Effectiveness of Sulfuryl Fluoride for Control of Different Life Stages of Stored-Product Psocids (Psocoptera)

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J. Econ. Entomol. 105(1): 282–287 (2012); DOI: http://dx.doi.org/10.1603/EC11209

ABSTRACT With the phase-out and impending ban of methyl bromide, sulfuryl fluoride is among the most promising alternative fumigant insecticides for control of stored-product insect pests. It has been evaluated for control of several stored-product insect pests, but there are few data available on its efficacy for control of stored-product psocids (Psocoptera). We evaluated sulfuryl fluoride for control of different life stages of the psocids *Liposcelis paeta* Pearman, *L. entomophila* (Enderlein), *L. bostrychophila* Badonnel, *L. decolor* Pearman, and *Lepinotus reticulatus* Enderlein (Trogiidae) in 48-hr trials at 27.5 °C. Adults and nymphs were susceptible to sulfuryl fluoride. Complete (100%) adult and nymphal mortality was recorded at concentrations between 4 and 8 g/m^3, except for *L. decolor* for which all adults were only killed at 24 g/m^3. Eggs were tolerant to sulfuryl fluoride. Complete egg mortality was achieved at 24 and 72 g/m^3 for *L. reticulatus* and *L. decolor*, respectively. Survival of *L. paeta* eggs was recorded even after exposure to 96 g/m^3. Given that the highest United States label concentration for sulfuryl fluoride for a 48-h exposure interval is 31.25 g/m^3, our study indicates that high doses and/or longer exposures are needed for complete mortality of eggs of *L. decolor* and *L. paeta*. Moreover, the present work suggests that there is considerable variation in efficacy of sulfuryl fluoride for control of different psocid species.

KEY WORDS sulfuryl fluoride, fumigation, Psocoptera, *Liposcelis*, *Lepinotus*

Completion of the phase-out of methyl bromide, which is scheduled for developing countries in 2015, is expected to create significant gaps in stored-product pest control because reliable alternatives are few and often case-specific (United Nations Environment Programme [UNEP] 1998). Therefore, there is an urgent need for further information on efficacy of these alternatives that show promise for use in a wide variety of facilities and commodities. Sulfuryl fluoride is the most commonly used alternative fumigant insecticide for methyl bromide for control of stored-product insect pests. Sulfuryl fluoride is an odorless, colorless, and generally nonreactive chemical with a desirably low boiling point (Reichmuth et al. 1997, Bell et al. 1999, Drinkall et al. 2003, Small 2007, Baltaci et al. 2009). In addition to use in stored-product protection, sulfuryl fluoride is a promising alternative for quarantine and preshipment treatments, household pests, and wood borers (Reichmuth et al. 1997, Barak et al. 2006, Buckley et al. 2010). Sulfuryl fluoride has certain advantages over the use of other fumigant insecticides in storage facilities. For example, in comparison with phosphine, which is also a possible alternative for some uses of methyl bromide, sulfuryl fluoride is nonflammable, noncorrosive, and it is generally effective at considerably lower exposure intervals. Moreover, the cost of fumigating using sulfuryl fluoride is, in many cases, comparable to that of methyl bromide (Adam et al. 2010).

Psocids (Psocoptera) are pests that damage grain and related amylaceous commodities. Psocids can develop in a large variety of cereals and cereal products (Opit and Throne 2008, Athanassiou et al. 2010a), and they are able to multiply rapidly in sound grain kernels (Opit and Throne 2008; Athanassiou et al. 2009, 2010a). Grain weight loss because of infestation by psocids can exceed 10% (Kučerová 2002). Psocids have a natural tolerance to several types of insecticides at doses that are lethal to other stored-product pests, such as beetles and moths. For example, Nayak et al. (1998) found that the organophosphorous insecticides chlorpyriphos-methyl and pirimiphos-methyl were unable to control *Liposcelis entomophila* (Enderlein) and *L. paeta* Pearman (Liposcelididae). In the same study, bioassays on field populations indicated the

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existence of natural tolerance and not a resistance that is related to previous exposure to these insecticides. Athanassiou et al. (2010b) reported that *Liposcelis bostrychophila* Badonnel, *L. decolor* (Pearman), *L. paeta*, *L. entomophila*, and *Lepinotus reticulatus* Enderlein (Trogiidae) were tolerant to the insect growth regulator methoprene, even at doses higher than the label rate in the United States. In another study, Athanassiou et al. (2009) found that the bacterial metabolite spinosad was not effective against *L. bostrychophila* and *L. paeta*. *L. bostrychophila* also was tolerant to the fumigant phosphine, especially at the egg stage (Nayak et al. 2003, Nayak and Collins 2008). However, there are few data available on the effectiveness of sulfuryl fluoride for control of stored-product psocids, despite the fact that this insecticide is expected to play an important role in the future, including its use in managing resistant insect populations. In the present work, we conducted laboratory bioassays to evaluate the efficacy of sulfuryl fluoride against different life stages of several stored-product psocid pests.

Materials and Methods

Insects. Eggs, nymphs, and adults of the psocid species *L. bostrychophila*, *L. decolor*, *L. entomophila*, *L. paeta*, and *L. reticulatus* were used in the tests. Psocids were reared on a mixture of 97% cracked wheat kernels, 2% Rice Krispies breakfast cereal, and 1% brewer's yeast at 30°C and 70% RH. We followed the procedure described by Opit and Throne (2008) to obtain individuals of standardized age for experimentation. Based on this technique, 30 female adults of unknown age of each species were left to oviposit for 3 d in 3.5-cm-diameter petri dishes containing 1 g of red-colored psocid diet. The females were then removed from the dishes, and eggs, nymphs, and progeny adults were taken later from these dishes for the bioassays. Hence, eggs that were used in the bioassays can be considered as 1–3 d old. Nymphs used were at the N1 and N2 stage; older nymphs were not included in the tests. Adults used were <21 d old.

Insecticide. Sulfuryl fluoride (Profume, containing ≈99% sulfuryl fluoride) was provided by Dow AgroSciences (Indianapolis, IN) in a cylinder and stored at ambient conditions. Small quantities of sulfuryl fluoride were removed from the cylinder before each series of bioassays and transferred to the laboratory as suggested by Scheffrahn et al. (1987).

Bioassays. Experiments were conducted at the Department of Entomology at Kansas State University. Glass jars (10.3-liter capacity) were used as the fumigation chambers. The jars were airtight and equipped with a port in the center of the metal screw-on cap that was fitted with a rubber injection septum that was used for the introduction and sampling of the fumigant. There were three jars for each tested sulfuryl fluoride concentration, which were used as replicates. The target concentrations evaluated were 0 (control), 1, 1.5, 2, 4, 6, 8, 10, 12, 24, 48, and 96 g/m^3. In all concentrations, the psocid species and life stages tested were *L. paeta* eggs, nymphs, and adults; *L. reticulatus* eggs, nymphs, and adults; *L. bostrychophila* adults; and *L. entomophila* nymphs and adults. Additional tests were carried out with *L. decolor* eggs and adults at concentrations of 0 (control), 2, 4, 6, 24, 48, 72, and 96 g/m^3. We tested all stages of *L. reticulatus* and *L. paeta* because these species were more and less susceptible, respectively, to insecticides than the other three psocid species (Athanassiou et al. 2010a).

The test insects were introduced into the fumigation chambers in small cylindrical glass vials (4.5 cm in height, 1.2 cm in diameter) before the introduction of the fumigant. The vials were placed with no lid in the jars, and the neck of the vial was covered with Fluon (polytetrafluoroethylene; Northern Products, Woonsocket, RI) to prevent psocids from escaping. There were 10 individuals (eggs, nymphs, or adults) in each vial. A small quantity of diet was added to each vial that contained eggs to avoid nymphal cannibalism after egg hatch. There were three vials within each jar for each species-life stage-concentration combination, and these vials were considered as subreplicates in the experimental design.

For the fumigation, the required quantity of sulfuryl fluoride (99+% pure) was calculated and introduced separately into each jar through the rubber septum by using a gas-tight syringe after removal of an equivalent volume of air from the jar using a syringe. Conditions inside the jars were 27.5°C and ≈70% RH. The jars were opened 48 h later, and all vials were removed. Mortality was assessed the same day in vials that contained adults or nymphs. Vials containing eggs were incubated for 8 d under the aforementioned conditions, and then they were examined for the presence of newly hatched nymphs. The gas concentration within each jar was measured just after the introduction of the fumigant and again before the termination of the 48-h exposure interval by quantitative Gas Chromatography/Mass Spectrometry (GC/MS) using the external standard curve method (Sekhon et al. 2010) to ensure that the average gas concentration did not deviate by >10% from the desired target concentration.

Data Analysis. Untreated control mortality was low (<10% for adults and nymphs and <20% for eggs). The data for each species and life stage were analyzed using a one-way analysis of variance (ANOVA) to determine the influence of gas concentration on mortality. Means were separated by the honestly significant difference (HSD) test at 0.05. We do not report the results of probit analyses on the data because estimates of LC values were generally poor because the slope of the mortality curve was so steep that there were generally only a few concentrations that could be included in the analyses, despite the large number of concentrations included in our experimental design.

Results

Adult Mortality. Mortality of *L. paeta* adults exceeded 74% at a concentration of 2 g/m^3, and all adults were killed at concentrations >4 g/m^3 (Fig. 1A). Sim-

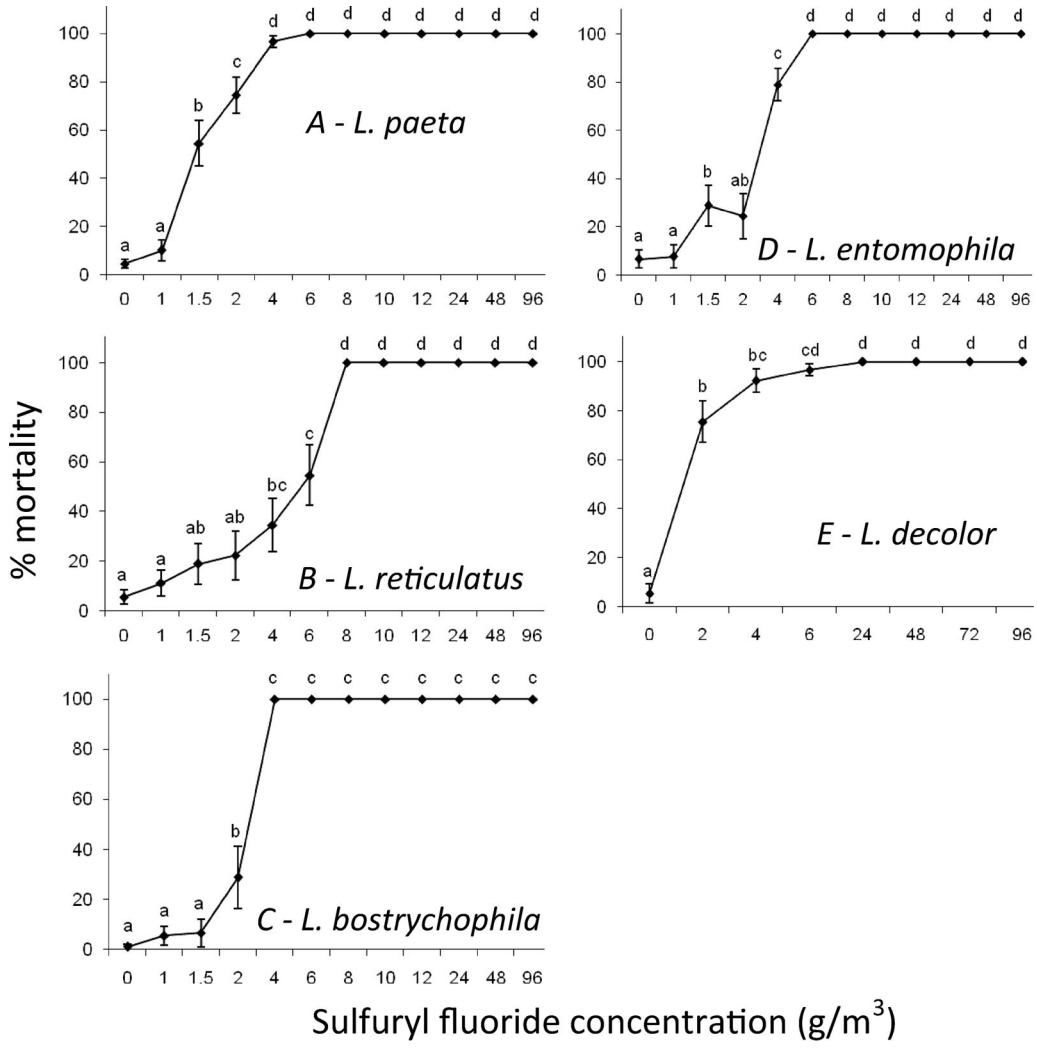

Fig. 1. Mortality (mean % ± SE) of adults of *L. paeta* (A), *L. reticulatus* (B), *L. bostrychophila* (C), *L. entomophila* (D), and *L. decolor* (E). Means for each species with the same letter are not significantly different (HSD test at 0.05; for *L. paeta* $F = 8.9$, for *L. reticulatus* $F = 44.2$, for *L. bostrychophila* $F = 80.6$, for *L. entomophila* $F = 118.9$, and for *L. decolor* df = 7,64, $F = 76.9$, $P < 0.001$; in all the other cases df = 11,96, $P < 0.001$).

ilar results were found for *L. bostrychophila* (Fig. 1C). Mortality of *L. reticulatus* and *L. entomophila* adults was 100% at 8 g/m^3 (Fig. 1B and D). Adults of *L. decolor* were the least susceptible among the species tested, and complete (100%) mortality was achieved only at concentrations of 24 g/m^3 or higher (Fig. 1E).

Nymphal Mortality. Generally, the results for nymphs were similar to those for adults. Mortality of *L. reticulatus* nymphs was 85% at 2 g/m^3, while all nymphs were killed at 4 g/m^3 (Fig. 2B). Similarly, >90% of *L. entomophila* and *L. paeta* nymphs died at 4 g/m^3, and mortality was 100% at 6 g/m^3 (Fig. 2A and C).

Egg Mortality. Eggs were by far less susceptible than adults or nymphs. Mortality of *L. paeta* eggs did not exceed 99% for any of the concentrations tested, and mortality did not exceed 27% at 24 g/m^3 or lower (Fig. 3A). Eggs of *L. reticulatus* were less tolerant, and complete mortality was recorded at concentrations ≥24 g/m^3 (Fig. 3B). Mortality at 12 g/m^3 was >92%. Mortality of *L. decolor* eggs was 100% at concentrations ≥72 g/m^3 (Fig. 3C), and mortality was negligible at ≤24 g/m^3.

Discussion

Our study clearly indicates that adults and nymphs of the species tested were susceptible to sulfuryl fluoride. In a recent field study with the red flour beetle, *Tribolium castaneum* (Herbst) (Coleoptera: Tenebrionidae), Hartzer et al. (2010) showed that young larvae, pupae, and adults were susceptible to sulfuryl fluoride, while large larvae were tolerant. Bell et al. (2003) reported that sulfuryl fluoride used at 25°C was

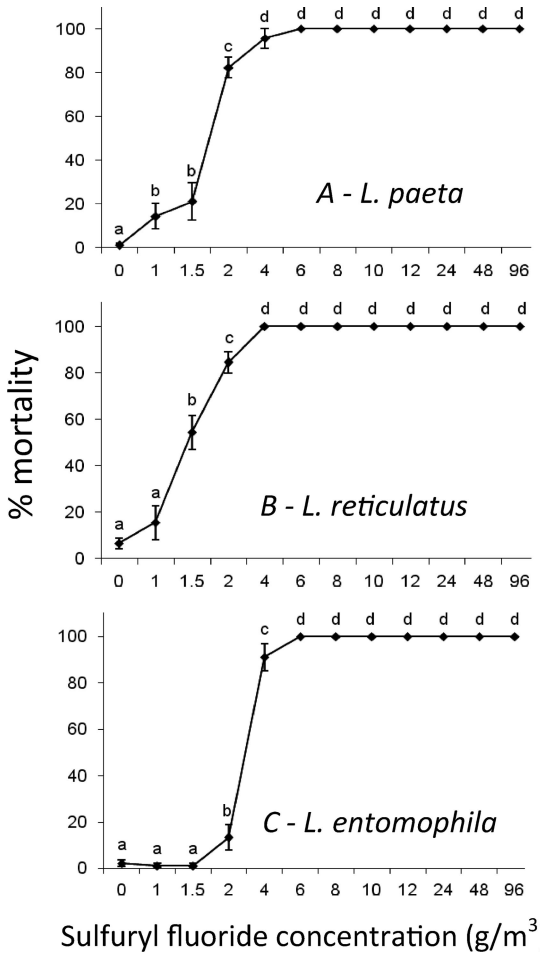

Fig. 2. Mortality (mean % ± SE) of nymphs of *L. paeta* (A), *L. reticulatus* (B), and *L. entomophila* (C). Means for each species with the same letter are not significantly different (HSD test at 0.05; for *L. paeta* $F = 125.5$, for *L. reticulatus* $F = 109.0$, for *L. entomophila* $F = 374.7$, in all cases df = 11,96, $P < 0.001$).

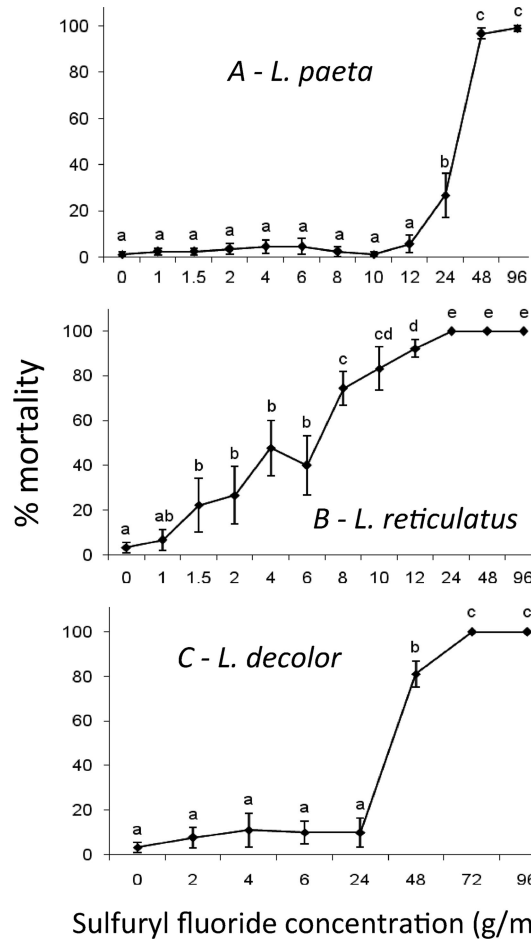

Fig. 3. Mean (%) mortality (±SE) of eggs of *L. paeta* (A), *L. reticulatus* (B), and *L. decolor* (C). Means for each species with the same letter are not significantly different (HSD test at 0.05; for *L. paeta* $F = 107.4$, for *L. reticulatus* $F = 20.7$, for *L. decolor* df = 7,64, $F = 83.3$, $P < 0.001$; in all the other cases df = 11,96, $P < 0.001$).

able to control adults, pupae, and larvae of the granary weevil, *Sitophilus granarius* (L.) (Coleoptera: Curculionidae), the Mediterranean flour moth, *Ephestia kuehniella* Zeller (Lepidoptera: Pyralidae), and *T. castaneum* at 42, 73, and 100 g-h/m^3, respectively, which were comparable to the lowest concentration used in our study (2 g/m^3 for 48 h). Similarly, Small (2007) found that sulfuryl fluoride was very effective in United Kingdom flour mills for control of the confused flour beetle, *Tribolium confusum* Jacquelin du Val, and *E. kuehniella*, while Tsai et al. (2006) reported similar levels of efficacy for mills in the United States for the Indianmeal moth, *Plodia interpunctella* (Hübner) (Lepidoptera: Pyralidae), and *T. castaneum.* In addition, Phillips et al. (2008) reported that complete (100%) mortality of adults and larvae of the red-legged beetle, *Necrobia rufipes* (DeGeer) (Coleoptera: Cleridae), occurred between 4 and 6 g/m^3. Baltaci et al. (2009) found that 100% mortality of *Ephestia elutella* (Hübner) (Lepidoptera: Pyralidae) larvae and pupae could be achieved at 11.6 g/m^3 after 18 h of exposure, regardless of the temperature. The U.S. Environmental Protection Agency (EPA) maximum label rate for sulfuryl fluoride in food storage and processing facilities is, in a concentration-time product scale (CTP), 1,500 g-h/m^3. Consequently, this maximum concentration corresponds to 31.25 g/m^3 for the 48-h exposure interval tested in our study. Based on these reports, 100% efficacy of nymphs/larvae and adults is achieved well below the EPA maximum concentration level during the 48-h exposure period for all of the insect species mentioned above. These gas levels are also effective for adults and nymphs of the species tested in the current study, because, with the exception of *L. decolor* adults, all mobile stages of psocids died at concentrations ranging between 4 and 8 g/m^3. However, Philips et al. (2008) found that sulfuryl fluoride, was by far less effective for adults and nymphs

of the mold mite, *Tyrophagus putrescentiae* (Schrank) (Astigmata: Acaridae), than the species mentioned above, given that 100% mortality occurred at 100.3 g/m^3, which is considerably higher than the maximum label rate of 1,500 CTP.

In contrast with adults and nymphs, sulfuryl fluoride was not very effective against psocid eggs. Based on our results, there are considerable variations among the species tested; for example, for *L. reticulatus*, 100% egg mortality occurred at 24 g/m^3, which is lower than the maximum CTP level. Bell et al. (2003) also reported that for 100% mortality of *L. bostrychophila* eggs, the concentration should be as high as 1,000 g-h/m^3, which is equivalent to 21 g/m^3 for a 48-h exposure interval. However, even the highest concentration of 96 g/m^3, which is approx. three times higher than the maximum CTP, was unable to provide complete control of *L. paeta* eggs in our study. Similarly, 100% mortality of *L. decolor* eggs was achieved only at 72 g/m^3. Taking into account the above data, we can conclude that the level of 31.25 g/m^3 is not effective for control of *L. paeta* and *L. decolor* eggs, and considerably higher concentrations, in conjunction with longer exposures, are necessary to achieve complete egg control. This also is likely to occur with other psocid species. Phillips et al. (2008) reported that *T. putrescentiae* eggs were extremely tolerant to sulfuryl fluoride, given that mortality did not exceed 95% at 100.3 g/m^3.

Reduced efficacy of sulfuryl fluoride for eggs is well documented in several studies (Bell and Savvidou 1999, Bell et al. 1999, Reichmuth et al. 2003, Baltaci et al. 2009). According to Outram (1967), this poor ovicidal effect could be attributed to the presence of the "protective" zone at the external egg part, where most of the fumigant is held by the embryonic membranes and the proteinaceous egg shell. Bell and Savvidou (1999) reported that high concentrations were needed to control *E. kuehniella* eggs, and that younger eggs (1–2 d-old) were more tolerant than older ones (3–4 d-old). Hartzer et al. (2010) indicated that methyl bromide was superior to sulfuryl fluoride for control of *T. castaneum* eggs, and egg survival could occur even at 1,300 g-h/m^3 of sulfuryl fluoride (27.1 g/m^3 in the case of 48 h of exposure). Similar results have been also reported by Tsai et al. (2006). Baltaci et al. (2009) found that there were variations in susceptibility to sulfuryl fluoride among eggs of *E. elutella* of different ages, but the overall data suggested that 21.3 g/m^3 is required for 100% control after 48 h of exposure at 15°C. Drinkall et al. (2003) also reported complete mortality of eggs of *T. castaneum*, *T. confusum*, *P. interpunctella*, and *E. kuehniella* in a flour mill at a mean sulfuryl fluoride concentration of 1,353 g-h/m^3, which is equivalent to 28.2 g/m^3 for 48 h.

All the above studies indicate that eggs of the major stored-product insect pests, such as beetles and moths, are remarkably less tolerant than the eggs of *L. paeta* and *L. decolor*, and have a similar level of susceptibility with the eggs of *L. reticulatus*. Hence, with the data available so far, psocids can be classified among the most tolerant stored-product pest species to sulfuryl fluoride during the egg stage. Consequently, this fumigant may be ineffective for control of psocids, at least under the combination of factors examined here. For the improvement of sulfuryl fluoride efficacy against psocid eggs, the effects of longer exposures should be tested for a wider range of psocid species. Also, given that higher temperatures increase the efficacy of sulfuryl fluoride (Baltaci et al. 2009), psocid egg mortality is likely to be higher at temperatures >27.5°C. However, at least for some species, sulfuryl fluoride may still be ineffective at the maximum label rate, and the application of contact insecticides may be preferable for a satisfactory level of control (Athanassiou et al. 2009).

Acknowledgments

We thank Ann Redmon and Ngunza Kisangani for technical assistance, and Dow AgroSciences for the sulfuryl fluoride sample. We also thank George Opit for his comments on an earlier version of this manuscript. Partial funding for this work came from the Kansas Agricultural Experiment Station and from a grant from the USDA program titled "Risk Avoidance and Mitigation" under agreement no. 2005-03824. USDA is an equal opportunity provider and employer.

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Received 29 June 2011; accepted 29 August 2011.