Ovicidal Activity of Sulfuryl Fluoride to Anobiid and Lyctid Beetle Eggs of Various Ages[1,2]

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J. Entomol. Sci. 25(3): 366-375 (July 1990)

ABSTRACT Fumigation with sulfuryl fluoride (Vikane® Gas Fumigant)[2] is often used to control structural infestations by anobiid and true powderpost beetles that are widespread or inaccessible to insecticidal surface treatments. Beetle eggs show a wide range of tolerance to sulfuryl fluoride that varies with the age of eggs making them the limiting factor for control. To study dosage requirements for eggs, ovicidal activity tests of sulfuryl fluoride were done by exposing 1- to 7-day-old anobiid, *Euvrilletta peltata* (Harris) and lyctid, *Lyctus brunneus* (Stephens), eggs respectively, during tent fumigations of a house. Fumigations resulting in mg-h/liter accumulations of 470 and 289, 5.2 and 3.2 times the drywood termite dosage of 90 mg-h/liter for 22.2°C, permitted some survival and subsequent hatching of lyctid eggs aged 1 and 2 days, with the latter being most tolerant. All other ages of eggs were susceptible to these dosages. At the 3.2-fold rate, the mean survival for eggs of all ages was 11.6%, but 70.2% of the 2-day-old eggs survived. At the 5.2-fold rate, only 3.9% of the eggs survived, primarily due to 24.7% survival of 2-day-old eggs. No differeences were observed between 289 and 470 mg-h/liter dosages for anobiid beetle eggs; the least susceptible apparently were eggs aged 2 to 4 days old. Additional studies are recommended to better define effective rates for controlling eggs of these beetles with reduced levels of gas. To aid logistics, such studies could be confined to the ovicidal activity of 6-fold and lower dosages on the most tolerant ages of eggs.

KEY WORDS fumigation, ovicides, *Euvrilletta peltata, Lyctus brunneus,* Vikane®, structural pest control.

Beetle species that commonly infest structural wood include the anobiid beetle, *Euvrilletta peltata* (Harris), (Coleoptera: Anobiidae), formerly *Xyletinus peltatus* (Harris) (White 1985), and several species of the true powderpost beetles, represented in this study by *Lyctus brunneus* (Stephens) (Coleoptera: Lyctidae) (Williams 1973a, 1980, 1985).

[1] Accepted for publication 27 March 1990.

[2] Mention of a company or trade name is for identification purposes only and does not imply endorsement by the U.S. Department of Agriculture.

This publication reports research involving pesticides. It does not contain recommendations for their use, nor does it imply that the uses discussed have been registered. All pesticides must be registered by appropriate State and/or Federal agencies before they can be used.

CAUTION: Pesticides can be injurious to humans, domestic animals, desirable plants, and fish or other wildlife, if they are not handled or applied properly. Use all pesticides selectively and carefully. Follow recommended practices for the disposal of surplus pesticides and pesticide containers.

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Anobiid beetles typically fly to and infest only pine among the softwood timbers used in crawl spaces of structures in the South (Williams and Smythe 1979) but may also infest hardwood timbers from broad-leaved trees. With time and sufficient wood moisture (Williams 1973b, 1983), infestations may spread upward into wood within structures.

Lyctid beetles only infest hardwoods. Infestations usually begin when wood is stored at processing or distribution points. These infestations often are introduced into structures with products such as cabinets, flooring, millwork, or picture framing.

Beetle infestations that are widespread or inaccessible to insecticidal surface treatments often are fumigated with methyl bromide or sulfuryl fluoride, the active ingredient of Vikane® Gas Fumigant produced by Dow Chemical Co., Midland, MI. Because methyl bromide is known to cause persistent odors in certain household products, sulfuryl fluoride often is used for fumigation of structures.

Beetle eggs exhibit a wide range of tolerance to sulfuryl fluoride (Kenaga 1957), making them the limiting factor when attempting control of an infestation. Eggs of *L. planicollis* LeConte that are midway through the incubation period (3 to 4 days old) are the most difficult to kill; newly laid eggs and those near eclosion are the most susceptible (Doty and Whitney 1967).

Because the dosage requirements for eggs are not clearly defined, the label-recommended dosage of sulfuryl fluoride for anobiid and lyctid eggs is 10 times the dosage used to fumigate drywood termites. This paper reports the results of ovicidal activity tests of sulfuryl fluoride on 1- to 7-day-old anobiid and lyctid eggs, *E. peltata* and *L. brunneus* respectively, during tent fumigations of a house.

Materials and Methods

Fumigation. The test building was a vacant three-bedroom house with a crawl space and partial basement on the Harrison Experimental Forest of the Southern Forest Experiment Station located 48.3 km north of Gulfport, Mississippi. The building was covered with 6-mil clear polyethylene plastic, and all seams were sealed with clamps and duct tape. After soaking the perimeter soil with water, a ground seal (cover-to-ground junture) was created by placing the rolled ends of the tent in a trench and covering the plastic with wet sand continuously around the structure.

Gas was introduced into the basement and kitchen hallway and rapidly distributed throughout the structure with the use of fans (Fig. 1). Fans were also used to aid gas aeration following exposure and a Miran 101V® (Foxboro Company, South Norwalk, CT) was used to verify sufficient degassing of the structure. The cover remained in place for four successive fumigations, and was opened only for aeration and removal and replacement of beetle egg test units for each test.

A "slide rule" calculator developed by the fumigant manufacturer, FUMIGUIDE® B, was used to estimate how well the structure confined the gas based on the following conditions: cover condition — excellent, seal condition — excellent, wind — 0 to 8.1 km/h, volume — 708 m^3, underseal — sandy loam, soil temperature — 22.2°C. This dosage calculation procedure is commonly used to compensate for field variations when: instruments are not used to monitor and confirm the half-loss time (HLT) and no consideration is given to laboratory determined dosages for drywood termite mortality based on time and temperature relationships (Table 1) (Stewart 1962).

Table 1. Comparison of estimated concentrations and mg-h/liter of sulfuryl fluoride required for 100 percent mortality of drywood termites at 23.9° C (75° F), 24 hours exposure, and no gas loss*.

Concentration	Laboratory †	Fumiguide® Y ‡	Fumiguide® B §	16 mg/liter
mg/liter	2.2	3.4	5.0	16.0
mg-h/liter	54.0	81.0	120.0	384.0

* By coincidence, mg/liter and mg-h/liter are equivalent to ounces/1,000 ft^3 and ounce hours.

† From Stewart 1962.

‡ Ounce-hour scale, side A of Fumiguide® Y "slide rule" calculator, ounces per 1,000 ft^3 - 81 ounce hours for 24 hours.

§ Side B of Fumiguide® B "sliderule" calculator, ounce hours = 5.0 × 24 hours.

Because exposure time apparently is equally as important as concentration (Su et al. 1989), the actual dosages for these tests were recalculated based on the known HLT, the initial concentration introduced, and the hours of exposure (Table 2). These recalculations of accumulated milligrams/hour/liter (mg-h/liter) (ounce-hours) to obtain 100% mortality at the existing temperature allowed dosage comparisons with the drywood termite dosage of 90 mg-h/liter (coincidentally equivalent to oz./1,000 ft^3) for 22.2° C from the FUMIGUIDE® Y, another manufacturer-developed "slide rule" calculator for use when HLT is measured. The label-recommended powderpost beetle dosage for this temperature is 900 mg-h/liter (10-fold).

Table 2. Comparisons of estimated and actual conditions, rates, and corresponding drywood termite (DWT) factors for four different tent fumigation tests.

	Test 1	Test 2	Test 3	Test 4
Hours exposure	18	16	18	6.5
Estimated HLT	5.4	5.4	5.4	5.4
Estimated DWT dosage in kg (lbs) (Fumiguide Y®)	8.84 (19.5)	9.07 (20.0)	8.84 (19.5)	15.0 (33.0)
Actual sulfuryl fluoride introduced in kg (lbs)	30.8 (68)	61.7 (136)	92.5 (204)	46.3 (102)
Estimated DWT factor	3.5X	6.8X	10.5X	3.1X
Actual HLT	>72	>72	46	12.5
Calculated DWT mg/liter required based on conditions	5.4	6.4	5.6	16.5
Mean actual intial mg/liter	28.2	74.7	118.0	52.7
Actual DWT factor (Fumiguide Y®)	5.2X	12.4X	21.1X	3.2X
Required mg-h/liter for DWT (22.2°C (72°F) Fumiguide Y®)	90	90	90	90
Actual mg-h/liter accumulated	470	1,120	1,900	289
Actual DWT factor (Fumiguide Y®)	5.2X	12.4X	21.1X	3.2X

Monitoring. Concentrations within the structure were monitored 4 to 7 times during each fumigation with a Fumiscope Model E-V® (Robert K. Hassler Co., Mammoth Lakes, CA) at the seven exposure sites for beetle eggs. For concentrations in the structure that exceeded the unit scale of the Fumiscope®, an appropriate quantity of gas was removed through the monitoring line with a 1-liter syringe. This sample then was diluted with a known volume of fresh air and injected into the Fumiscope®. For example, when 500 ml of fumigant were pulled into the syringe from the structure, an additional 500 ml of fresh air were mixed to bring the volume to 1 liter. After injecting into the Fumiscope®, the resulting reading was doubled to obtain the actual concentration in the structure.

Beetle egg test units. Because *E. peltata* beetles generally oviposit in cracks and crevices on wood surfaces and *L. brunneus* beetles generally insert their eggs into pores of wood, different types of test units were devised for each beetle species.

For *E. peltata,* sanded yellow-poplar (*Liriodendron tulipifera* L.) sapwood blocks (12.7 by 51 by 76 mm) were nutrient treated (Berry 1972) and dried. Approximately 125 egg-laying sites were impressed on each tangential surface of each block with a 44.5-mm diameter metal cut-flower holder. A plastic spacer ring exposed only the points of the metal tines. From 2 to 10 laboratory-reared adults were confined over the egg-laying sites in an inverted hollow polyethylene stopper held in place with a rubber band. A 32-mesh copper screen fused in place of the stopper bottom allowed air movement into the unit.

For *L. brunneus,* yellow-poplar wood wafers (3.2 mm thick and the same size as microscope slides), containing pores too small for insertion of eggs, were soaked in 3.0% starch solution to make them attractive to egg-laying females and then dried. Two starch-soaked wafers were sealed between two glass microscope slides by dipping each end of the "sandwich" in melted paraffin. Individual test units were confined with 5 to 40 adults in Pyrex® storage jars containing a layer of plaster of paris on the bottom as a foothold for crawling beetles.

Anobiid and lyctid beetles were placed on new test units every 24 hours until sufficient test units with eggs aged 1 to 7 days old were obtained for each of four fumigations. After 24 hours exposure, the beetles were removed and units with eggs were marked with the day's date as "0" day. Each age, day 1 to day 7, represents 48 hours; therefore, day 1 is 1- to 2-day-old eggs, day 2 is 2- to 3-day-old eggs, etc.

Anobiid eggs were laid in the prepared sites on the wood surface and all of them could be counted. Some lyctid eggs were visible underneath glass, while others were hidden between the wafers of wood. Because the total number of lyctid eggs could not be determined before testing, two units with **any** eggs visible underneath the glass were fumigated for each age of eggs in all tests. Varying numbers of eggs per unit were tested because equal numbers for seven ages of eggs from two beetle species for four tests within 1 week were logistically impossible.

Egg exposure procedures. One test unit of each age, day 1 to day 7, of anobiid eggs was exposed at each of six locations while two units with each age of lyctid eggs were exposed at each of three locations (Al-A6 and L1-L3 respctively, Fig. 1). Controls consisted of three units of every age for both species that were brought to the fumigation site and stored in a nearby, untarped building. Before and after fumigation, all egg test units and comparable controls were stored at

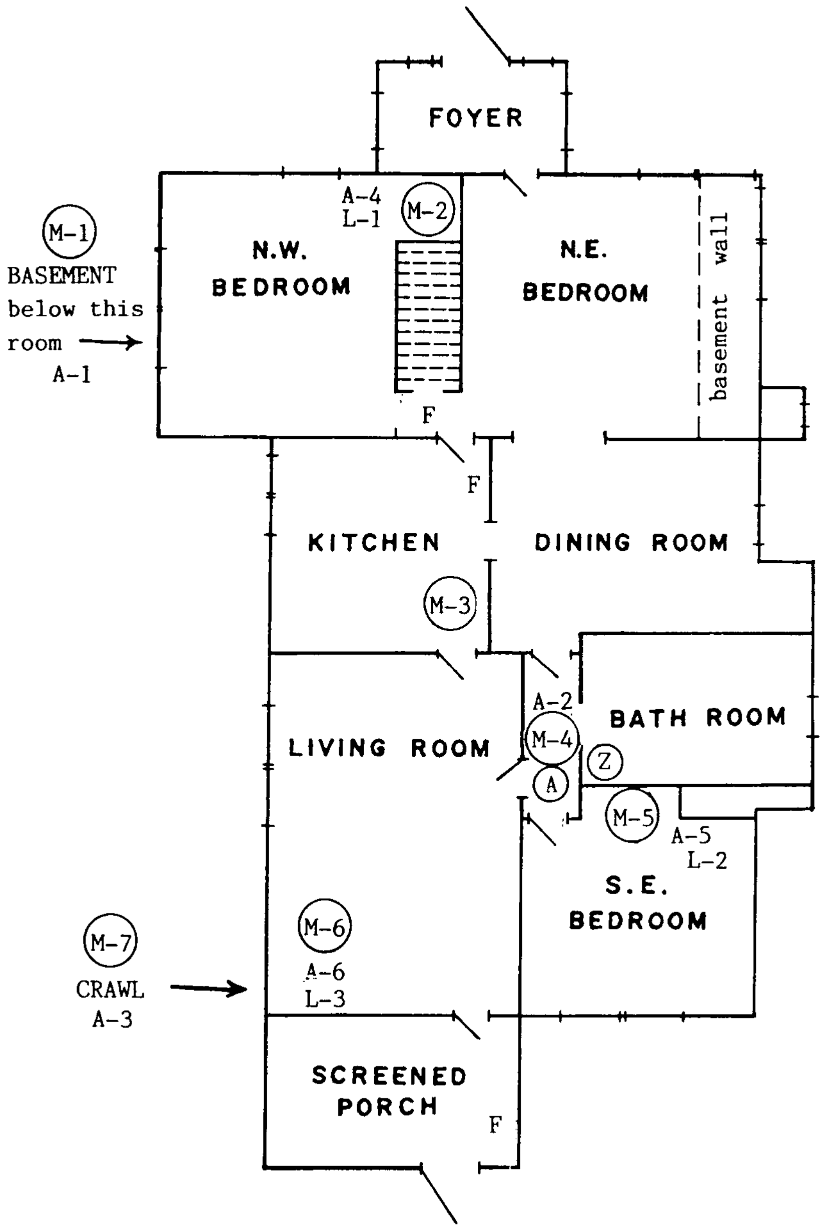

Fig. 1. Floor plan (scale 0.5 cm = 1 ft) of the test house with letter - numeral notations indicating the location of the following items: A = attic entrance, A-1 through A-6 exposure sites of *Euvrilletta peltata* (Harris) eggs, L-1 through L-3 = exposure sites of *Lyctus brunneus* (Stephens) eggs, F = gas distribution or exhaust fans, M-1 through M-7 = monitoring tubes leading to Fumiscope®, and Z = maximum-minimum thermometer.

25 ± 2.0° C and 60 to 70% RH until mortality of eggs (failure to hatch or develop embryo) was determined.

Egg mortality determinations. Lyctid test units were examined 10 days after eggs were laid because hatching should occur within 7 to 10 days in the described storage conditions (Rosel 1969). Eggs beneath the glass were examined first with a microscope. Then the paraffin-sealed ends were separated so eggs on the inner surfaces of each wood wafer could be examined. Mortality of control eggs was determined by counting eclosed larvae. Because many eggs contained developing embryos that may have survived, this evaluation procedure provided a conservative estimate for comparison with survival in treated test units. Survival of larvae could not be determined because the test units were destroyed as they were examined.

First-instar anobiid larvae bored into the wood directly beneath the egg without moving the shell; thus, egg shells were ruptured carefully with a needle to determine if fumigation arrested development of the embryo. Developing embryos may have been injured by rupturing the egg shell, particularly when eggs were deep within cracks; therefore, two evaluation procedures were used for further confirmation of mortality. Contents of half of the eggs on each test block were examined after 14 days for developing live larvae by rupturing egg shells. Blocks bearing the remaining eggs were stored for 10 months at 25 ± 2.0°C and 60 to 70% RH, then x-rayed to count the developing larvae within wood.

Results and Discussion

Because half-loss times were longer than expected, two of the fumigations, Tests 2 and 3, exceeded the label rate of 900 mg-h/liter, with actual accumulated dosages of 1,120 and 1,900 mg-h/liter as calculated in Table 2. These accumulated dosages were 12.4 and 21.1-fold the recommended drywood termite dosage of 90 mg-h/liter. Both Tests 2 and 3 produced 100% mortality, are not further discussed, and the data are not included.

The initial concentration for Test 1 with 18 hours exposure was 28.2 mg/liter. For Test 4 with 6.5 hours exposure, the concentration was 52.7 mg/liter. Based on the respective half-loss times of 72 and 12.5 hours, the mg-h/liter accumulations for Tests 1 and 4 were 470 and 289, respectively, which are 5.2 and 3.2-fold the drywood termite dosage.

Lyctid beetle eggs. Effects of fumigating a total of 1,568 *L. brunneus* eggs with accumulated concentrations equivalent to 3.2 and 5.2-fold the dosage required for fumigation of drywood termites are shown in Table 3. In both tests, there was some survival and subsequent hatching of eggs aged 1 and 2 days, with the latter being most tolerant. All other ages of eggs were susceptible to these dosages. At the 3.2-fold rate, the mean survival for eggs of all ages (625 fumigated eggs) was 11.6%, but 70.2% of the day 2 eggs survived. This survival was better than for similar-aged control eggs where survival was based only on eclosed larvae. At the 5.2-fold rate, only 3.9% of 943 fumigated eggs of all ages survived, primarily due to 24.7% of the day 2 eggs surviving. This survival rate was about a third of the rate for similar-aged eggs in the control, suggesting that an effective accumulated dosage rate was being approached in this test. Surviving day 2 eggs could have been 49 to 72 hours

Table 3. Mean percent survival of 1- to 7-day-old *Lyctus brunneus* (Stephens) eggs following tent fumigations with sulfuryl fluoride.

mg-h per liter	Drywood termite factor	Mean percent survival of eggs aged 1 to 7 days*							
		7	6	5	4	3	2	1	all ages†
289	3.2X	0.0	0.0	0.0	0.0	0.0	70.2	11.1	11.6
Control	0.0X	57.5	61.3	61.1	40.9	88.4	31.9	82.9	53.4
470	5.2X	0.0	0.0	0.0	0.0	0.0	24.7	2.3	3.9
Control	0.0X	41.2	96.1	32.8	57.5	61.3	61.1	40.9	55.8

* Data for all treated eggs are means of three replicates (two test units at each of three exposure sites). Data for each age of control eggs are means for three test units for each day that eggs were laid.
‡ Total number of eggs for 3.2X rate = 625, control = 394; for 5.2X rate = 943, control = 591. Percentages of hatch were based on examination at 10 days after eggs were laid. Survival for control eggs includes only eclosed larvae, many eggs contained developing embryos that may have hatched later.

old when fumigated because eggs were designated as "0" day when adult beetles were removed after a 24-hour egg-laying period.

Apparently, 1- to 2-day-old (probably included some 2- to 3-day-old) eggs were the least susceptible to sulfuryl fluoride in these comparisons, whereas Doty and Whitney (1967) reported 3- to 4-day-old *L. planicollis* eggs (actually same as 2- to 3-day-old eggs in this study) as being the most tolerant. Probable reasons for observed tolerances to the gas during early to mid development of eggs are: (1) the egg shell is impervious to the gas (Outram 1967) and higher concentrations are required for sufficient diffusion of a toxic concentration; and (2) the embryo has not yet formed (Rosel 1969) which reduces the inhibitory action of the fluorine portion of the gas on metabolism, primarily of proteins (Meikle et al. 1963).

Anobiid beetle eggs. Unlike results observed by lyctid eggs, only a few *E. peltata* eggs survived exposure to the 289 mg-h/liter and 470 mg-h/liter dosages (3.2 and 5.2-fold, respectively). Data obtained after rupturing a total of 1,103 fumigated egg shells suggested that some eggs aged 2 to 7 days old survived both rates, with the least susceptible being those eggs aged 2 to 4 days old (Table 4). However, no larvae were detected from 1,154 eggs by x-rays of treated blocks 10 months after eggs were laid for either treatment (Table 5). Unfortunately, poor survival of larvae occurred for the x-rayed control blocks, making conclusions difficult.

Table 4. Mean percent survival of 1- to 7-day-old *Euvrilletta peltata* (Harris) eggs following tent fumigations with sulfuryl flouride as determined by the rupture method.*

mg-h per liter	Drywood termite factor	Mean percent survival of eggs aged 1 to 7 days							
		7	6	5	4	3	2	1	all ages†
289	3.2X	6.0	0.0	7.6	15.0	8.7	7.4	0.0	6.4
Control	0.0X	100.0	100.0	-	-	-	100.0	—	100.0
470	5.2X	0.0	0.0	0.0	15.2	31.7	15.8	0.0	9.0
Control	0.0X	57.9	95.8	65.0	100.0	100.0	-	-	83.7

* Egg shells were ruptured 14 days after eggs were laid to count degenerating or developing embryos. Means for treated eggs are based on half of the eggs exposed at each of six sites. Means for controls are based on half of the eggs on three replicates for each day that eggs were laid; dashes indicate missing data.
† Total number of eggs for 3.2X rate = 613, control = 148; for 5.2X rate = 490, control = 204.

Table 5. Mean percent survival of 1- to 7-day-old *Euvrilletta peltata* (Harris) eggs following tent fumigations with sulfuryl fluoride as determined by x-ray examination for developing larvae 10 months later.

mg-h per liter	Drywood termite factor	Mean percent survival of eggs aged 1 to 7 days*							
		7	6	5	4	3	2	1	all ages†
289	3.2X	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Control	0.0X	20.0	88.9	50.0	53.3	50.0	62.9	54.2	54.2
470	5.2X	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Control	0.0X	—	—	33.3	20.0	88.9	50.0	43.3	49.1

* Means for treated eggs are based on half of the eggs exposed at each of six sites. Means for control eggs are based on half of the eggs on three replicates for each day that eggs were laid; dashes indicate missing data.

† Total number of eggs for 3.2X rate = 628, control = 304; for 5.2X rate = 526, control = 180.

Possible reasons for the discrepancy between visual and x-ray evaluations include: (1) humidity during storage was too low for development of larvae; (2) larvae were growing slowly and had not reached sufficient size to be detected by x-ray analysis; (3) embryos were incorrectly assessed as being alive and developing when egg shells were ruptured; and (4) death occurred after examination due to delayed effects of the fumigant. The first two reasons may offer the best explanation because the mean survival of eggs as 10-month-old larvae was only 54.2% and 49.1% for Test 1 and Test 4 controls, respectively.

Conclusions

The results of this study suggest that eggs of *L. brunneus* beetles that have been laid 48 to 72 hours before fumigation and eggs of *E. peltata* that have been laid <4 days before fumigation are the respective ages most tolerant to sulfuryl fluoride. This difference in ages of tolerant anobiid and lyctid eggs is probably due to the longer time required for formation of embryos in anobiid eggs.

Although no definitive rate was determined, results from these test fumigations indicate that the label rate of 10 times the drywood termite dosage may be excessive for lyctid and anobiid eggs, particularly the latter. X-rays indicated no anobiid larvae survived the 3.2 and 5.2-fold accumulated drywood termite dosages, but examinations of embryos soon after fumigation suggested that some survival of larvae was possible. Poor larval survival for anobiid controls prohibited a definitive conclusion. Some 2-day-old and younger lyctid eggs survived the less than label-recommended treatment rates (289 and 470 vs 900 mg-h/liter) while 3- to 7-day-old eggs did not. For this reason, higher accumulated concentrations would be needed for total mortality; but, it is doubtful that the requirement is as high as 10 times the drywood termite rate of 90 mg-h/liter.

Control of most eggs of both species with a 6.5-hour exposure at the lower rate (289 mg-h/liter) suggests that the toxic activity of sulfuryl fluoride might occur quickly for beetles, but the time component was as important as concentration for at least one termite species (Su et al. 1989). The quick action of sulfuryl fluoride appears particularly true for anobiid eggs based on results obtained by rupturing egg shells. Additional studies are warranted to better identify the effective rates for controlling various ages of eggs of common wood-infesting beetle species with reduced levels of gas. For logistical purposes, such studies could be confined to the ovicidal activity of 6-fold and lower dosages on the most tolerant ages of eggs.

Because anobiid beetles produce only one generation per year, there is an alternative strategy to using the high rates necessary to get complete kill of the more tolerant ages of eggs that may represent a small percentage of the total population. Fumigations could be done before or after the period when anobiid adults are likely to be emerging and laying eggs (before April and after August in the southern United States). The drywood termite dosage could be used because eggs would be absent, and only susceptible life stages would be present.

During times of unknown anobiid beetle development or for lyctid beetle infestations when newly laid eggs might be present, high gas rates also might be avoided, depending upon the accessibility of infestations, by following the fumigation with insecticidal treatments on all exposed surfaces. These treatments may ultimately kill the emerging adults or feeding larvae resulting from eggs surviving fumigation.

Acknowledgments

We thank Redd Pest Control, Inc. of Gulfport, MS, for assistance in tenting the structure used in this study. We thank Drs. Jeffery P. LaFage, Harry B. Moore, and Nan-Yao Su for helpful reviews of an earlier draft of this manuscript.

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