphuryl fluoride is less strongly sorbed than many fumigants. Comparative studies with MB have indicated that on a range of substrates there is a hundred-fold less initial sorption with SF (Meikle and Stewart, 1962). Rayon, corn oil, feathers, wheat gluten and flour gave the highest levels of sorption, but most gas aired off within 24 hours. Gray (1960) makes a passing reference to SF being less strongly sorbed on wheat than MB. Bell *at al*. (2004) found that the sorption of SF on flour was less than 80 mg/kg (too low to be measured by their method of detection) while the sorption of MB was at least an order of magnitude higher, at 750 mg/kg. They found that the sorption of SF on feed wheat was less than 50 mg/kg (again too low to be measured) while the sorption of MB was 110 mg/kg. It would seem unlikely that sorption levels would be high enough to affect dosing strategies for structures containing cereals or cereal products.

In studies on the effect of SF on various materials common in dwellings, polystyrene proved the most potent source of SF desorption for a 40-day period following fumigation (Scheffrahn *et al.*, 1987). Other studies have concentrated on the effectiveness of plastic films in the prevention of gas sorption in various foods (Osbrink *et al.*, 1988; Scheffrahn *et al.*, 1990; 1992a; 1994). The principal finding of these studies was that enclosure in nylon bags can virtually eliminate SF sorption in a wide range of food products. Enclosure in polyethylene bags also affords considerable protection; a double layer of 2 mil thickness (0.051 mm) film reduced sorption by over 96 % at 27^{o}C (Osbrink *et al*., 1988). In unprotected foodstuffs, even with a very high dosage rate of 360 mg/l for 20 hours, SF desorbed to less than 100 parts per billion (ppb) in less than 5 days in all commodities other than vegetable oil, which required 20 days (Osbrink *et al.*, 1988).

Apart from the penetration of the gas through films, there have been a number of investigations of the penetration of the gas into various materials including wood and flour. Basically SF is a very good penetrant, performing better than MB. In chamber tests SF diffused through a 28 cm column of sawdust without difficulty (Stewart, 1957); through 9 inches (23 cm) depths of flour, ground tobacco and dried milk powder (Kenaga, 1957); and through 2 inch (5 cm) thick Douglas fir blocks (Gray, 1960).

In a more recent study based on gas measurements rather than bioassay results (Scheffrahn *et al.*, 1992b), SF penetrated pine wood disks of 2.5 cm thickness within 20 hours. Diffusion through hard wood was much slower, and was further restricted if the wood was painted or hydrated by soaking. MB performed better only in the hydrated samples. Bell *et al*. (2004) showed that the speed at which SF penetrated densely packed wholemeal flour at 60% of the surface concentration was 10.5 cm/hour. In a similar test with MB the speed of the 60% concentration front was only 0.8 cm/hour. The large difference was explained by the higher level of sorption given by MB.

8. TAINT AND RESIDUES

Few studies have examined the effect of SF in tainting of foodstuffs, although fluoride and sulphate residues have been quantified. Early studies by Kenaga (1957) reported no odours from treated household items such as cork, rubber, wool, cotton, rayon, paper, leather and hides, and these findings have been quoted in subsequent trade or general articles and borne out by experience. Meikle and Stewart (1962) established that at least ten times the level of SF residue occurred on products such as cheese or flour as temperature was increased from 40 to 70^{o}F. Of the range of typical foodstuffs tested, the highest residue levels of SF 40 days after treatment, occurred in dried beef. Protein and fats were the principal sources of residues and reactions were relatively rapid. Typical reaction products predicted from the data, in the absence of definitive identification methods, were N-fluorosulfonyl derivatives and fluorides (Meikle, 1964; Dhaliwal, 1974).

Studies by Scheffrahn *et al*. (1989a and 1989b) evaluated the levels of fluoride and sulphate in treated foods. Some highly sorptive commodities such as vegetable oils gave rise to virtually no anionic residues while dried beef and dried milk gave rise to the highest fluoride and sulphate residues respectively. The residue level in many commodities did not alter between 1 and 15-days after aeration, indicating some stability of reaction products. Typical residue levels of fluoride after treatments at a level of 36 g m^{-3} for 20 hours (typical dosage for wood boring beetles) at about 25^{O}C were 200 parts per million (ppm) for dried beef, 100 ppm for dried milk and 60 ppm for flour.

The uptake of residue at low temperatures was examined to simulate the effect on freezer contents during building fumigations. At -20^{O}C, uptake of SF was much less and much lower fluoride residues were obtained than at 25^{O}C (Scheffrahn *et al.*, 1989b). After a 20 hour exposure at 36 mg/l, fluoride residues in dried beef and flour were 2.5 and 5.9 ppm respectively. Enclosure of products in plastic bags reduced residue levels to below the detectable limit of 0.8 ppm even when a ten times higher SF dosage was applied. As a comparison, a normal dietary intake of up to 8 ppm fluoride in water is considered safe (Dunning, 1965).

The formation of fluoride residues would present a potential problem for use in the malt industry and there is a need to determine the level of residues likely to be generated.

9. CORROSION

Kenaga (1957) lists stainless steel, brass, copper, aluminium, zinc and silver among materials showing no sign of corrosion or colour change after a 16 hour exposure at 3 lbs per 100 cu ft (48 mg/l) at 80^{O}F, and a review by Derrick *et al.* (1990) describes corrosive effects only of liquid SF on metals. Tests on museum materials have revealed that SF may degrade cellulosic and ligneous fibres (Burgess and Binnie, 1991) but successful treatments of historical buildings continue to be reported with no evidence of damage to precious art works or artefacts (e.g. Binker, 1993).

10. REGISTRATION

Sulphuryl fluoride has registration for use in flour mills in the United Kingdom under the trade name Profume®. There are current plans for the registration to be extended to cover the fumigation of wheat, rye and food manufacturing structures to improve the cost effectiveness of its use in flour mills. The registration is also to be extended to cover the fumigation of ground nuts.

There are no plans to extend the registration of Profume® to cover malt or barley. Unless the registration can be extended to cover these commodities, the use of SF in the malting industry would be limited to the fumigation of structures empty of barley and malt.

11. CONCLUSIONS

It is unfortunate that there are no plans to extend the registration of Profume® to cover malt or barley. Unless the manufacturers can be persuaded of a commercial advantage in extending the registration beyond their current plans, the use of SF in the malting industry would be limited to the fumigation of structures empty of barley and malt.

Numerous studies have shown SF to be effective against pests likely to be encountered in the malting industry and there have also been a number of studies that have shown SF to be an effective replacement for MB in flour mills. However, in the United Kingdom heating is required to keep the temperature to 25^{o}C to improve efficacy in order to make a 24 hour treatment possible.

There is no evidence that sorption of SF or corrosion due to SF gives any cause for concern. However, SF fumigations can give rise to fluoride and sulphate residues. There is a need to determine the level of residues likely to be generated on malt and barley.

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