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Author(s): Alan V. Barak, Matthew Messenger, Paul Neese, Ellen Thoms, and Ivich Fraser

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Sulfuryl Fluoride as a Quarantine Treatment for Emerald Ash Borer (Coleoptera: Buprestidae) in Ash Logs

ALAN V. BARAK,[1] MATTHEW MESSENGER,[2] PAUL NEESE,[3]
ELLEN THOMS,[4] AND IVICH FRASER[5]

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ABSTRACT *Fraxinus* spp. logs infested with *Agrilus planipennis* Fairmaire (Coleoptera: Buprestidae) were fumigated with sulfuryl fluoride (SF) in 432-liter chambers at 15.6 and 21.1°C for 24 and 48 h. Concentration × time (CxT) exposures (g-h/m^3) of SF obtained were 3,382 (24-h exposure) and 5,466 (48-h exposure) at 15.6°C and 3,329 (24 h) and 4,385 (48-h exposure) at 21.1°C after doses of 144, 128, 128, and 104 g/m^3, respectively. After aeration, logs were placed in modified fiber drums for 8 wk to capture emerging beetles. No adults emerged from any of the fumigated logs, whereas 933 adults emerged from control logs. Eggs were fumigated at CxT exposures similar to log fumigations (3,240 and 4,262 g-h/m^3, respectively) and again at doses 16 g/m^3 lower, at 21.1°C for 24 and 48 h. No hatch was observed at CxT dosages >4,262 g-h/m^3. No larvae continued development on artificial diet after hatching from eggs fumigated at all tested dosages, whereas 10 control larvae developed to instar I or II. Chamber fumigations with 31 and 46% load factors provided additional sorption and concentration data. *A. planipennis*-infested logs in tarped, 149.1-m^3 cargo containers were fumigated at dosages used in successful trials. Logs were monitored for 8 wk for adult emergence. There was no adult emergence, but 621 adults emerged from a similar quantity of control logs. CxT dosages of SF for 100% control of *A. planipennis* at 15.6 and 21.1°C for 24- and 48-h exposure can be obtained under commercial fumigation conditions. A quarantine treatment schedule for SF is proposed.

KEY WORDS *Agrilus*, *Fraxinus*, fumigation, quarantine, sulfuryl fluoride

As of 1994, 368 insect species that damage trees and shrubs have been introduced and have become established into United States forests, primarily as a result of containerized world trade (Haack and Byler 1993, Mattson et al. 1994). More recently, emerald ash borer, *Agrilus planipennis* Fairmaire (Coleoptera: Buprestidae) (Fig. 1A), has become established in the lower Great Lakes Region (Haack et al. 2002) and poses a serious threat to North American ash (*Fraxinus* spp.) trees (Poland and McCullough 2006), on which it feeds exclusively (Cappaert et al. 2005). *A. planipennis* was discovered in southeastern Michigan in summer 2002 and probably arrived in the United States on solid wood packing material originating in its native Asia. Since then, *A. planipennis* was found in Ohio, Indiana, Illinois, Maryland, Pennsylvania, West Virginia, Virginia, Wisconsin, Missouri, New York, Minnesota, and Kentucky and also in Ontario, and Quebec, Canada (http://pest.ceris.purdue.edu/searchpest.php?selectName=INAHQJA).

Merchantable ash logs are processed into diverse products, including tool handles, baseball bats, furniture and cabinets, flooring, mill work, railroad ties, and cargo crates. The value of ash timber grown in the eastern United States for these uses is estimated at US$25.1 billion (USDA–APHIS 2003). *A. planipennis* can survive and emerge from wood cut from infested trees for up to 2 yr after these trees are cut (Petrice and Haack 2006). However, this wood was exposed to local populations of *A. planipennis*, so reinfestation of cut logs was possible. Therefore, transport of all ash logs from infested counties to uninfested areas is regulated by the current federal quarantine (USDA–APHIS 2003) (www.aphis.usda.gov/plant_health/plant_pest_info/emerald_ash_b/quarantine.shtml). The currently accepted treatment method of ash logs to ensure mortality of *A. planipennis* life stages is to grind wood into 2.54-cm (1-in.) or smaller pieces (USDA–APHIS 2003, McCullough et al. 2007) heat treat (Plant Protection Quarantine [PPQ] Schedule T-314-a) or removing bark to a depth of 1.27 cm (0.5 in.). The USDA–Animal and Plant Health Inspection Service (APHIS)–PPQ is working to find quarantine

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[1] Corresponding author: USDA–APHIS–PPQ, CPHST Otis Laboratory, 1398 West Truck Rd., Otis ANGB, Buzzards Bay, MA 02542-1329 (e-mail: al.barak@aphis.usda.gov).

[2] USDA–APHIS–VS, Riverdale, MD (formerly Dow AgroSciences LLC, Indianapolis, IN).

[3] BASF Corp., 26 Davis Dr., Research Triangle Park, NC 27709 (formerly Dow AgroSciences LLC, Indianapolis, IN).

[4] Dow AgroSciences LLC, 7257 NW 4th Blvd., Gainesville, FL 32607.

[5] USDA–APHIS–PPQ, 5963 Ford Court, Suite 200, Brighton, MI 48116-8511.

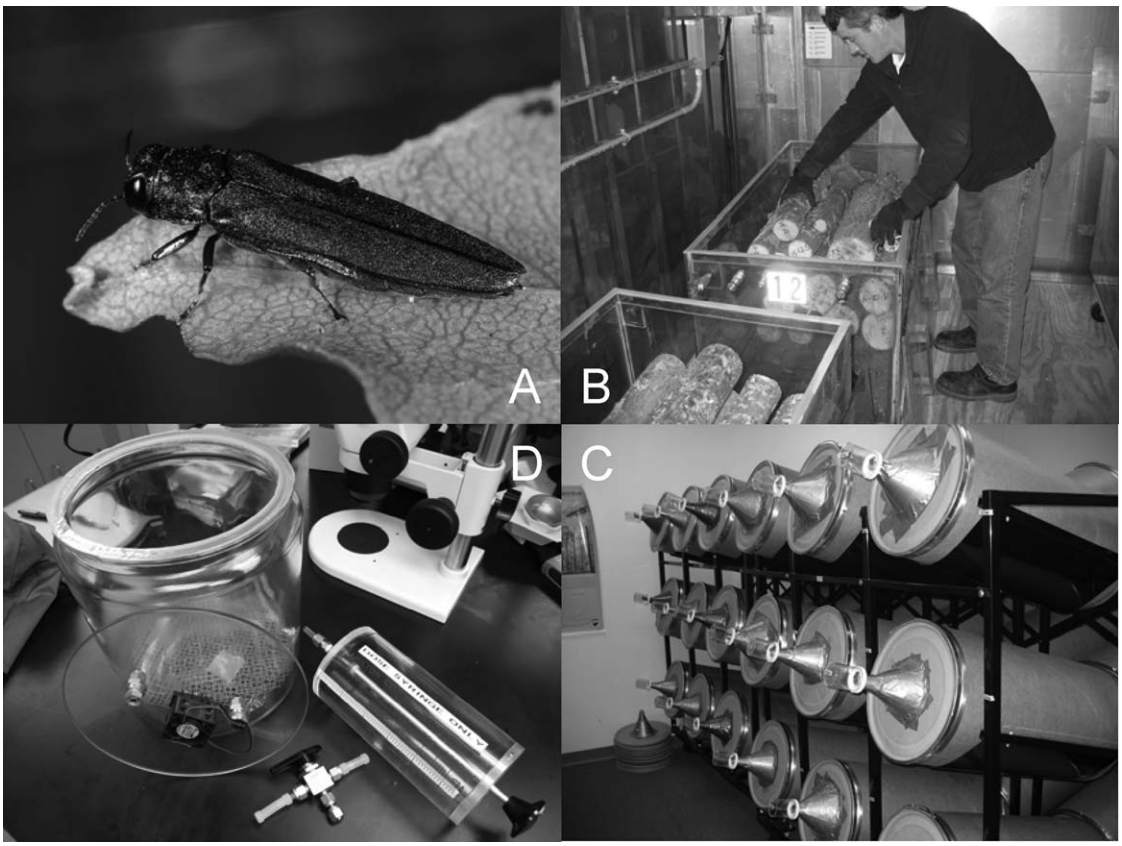

Fig. 1. (A) *A. planipennis* adult on ash leaf. (B) Loading of 432-liter fumigation chambers with infested logs. (C) Fiber drums for rearing out adults from infested, fumigated lash logs. (D) Fumitorium (10 liter) and gastight syringe for gas transfer used to fumigate eggs of *A. planipennis*.

treatment options to chipping and heat for logs infested by species of quarantine significance in international and domestic trade, where these are not practical or may damage log quality.

Fumigation of wood packing materials with sulfuryl fluoride (SF) has been documented to effectively control Asian longhorned beetle, *Anoplophora glabripennis* (Motschulsky) (Barak et al. 2006). Sulfuryl fluoride has been evaluated for control of many other wood-destroying beetles (Williams and Sprenkel 1990; Soma et al. 1996, 1997; Mizobuti et al. 1996). Marketed as Vikane gas fumigant (Dow AgroSciences, Indianapolis, IN), SF has been used since 1961 in the United States for the control of lyctid, anobiid, and cerambycid beetles infesting structural wood, furnishings, and artwork.

The purpose of this research was to evaluate SF fumigation as a quarantine treatment for emerald ash borer in ash logs as an alternative to methyl bromide (because of ozone depletion potential) fumigation (International Standards for Phytosanitary Measures [ISPM] Rule 15, Anonymous 2000). This report provides confirmatory testing for dosages of SF for 24- and 48-h exposure at 15.6 and 21.1°C required for control of *A. planipennis* infesting ash logs. Proposed dosages compensate for effects of wood moisture content, wood loading ratios, and pest life stage on fumigant efficacy.

Materials and Methods

Log Fumigation Chambers. Log fumigations were conducted in six 432-liter Lexan chambers described by Barak et al. (2005, 2006) (Fig. 1B). Two brass 12.7-mm bulkhead compression fittings (The Swagelok Company, Solon, OH) were added to the chambers to allow direct connection of the fumigant monitor (described below) to the chamber to measure SF concentrations. An additional stainless steel 6.35-mm Swagelok compression fitting was added to provide access for thermocouple wires and electricity to a 12-V ventilation fan placed inside each chamber to facilitate gas circulation. The chambers were kept in a 6.1-m-long refrigerated marine cargo container ("reefer") capable of holding set point ±0.1°C.

Log Preparation. Logs and large branches of up to ≈30 cm in diameter, harvested from infested ash trees in the Brighton, MI, area, were cut to lengths of ≈70–72 cm to fit in the *A. planipennis* emergence barrels. Each log was weighed and measured for length and diameter to calculate its volume and bark surface area. Logs were cut in late fall to ensure that

many insects were in the overwintering prepupal stage. They were then stored until use at 4.0 ± 0.1°C. Logs were gradually acclimated to the desired fumigation temperature 3–4 d before fumigation.

Ten to 12 logs were loaded in each fumigation chamber (Fig. 1B), selected to result in similar replicate weights (107.08 ± 8.28 kg [mean ± SEM]) and load factor (28.67 ± 2.06% (vol:vol) (Tables 1 and 2). Before each fumigation, three ≈2.5-cm-thick sections were sawn from the middle of three representative log sections and were oven-dried to determine mean wood moisture contents according to American Society for Testing and Materials (ASTM) test method D-4442-92 (ASTM 1992). Internal wood temperatures for each chamber were monitored by inserting a type T thermocouple into holes drilled ≈2.5 cm in depth into a large log, which was then plugged with electrician's duct putty. When the wood reached the desired temperature, the chamber lids were put on, sealed with heavy duct tape, and vacuum-tested for tightness with a digital manometer (Dwyer Instruments, Michigan City, IN). Container temperature set point was frequently monitored to maintain the target wood temperature ±0.5°C.

Log Chamber Fumigation Trials. Commercial grade SF (99.8%; Dow AgroSciences, Indianapolis, IN) was used in all trials. Fumigations were conducted at 10.0 and 15.6°C for 24 and 48 h during May 2007 at applied doses of 144, 112, 136, and 104 g/m^3, respectively (Table 1). Based on those results, fumigations at four doses—144 and 128 g/m^3 for 24 and 48 h, respectively, at 15.6°C and 128 and 104 g/m^3 for 24 and 48 h, respectively, at 21.1°C—were conducted and evaluated during September–November 2007 (Table 2).

The fumigant dose, based on the known volume, was computed volumetrically by calculating the volume of the required grams of SF gas, according to equation V = nRT/P (the ideal gas law), that compensated for ambient gas temperature and atmospheric pressure. Fumigant was introduced from 10- to 20-liter gas bag reservoirs (Calibrated Instruments Inc., Hawthorne, NY) by drawing a slight chamber vacuum, thus allowing the dose to infuse from a connected gas bag, as described by Barak et al. (2005, 2006).

Fumigant Monitoring. Fumigant concentrations in the test chambers were measured at six intervals for 24-h exposures (0.1, 1, 2, 4, 20, and 24 h after gas introduction) and at eight intervals for 48-h exposures (0.1, 1, 2, 4, 19 or 21, 24, 43, 45, and 48 h after gas introduction) by using a SapphIRe infrared monitor (Thermo Fisher Scientific, Waltham, MA). Accuracy was tested in the laboratory and found to be within 1% from 0 to 40,000 ppm when checked with prepared mixtures. The ppm SF measured by the SapphIRe was converted to g/m^3 based on the temperature and pressure at which the SapphIRe application was developed (1 g/m^3 = 238.1 ppm). The g/m^3 calculation also was measured by making 10 mixtures of various ppm SF, recording the SapphIRe ppm determination, and correlating that with the same sample monitored in g/m^3 by a Spectros IR SF monitor (Spectros Instruments, Hopewell, MA) developed for fumigation monitoring. The result correlation was Spectros g/m^3 = 0.00429 × Sapphire ppm, with $r2$ = 0.9992. The average of both methods of measuring g/m^3 was used as the accepted g/m^3 concentration and calculated concentration × time (CxT) product. The average maximum difference between the two methods was 1.9 ± 0.53 g/m^3 (mean ± SEM) and minimum differences were between 0.25–2.8 g/m^3. After 24- or 48-h exposures, each fumigation chamber was opened and aerated ≈30 min, by using the containers air exchange capacity. Each fumigant dose and exposure period was replicated six times per temperature. There was one untreated control per exposure period and temperature.

***A. planipennis* Emergence Assays.** To measure *A. planipennis* emergence, three to five logs were placed in each a number of in modified fiber drums (39.4 cm in diameter by 77.5 cm in height) (Barrels Boxes and More, Bolton, CT) in metal barrel racks and maintained at 20–23°C and ambient relative humidity. The covers of the drums had a large plastic funnel attached, allowing the adults to crawl out where they were then trapped in an attached plastic cup (Fig. 1C). The number of beetles emerging from logs was recorded after fumigation until the time that no further adults were collected from control logs for at least two more weeks, which lasted up to 8 wk postfumigation. The adult emergence from control logs was expressed as emergence per square meter of bark surface (Petrice and Haack 2006) from which estimated potential treatment emergence was extrapolated from total bark surface area, a method used by.

Cargo Container Commercial Fumigations. The infested ash wood was fumigated June 2008 in four commercial 149.1-m^3 cargo containers (13.7 by 4.0 by 2.7 m). The purpose was to confirm that sufficient periodic concentrations could be maintained with actual commercial fumigation methods and load factors. Infested logs were similar in size to those used in the fumigations described here. One week before logs were fumigated, the storage temperature was increased to 12.0°C to begin the warming of the logs to the target temperatures of 15.6 and 21.1°C. A small spot of paint was sprayed on the cut end of each log to indicate treatment temperature and exposure times. After temperature conditioning, logs were loaded into the sealed cargo box of a truck and transported directly to Indianapolis, IN, for fumigation in tarped, open-door cargo containers on a truck chassis inside a warehouse. Before loading, each infested ash log was weighed to obtain a treatment size of 270.63 ± 4.22 kg (mean ± SEM) ash per container. There were 22–29 ash logs fumigated per cargo container. Nonfumigated control ash logs (229.54 kg; 22 logs) were maintained in the warehouse throughout the fumigation trials.

Large commercial grade hardwood logs (*Quercus* spp. and *Prunus* spp.) supplemented ash logs to load containers and obtain more realistic container fill ratios ranging from 50 to 80% (see Table 4). Internal wood temperature was monitored by thermocouples as described above. A large electric fan of >33-m^3/min capacity was placed on top of the load near the rear

doors to maximize SF circulation. Each container was sealed by covering with nylon-coated tarpaulins which were secured to the ground using 1-m-long sand snakes.

Fumigations were conducted at two target g-h/m^3 dosages per temperature as follows: 144 g/m^3 × 24 h and 128 g/m^3 × 48 h at 15.6°C and 128 g/m^3 × 24 h and 104 g/m^3 × 48 h at 21.1°C. Each fumigant dose and exposure period was conducted once per temperature/dose. Fumigant was weighed and introduced into a container through 0.67-cm-i.d. LLDP hose (Hudson Extrusion Inc., Hudson, OH) of ≈30 m in length.

Gas samples were extracted through tubing (6.35-mm-o.d., 4.8-mm i.d. polyethylene) placed in three standard locations within each container: front-high, center-middle of load, and low-back near the floor. The tubing terminated at a monitoring station in an area separate from the containers. The fumigation start time was considered the end of the fumigant introduction period. Concentrations of SF were monitored with the Spectros infrared monitor with all three sample lines at 5 min and 0.5, 1, 2, 4, 16, and 24 h and (for extended exposures) 48 h after introduction. Accuracy was tested in the laboratory and found to be within 1% at several concentrations from 2,500 to 50,000 ppm. Fumigated and nonfumigated ash logs were transported by truck back to the USDA–APHIS–PPQ facility in Brighton, MI, where adult emergence was monitored as described above, and expected emergence was calculated based on emergence per kilogram of treated logs.

Egg Fumigations. When eggs became available from other projects, the eggs were fumigated in 10-liter glass fumatoria (Fig. 1D) made from glass storage jars (Anchor Hocking, Lancaster, OH). A silicone gasket was made to gastight seal of the plate glass lid. Each lid was fitted with two Swagelok 6.35-mm (0.25-in.) stainless steel compression fittings closed with a 10-mm silicone septa to introduce the fumigant and monitor concentrations. A 12-V microfan was also attached to the inside cover to provide gas circulation immediately after the gas introduction and before each sampling period.

Eggs were collected by allowing 10 mixed sex adults to mate and freely oviposit through nylon mesh onto filter paper held in the lids of ≈2.13-liter wide-mouthed plastic jars (Rez-Tech Corp., Kent, OH). The filter papers were cut apart to select six batches with approximately equal number of eggs. Eggs which microscopically observed to have changed from the yellow color of newly oviposited eggs to a darker color during development and remained plump were observed to be viable (Bauer et al. 2004), whereas those that shriveled were nonviable. Filter papers with eggs were placed in a 177-ml plastic cup inside each fumigation jar. Lids were sealed with silicone vacuum grease (Dow Corning Corp., Midland, MI) before fumigation. The doses used were calculated to approximate the CxT achieved by the successful chamber fumigations at 21.1°C for 24 and 48 h (see Table 2). Two additional fumigations were conducted for the same times and temperature, but doses 16 g/m^3 lower than the first two fumigations. Each dose and exposure period was conducted once per temperature, due to the difficulty of collecting large numbers of eggs. SF gas was introduced by withdrawing an air sample equal to the dose volume plus 50 ml and then replacing the air with the dose gas measured in a 500-ml gastight syringe (Hamilton Co., Las Vegas, NV) by means of the three-way Swagelok valve specified above. Pressure was equalized, and gas remaining in the fittings was pulled into the fumitorium when the syringe was disconnected. Fans were operated for 1 min after the fumigant was introduced to achieve full mixing. Fumigant concentrations were measured after 1 min and 0.5, 4, 20, 24, and 48 h. A 2.5-ml sample volume was drawn with a 2.5-ml gastight syringe inserted through one compression fitting. The sample was directly injected into a SapphIRe infrared (IR) monitor modified to create a closed loop system with a septum injection port and with a known internal volume 2,260 ml. The sample of 2.5 ml therefore resulted in a dilution factor of 905. The application was first authenticated by injecting 100 μl of 99.8% SF and comparing the readout to the calculated concentration of 44.2 ppm. The application for the monitor was created to operate over a range of 0–100 ppm by using the "long path" infrared light path length for maximal accuracy. Readings were multiplied by the dilution factor to obtain the ppm in each fumitorium. The ppm was then converted to g/m^3 as described above. The high-dose 24-h fumigation was terminated 1 h early to approximate target CxT, and the high-dose 48 h fumigation had a small quantity of atmosphere replaced by air after 20 h to reduce SF concentration and approximate target CxT more closely. After fumigation, each fumitorium was aerated and the plastic cups with eggs were removed. Eggs were then held at ambient laboratory temperature (≈23°C) where they were observed daily over a two week period until all eggs either hatched or were considered dead. Eclosion indicated possible survival, and partial eclosion was considered as mortality. Neonates were placed on artificial diet and observed until death or survival was confirmed.

Sorption and Load Factor. To measure fumigant sorption, fresh ash logs (13–27 cm in diameter) were cut in western Massachusetts in a forest area, free of *A. planipennis*, to determine sorption curves and minimum concentrations and CxT dosages obtained by using two chamber loading factors of ≈31.0 and 45.5% (vol:vol). Logs were cut into ≈75-cm lengths, weighed, and measured to yield similar load volumes in each replicate. Wet basis moisture content was determined by sampling a 2–3-cm slice from the center of two logs (ASTM 1992). Fumigations were conducted as described above in the log chamber fumigation experiment, except that a Spectros IR SF monitor was used to measure periodic concentrations for up to 48 h. There were four replicates for each dose (104, 128, and 144 g/m^3) for both loading factors, all done at 14–15°C. After fumigation, the logs and chambers were aerated for at least 24 h before using again for the next fumigation.

Table 1. Fumigation parameters, chamber loading factors, and observed and expected adult emergence of *A. planipennis* adults from ash logs fumigated with sulfuryl fluoride at two temperatures and four doses for 24 or 48 h in 432-liter chambers (April–May 2007, Brighton, MI)[a]

Temp (°C)	Duration (h)	Dose (g/m³)	CxT product (g-h/m³)	Log parameter			Adult emergence		Binomial CL ($P = 0.99$)
				kg (mean ± SEM)	% (vol:vol) (mean ± SEM)	Bark area total (m²)	Observed total (n/m²)	Total[b] expected	
10.0	24	0	0	100.6	24.6	2.946	170 (57.70)		
	24	144	3,647 ± 6.32	100.1 ± 0.19	26.9 ± 0.39	18.889	2	1090	0.9915–0.9999
10.0	48	0	0	100.7	27.7	3.211	92 (28.65)		
	48	112	4,873 ± 8.98	99.7 ± 0.22	25.6 ± 0.57	18.787	4	538	0.9768–0.9987
15.6	24	0	0	99.84	26.6	2.907	128 (44.03)		
	24	136	3,567 ± 6.08	100.4 ± 0.12	27.17 ± 0.95	17.298	0	762	0.9940–1.0
15.6	48	0	0	100.34	28.6	3.121	144 (46.13)		
	48	104	4,561 ± 8.25	100.1 ± 0.09	27.04 ± 0.34	19.201	3	886	0.9877–0.9996

[a] There were six treatment replicates and one control replicate, with 9–12 logs per replicate.
[b] Total expected is control emergence emerald ash borer adult emergence per square meter bark times total square meters bark in combined six replicates.

All results were tabulated and displayed with Microsoft Excel (Anonymous 2000). Statistical analysis (means and SEM) was performed using Statistix 9 (Analytical Software 2008), and binomial confidence limits (CL) were calculated using an online calculator (Sauro 2005).

Results

Log Chamber Fumigation Results. In the initial chamber fumigations, there was some adult emergence at 10 and 15.6°C fumigations (Table 1). As a result, we decided to suspend 10°C tests because of the very high doses (>144 g/m³) required at this low temperature.

The results of initial fumigations at 10.0 and 15.6°C led to the additional chamber fumigations at 15.6 and 21.1°C and with slightly higher doses (Table 2). These higher doses for 24- and 48-h fumigations at 15.6 and 21.1°C resulted in accumulated respective CxT dosages shown in Table 2. Bark surface in control treatments averaged 3.08 ± 0.061 m² (SEM); and in treatments, total bark area of combined replicates was 18.799 ± 0.271 m². From these data, the total expected emergence from the fumigations ranged from 271 to 2,457. No adults emerged from any of the fumigated logs. In comparison, 993 adults emerged from combined nonfumigated control logs.

The successful chamber fumigations were conducted using chamber loading factors between 25.8 and 30.5% (Table 2). Higher chamber loading did not result in great loss of SF through sorption in wood (see Sorption and Load Factor below). Moisture contents of representative control logs (average ± SEM % wet basis) were between 26.4 ± 0.55 and 29.6 ± 0.47% and were typical of those measured in untreated ash wood, stored outdoors, from which adults had successfully developed and emerged (Petrice and Haack 2006). The logs used in sorption tests were from freshly felled logs and had a wet basis moisture content of 32.75 ± 0.07%. (ASTM 1992).

Cargo Container Commercial Fumigations. Actual wood core temperatures were higher than planned; a mean of 8.8°C higher for 15.6°C and 3.7°C higher for 21.1°C. This was the result of the commercial fumigations not being conducted in a climate-controlled environment, which represents typical conditions. The mean accumulated dosages for 24-h exposure were

Table 2. Fumigation parameters, chamber loading factors, and observed and expected emergence of *A. planipennis* from ash logs fumigated with sulfuryl fluoride at 15.6 and 21.1°C for 24 or 48 h in 432-liter chambers (September–November 2007, Brighton, MI)[a]

Temp (°C)	Duration (h)	Dose (g/m³)	CxT product (g-h/m³)	Log parameters			Adult emergence		Binomial CL ($P = 0.95$, $P = 0.99$)
				Kg (mean ± SEM)	% (vol:vol) (mean ± SEM)	Bark area total (m²)	Observed total (n/m²)	Total[b] expected	
15.6	24	0	0	100.1	25.8	2.983	43 (14.4)		
	24	144	3,723 ± 4.52	100.1 ± 0.07	26.8 ± 0.32	18.793	0	271	0.9890–1.0 0.9832–1.0
15.6	48	0	0	99.5	26.2	3.05	119 (39.0)		
	48	128	6,072 ± 7.05	100.3 ± 0.18	28.5 ± 0.42	19.417	0	758	0.9961–1.0 0.9939–1.0
21.1	24	0	0	114.72	29.8	3.429	426 (124.2)		
	24	128	3,172 ± 13.82	114.7 ± 0.19	30.4 ± 0.49	19.781	0	2457	0.9988–1.0 0.9981–1.0
21.1	48	0	0	115.1	30.5	2.993	345 (115.3)		
	48	104	4,210 ± 52.50	115.0 ± 0.12	29.9 ± 0.56	18.222	0	2100	0.9986–1.0 0.9878–1.0

[a] There were six treatment replicates and one control replicate, with 12 logs per replicate.
[b] Total expected was control emergence emerald ash borer adult emergence per square meter bark times total square meters bark in combined six replicates.

Table 3. Calculated and actual dosages of sulfuryl fluoride introduced into commercial shipping containers (June 2008, Indianapolis, IN), and subsequent emergence of *A. planipennis* from fumigated and nonfumigated ash logs

°C actual	Hours exposure	Load[a] factor	Kilograms of wood	SF to add calculated (kg)	Initial applied (kg)	Target (g/m^3)	Target CxT ($g\text{-}h/m^3$)	Actual CxT ($g\text{-}h/m^3$)	Adult emergence[b] predicted/observed $P = 0.99$ CI
23.9	24	75	281.84	21.54	21.55	144	3,382	2,877	764/0 0.9940–1.0
24.8	48	50	261.40	19.15	19.84	128	5,466	6,023	707/0 0.9935–1.0
23.5	24	75	270.06	19.15	20.91	128	3,329	2,980	731/0 0.9937–1.0
26.0	48	80	269.23	15.56	15.74	104	4,385	5,171	728/0 0.9937–1.0
			226.80	0	0	0	0		621

[a] Load factor is percentage of container only. Added space under a tarped truck chassis may increase fumigation volume by up to 50%.
[b] Predicted adult emergence was based on adults per kilogram of control logs times kilogram test logs in each treatment.

approximately equal, and for 48-h exposures were higher than the targeted dosages or doses obtained in small chambers (Table 3). No adults emerged from any of the fumigated logs. In comparison, 621 adults emerged from nonfumigated logs, demonstrating that the test logs were infested, and the fumigation dosages tested resulted in 100% control of *A. planipennis* infesting the logs. Estimated populations in the seeded, infested logs were 732.5 ± 11.78 (SEM), after calculating adult emerged per kilogram log weight of the controls.

Egg Fumigations. The result of egg fumigations is shown in Table 4. A high percentage, ≥84%, of control eggs hatched. Only eight eggs (mean, 5%) in total partially hatched from treatments with a CxT of 3,703 $g\text{-}h/m^3$ or less, and no eggs hatched at a CxT of 4,262 $g\text{-}h/m^3$. Neonates were placed on artificial diet. No fumigated larvae survived and developed after eclosion, including at a CxT dosage (3,042 $g\text{-}h/m^3$) below that in the proposed quarantine treatment schedule (see Table 6). Seven control larvae survived to instar I and three to instar II. Rearing *A. planipennis* on artificial diet from eggs and has been very inconsistent in the laboratory (as is oviposition) primarily due to difficulty in maintaining humidity and moisture content of the diet. Therefore, adults for oviposition have not been are not available in large numbers.

Table 4. Mortality of *A. planipennis* eggs fumigated at four doses at 21.1°C for 24 or 48 h in 10-liter glass fumitoria[a]

Applied dose (g/m^3) at 21.1°C	Duration (h) of fumigation	CxT dosage ($g\text{-}h/m^3$)	Viable eggs in treatment	Eggs hatched full/partial[b]	% hatched[c]
0, Control	24	0	71	69/2	97.18
129.6	24	3,042	60	5/1	8.33
145.5	22.8	3,240	31	2	6.45
0, Control	48	0	57	48/3	84.21
79.3	48	3,703	60	1	1.67
94.9	48	4,262	60	0	0

[a] Doses were calculated and adjusted to approximate CxT exposure of successful fumigation of larvae within ash logs.
[b] Partial hatch, larvae did not clear the chorion.
[c] Partial eclosion was not considered a surviving larva but as natural mortality. All fumigated larvae failed to survive following eclosion and were not found on diet.

Sorption and Load Factor. Fumigations at 104, 128, and 144 g/m^3 SF at 15.6°C, with the higher load factor had higher periodic concentrations than chambers with lower load factors even though there was more wood to absorb the SF (Table 5). In five of six treatments, the concentrations through 48-h exposure were higher than the calculated applied dose. Under these conditions, half loss time with large load factors was longer than the fumigation exposure time, thus showing that ash logs are only slightly sorptive of SF compared with highly sorptive methyl bromide, which was documented by Kenaga (1957). The relatively flat sorption curves and steep accumulation of CxT exposure (e.g., compared with highly sorptive methyl bromide) are illustrated in Fig. 2.

Discussion

A proposed SF quarantine treatment for *A. planipennis* (Table 6) represents dosages higher than those documented previously for larval and prepupal stages of other beetle species (Kenaga 1957; Mizobuti et al. 1996; Soma et al. 1996, 1997; Su and Scheffrahn 1990; Barak et al. 2006). These researchers have documented that egg stage, not the larval or prepupal stage, is the most tolerant life stage to SF. One possible explanation for the higher doses for larvae in our study may be that the *A. planipennis* larvae had reduced metabolism due to storage of infested logs at 4°C, as did larvae of Asian longhorned beetle (Barak et al. 2006). The logs in this study were slowly acclimated to fumigation temperatures for several days before fumigation to ensure wood cores reached the desired fumigation temperature, but it is unknown if the respiration rates and metabolic rates of *A. planipennis* larvae were comparable to nonchilled larvae. Reduced metabolic rates, such as those observed in diapausing prepupal insects, can require significantly higher fumigant dosages for control (Bell 1994). Another explanation is wood of higher density than pine (*Pinus* spp.) can reduce fumigant penetration up to 92% for sulfuryl fluoride and up to 98% for methyl bromide (Scheffrahn et al. 1992). These researchers also documented that water-saturated pine, compared with nonsaturated pine, reduced penetration of both fumi-

Table 5. Periodic sulfuryl concentrations during 48-h fumigations of heavy-barked *Fraxinus* spp. bolts in 432-liter chambers with load factors of 31 and 46% (vol:vol) at three doses

Applied dose (g/m^3)	Time (h) after gas introduction completed (mean concn ± SEM, g/m^3)								Mean CxT ($g\text{-}h/m^3$ ± SEM) (after 24 h, after 48 h)
	5 min	0.5	1 h	2 h	4 h	8 h	24 h	48 h	
31% loading									
104	143 ± 2.28	143 ± 0.46	141 ± 0.36	138 ± 0.43	134 ± 0.54	130 ± 0.56	123 ± 0.55	117 ± 0.47	3,104 ± 12.95 5,993 ± 24.74
128	180 ± 0.84	176 ± 0.86	174 ± 0.78	170 ± 0.66	166 ± 0.64	161 ± 0.68	153 ± 0.77	147 ± 0.86	3,851 ± 16.69 7,450 ± 35.68
144	204 ± 0.84	202 ± 0.25	199 ± 0.23	195 ± 0.08	190 ± 0.09	185 ± 0.40	172 ± 0.43	163 ± 0.42	4,387 ± 7.92 8,404 ± 13.85
46% loading									
104	183 ± 1.40	174 ± 1.83	167 ± 1.56	157 ± 1.75	144 ± 1.77	129 ± 1.80	98 ± 1.83	77 ± 1.83	2,995 ± 42.46 5,089 ± 86.24
128	219 ± 2.16	211 ± 2.94	205 ± 2.99	199 ± 2.89	190 ± 2.77	179 ± 2.58	156 ± 2.26	136 ± 2.28	4,218 ± 59.85 7,724 ± 113.6

gants by 90%. These reasons could explain why the *A. glabripennis* larvae and prepupae imbedded in green ash logs would receive lower total exposure at the same applied dose compared with eggs directly exposed on the bark surface or in open crevices (Bauer et al. 2004) for 100% mortality. Therefore, higher doses needed to control embedded larvae will exceed the required CxT exposure needed for naked eggs.

Eggs pose less of a risk than prepupae for survival and transmission of *A. planipennis* in logs. There is lower probability of eggs developing to mature adults on fresh logs after repeated handling and processing that could include debarking and timely processing requirements mandated by regulatory order. The proposed treatment schedules for SF will result in high mortality of fumigated eggs and delayed mortality of any larvae that hatch. Delayed mortality of all life stages of insects, including beetles, exposed to lethal concentrations of sulfuryl fluoride has been well documented (Su and Scheffrahn 1990). Bell and Savvidou

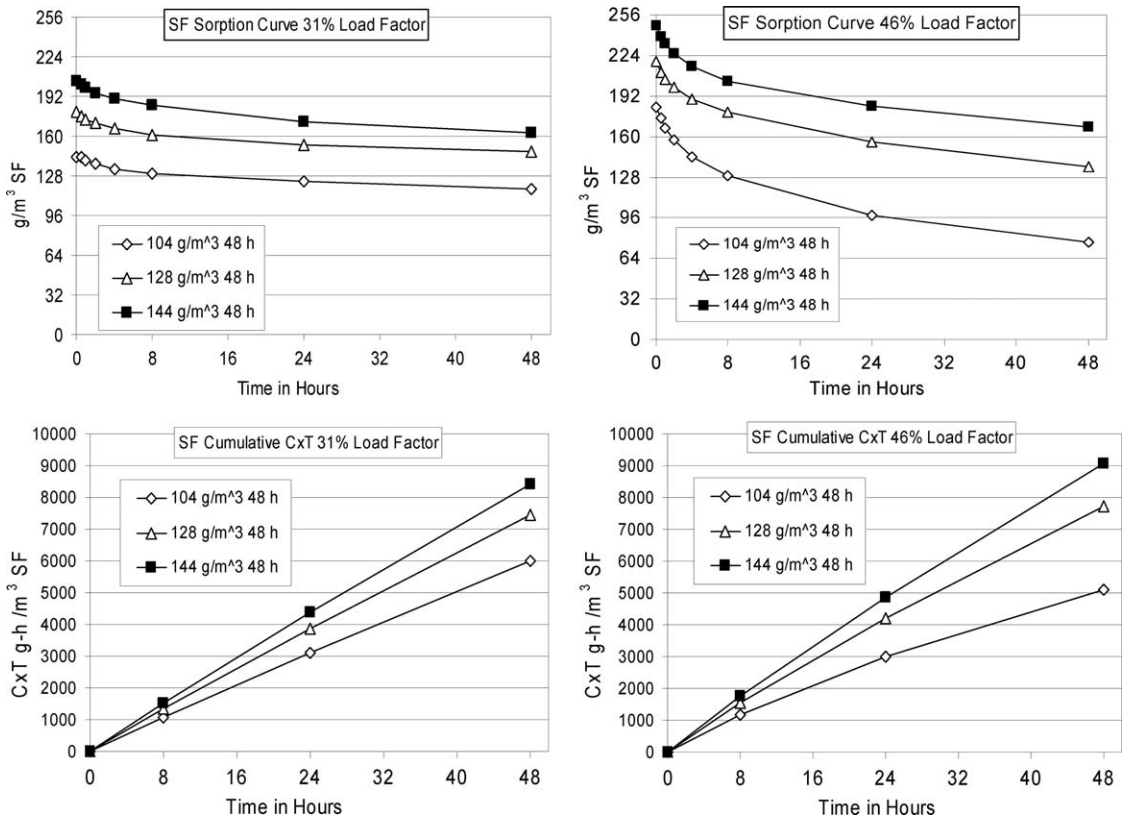

Fig. 2. Ash logs fumigated at three doses of sulfuryl fluoride for 48 h at load factors of 31.0 and 46.5% (vol:vol). Sulfuryl fluoride sorption curves (top) and accumulated CxT dosages ($g\text{-}h/m^3$) (bottom).

Table 6. Recommended applied doses and periodic concentrations of sulfuryl fluoride to eliminate *A. planipennis* emergence in ash logs at 15.6 and 21.1°C for 24 and 48 h[a]

Temp (°C)	Applied dose (g/m^3)	Minimum concn (g/m^3) at time indicated (h)					Required CxT exposure (g-h/m^3)
		0.5	2	4	24	48	
15.6	144	187	181	170	137		3,723
15.6	128	177	165	156	120	101	6,072
21.1	128	168	156	147	109		3,172
21.1	104	129	119	112	82	66	4,210

[a] Based on small chamber fumigations with load factors of 26.8–30.4% (vol:vol).

(1990) further documented that dosages of SF required for 100% control of the Mediterranean flour moth, *Anagasta kuehniella* (Zeller), could be reduced by ≈42% if delayed mortality after hatching of fumigated eggs was taken into consideration.

The Probit-9 standard for quarantine efficacy (99.99683%) was developed for tropical fruits with heavy infestations of fruit flies (Follett and McQuate 2001). Follett and McQuate (2001) also proposed that these standards may be too high for situations of lower risk and less abundant population. *A. planipennis*, as well as possibly other wood boring beetles in the families Cerambycidae or Buprestidae, arguably fits this situation, because it is not practical to obtain Probit-9 numbers of test subjects. Follett and McQuate (2001) describe situations where population and risk parameters would require control levels of 99.532 with as few as 639 insects per test. Stanley (1997) further discusses the USDA view that "the severity of the treatment be tailored to the risk of the commodity." The stated goal of USDA–APHIS–PPQ through ISPM Rule 15 (FAO 2002) the stated goal of USDA–APHIS–PPQ, through ISPM normalization is to "reduce the risk of introduction and/or spread of quarantine pests" and to describe measures that "significantly reduce the risk of pest spread," we believe this recommended schedule for *A. planipennis* can meet that requirement.

The commercial fumigations confirmed accumulation of CxT dosages necessary for quarantine treatment of *A. planipennis* in ash logs are practical using existing industry practices. The periodic concentrations measured throughout the 24- and 48-h fumigations periods and resulting accumulated dosages were similar for the commercial fumigations compared with the mean concentrations for the successful chamber fumigations with the comparable target doses (Fig. 3).

These tests confirm 100% control of naked eggs in chambers and larvae within logs at 15.6°C by CxT dosages of 3,723 and 6,072 g-h/m^3 of SF for 24- and

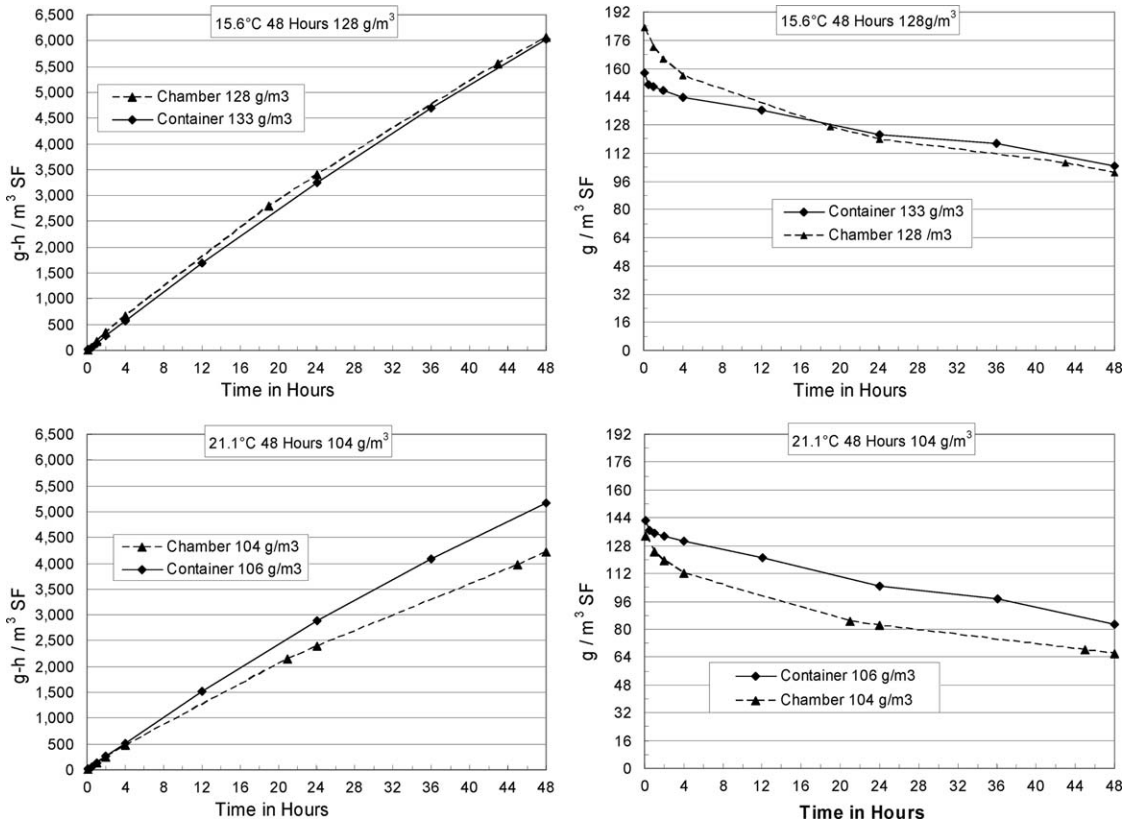

Fig. 3. Fumigation of hardwood logs in chambers versus tarped general cargo containers for 48 h at high dose (133–128 g/m^3) (top) and lower dose (104–106 g/m^3) (bottom). Accumulated CxT (eft), with sorption curves over time (right).

48-h exposure, respectively, and at 21.1°C by dosages of 3,172 and 4,210 g-h/m^3 SF for 24- and 48-h exposure, respectively. These proposed rates of application were effective under commercial fumigation conditions.

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