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Emission Characteristics of Elm Bark Beetle Aggregation Attractants from Controlled-Release Dispensers

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Abstract

Release rates of the aggregation attractants of the smaller European elm bark beetle, *Scolytus multistriatus* (Marsham), from laboratory-aged and field-aged Conrel and Hercon dispensers were monitored for 85 days by GLC analysis of cold-trapped volatiles. Both dispensers had relatively low and constant rates of decay for all three attractant components after an initial burst in emission rates. Within limits, the ratio of components released remained constant over time and at various temperatures.

Introduction

Controlled-release pheromone dispensers, herein called lures, are key elements in the study and effective use of insect attractants. Most identified attractants are a mixture of two or more chemicals. To be most effective, these chemicals must be released in minute, constant amounts at a particular ratio for several days or months and in a variety of environments—not an easy task.

Several companies have developed controlled-release lures. Hercon® laminated plastic dispensers (Health-Chem Corp., New York, NY) and Conrel® hollow-fiber dispensers (Albany International, Needham Heights, MA)¹ are among the most widely accepted for use in insect survey and suppression. The efficacy and longevity of these lures have been evaluated indirectly as part of studies of various insects and their attractants.

In both laboratory and field studies, many changes in controlled-release formulations may be required to optimize release rates or otherwise

improve the lures. The use of field tests to evaluate modification in formulation is prohibitively expensive and time consuming, so laboratory evaluations are more practical. Several methods have been used for determining release rates in the laboratory. These include: (1) measurements of weight loss; (2) extraction of residual semiochemicals; (3) direct observation of volume changes in capillary tubes; and (4) trapping of effluent vapors (see reviews by Roelofs 1979 and Weatherston et al. 1981).

Although problems can be encountered in trapping effluent vapors as a method for determining release rates, there are several advantages in using this technique, the most important of which is that it should give the most accurate measurements (Plimmer et al. 1977; Weatherston et al. 1981). Other studies in which this technique has been used for determining release rates have been reported (Beroza et al. 1975; Look 1976; Bierl-Leonhardt et al. 1979; Baker et al. 1980; Cross 1980; Cross et al. 1980). In this paper we describe aeration-vapor trapping and chemical assays of laboratory-aged and field-weathered Conrel and Hercon lures formulated to emit multilure, the three-component aggregation attractant (Pearce et al. 1975) of the smaller European elm bark beetle, *Scolytus multistriatus* (Marshall).

Materials and Methods

Attractant Collection Technique

The attractants released from lures were collected by an aeration and cold-trapping procedure similar to that described by Gore.² Three Hercon and three Conrel lures were placed in six separate glass chambers (approximately 24 cm long by 4 cm (inside diameter)) in a room maintained at $25^{\circ} \pm 1^{\circ}\text{C}$. The chambers were flushed with nitrogen for 30 minutes before each collection to expel moisture, air, and any excess pheromone on the lure surface. Each chamber was connected to a glass U-tube immersed in a dry ice-acetone bath. Attractants eluted from the lures were carried by the nitrogen stream (65 ml/minute) to the U-tubes where they were condensed and collected.

After 3 hours of aeration/cold-trapping, each U-tube was disconnected from the aeration chamber and removed from the bath. We immediately pipetted 2 ml of hexane into the U-tube through the glass arm previously attached to the chamber, sealed the tube, and agitated it carefully to dissolve the trapped attractants. No attempt was made to wash the walls of the aeration chamber, where a small amount of the attractants probably had accumulated.

¹The use of trade, firm, or corporation names in this publication is for the information and convenience of the reader. Such use does not constitute an official endorsement or approval by the U.S. Department of Agriculture or the Forest Service of any product or service to the exclusion of others that may be suitable.

²Gore, W. E. 1975. The aggregation pheromone of the European elm bark beetle (*Scolytus multistriatus*): Isolation, identification, synthesis, and biological activity. Ph.D. Thesis. Syracuse, NY: State University of New York; 242 p.

GLC Analysis

The hexane-attractant solution was analyzed by GLC to determine the relative amounts of each attractant trapped. A 3- μ l aliquot of each sample solution was injected into a Varian Aerograph Model-1200 gas chromatograph equipped with a flame ionization detector, and a 0.3-cm (outside diameter) by 6.1-m stainless steel column packed with Carbowax 20M on 60/80-mesh Chromasorb G. The helium flow through the column was maintained at 60 ml/minute. Operating temperatures were: injector 185°C, column 150°C isothermal, and detector 215°C. Under these conditions, retention times for the multilure components, 4-methyl-3-heptanol (H), α -multistriatin (M) and α -cubebene (C), were 2.2, 4.2, and 4.7 minutes, respectively.

After each sample was injected and analyzed, a 3- μ l aliquot of a standard consisting of known concentrations of the attractants in hexane was injected and analyzed. The amounts of H, M, and C in the standard varied (though the ratios remained constant), depending on the concentration of the three chemicals in the test samples (as indicated by GLC peak heights); that is, a standard with larger concentrations was used to compare with unaged lures, while a standard with lower concentrations of H, M, and C was used to compare

with baits that had been aged for some time in the laboratory or field. The peak heights of the samples were compared with peak heights of the standards to calculate the approximate relative concentration of each attractant component in the sample. We calculated release rates in terms of μ g/day by multiplying concentration by amount of solvent by aeration time.

Standard solutions were assayed at various aeration gas flow rates, aeration periods and temperatures to determine a set of operating parameters that yielded a high and consistent recovery rate.

Verification of Technique

To verify the precision and accuracy of this aeration-collection-assay technique, five standard solutions containing known amounts of H, M, and C in hexane were pipetted onto filter paper and placed in separate aeration chambers. Volatiles were collected for 4 hours, after which time analysis by GLC was performed. The five standard solutions contained the following dose equivalents (μ g/day H:M:C): 1440:360:2880, 720:180:1440, 288:72:576, 144:36:288, 72:18:144 (Table 1). Because the actual aeration time was only 4 hours, the amount of each chemical applied to the filter paper was actually one-sixth of the quantities listed.

Test Conditions

The release rates of lures were measured under two test conditions: (1) from carefully selected lures aged under uniform controlled laboratory conditions; and (2) from randomly selected lures aged in the field. For the first test we carefully selected lures from the general lot on the basis of apparent physical homogeneity; i.e., uniformly filled tubes in Conrel lures and uniform size and shape in Hercon lures. The lures, three each of Conrel and Hercon, were placed in aeration chambers (identical to those used to collect samples for determining release rates) at $25^\circ \pm 1^\circ\text{C}$ and 65 ml/minute air flow (from laboratory compressed air system) for 85 days. Lures were removed from this system at various intervals during this time and subjected to the aeration-collection-assay technique described.

Lures for the second test, three of each type, were indiscriminately selected from the general lot. They were stapled to a protective cover, attached to a sticky trap, and placed on utility poles (these are the same procedures used in survey or suppression studies) (Lanier 1978, 1979; Lanier et al. 1976; Peacock 1981, 1982) for 85 days. At various intervals, the lures were brought into the laboratory, assayed, and returned to the field.

Results and Discussion

Verification of Assay Technique

Under the conditions of these tests, the release of attractant from lures ranged from about 20:10:100 $\mu\text{g/day}$ of H:M:C to about 2000:500:2500 $\mu\text{g/day}$. The precision and accuracy of the aeration-collection-assay technique in measuring known amounts of attractants applied to filter paper

were fairly constant over this range (Table 1). The standard errors were less than 3 percent of the mean. The percent recovery, which measures efficiency of the system, was about 89 for H, 83 for M, and 75 for C. Although it is clear that the technique consistently underestimated the relative amounts of all three components, it was sufficient to differentiate between lures and to monitor changes in relative release rates over time.

Table 1.—Precision and accuracy of the aeration-collection-GLC technique as indicated by percent recovery after aeration-collection of known amounts of three multilure components

Dose of H:M:C aerated ^a	Dose recovered ($\mu\text{g/day} \pm \text{SE}$)			Percent recovery		
	H	M	C	H	M	C
1440:360:2880	1284 $\pm$ 12	312 $\pm$ 9	2066 $\pm$ 31	89	87	72
720:180:1440	641 $\pm$ 12	150 $\pm$ 6	1106 $\pm$ 17	89	83	77
288:72:576	251 $\pm$ 5	57 $\pm$ 2	445 $\pm$ 6	87	79	77
144:36:288	132 $\pm$ 1	30 $\pm$ 1	213 $\pm$ 1	92	83	74
72:18:144	63 $\pm$ 1	14 $\pm$ 1	112 $\pm$ 3	88	78	78

^aDose aerated refers to equivalent amounts of each component in $\mu\text{g/day}$ initially applied on filter paper in aeration chamber. H:M:C represents 4-methyl-3-heptanol: α -multistriatin: α -cubebene, the components of multilure.

Effects of Temperature

A temperature increase of 10°C generally more than doubled the release rates of all components from both types of lures in lab tests (Fig. 1). Hercon lures (Fig. 1A) were somewhat more temperature sensitive (steeper slopes) than Conrel lures (Fig. 1B) over the range tested. Temperature did not appreciably affect the ratio of components released even though the components have different volatilities. Along with the difference between types of lures, this suggests that temperature sensitivity depends more on the design of the lures than on the characteristics of the chemicals released. Since the ratio of components was not radically affected by temperature, these temperature-induced changes may be advantageous; that is, attractants are conserved during cool periods when beetles are inactive, but are released in large doses when temperatures are high and beetles are responsive.

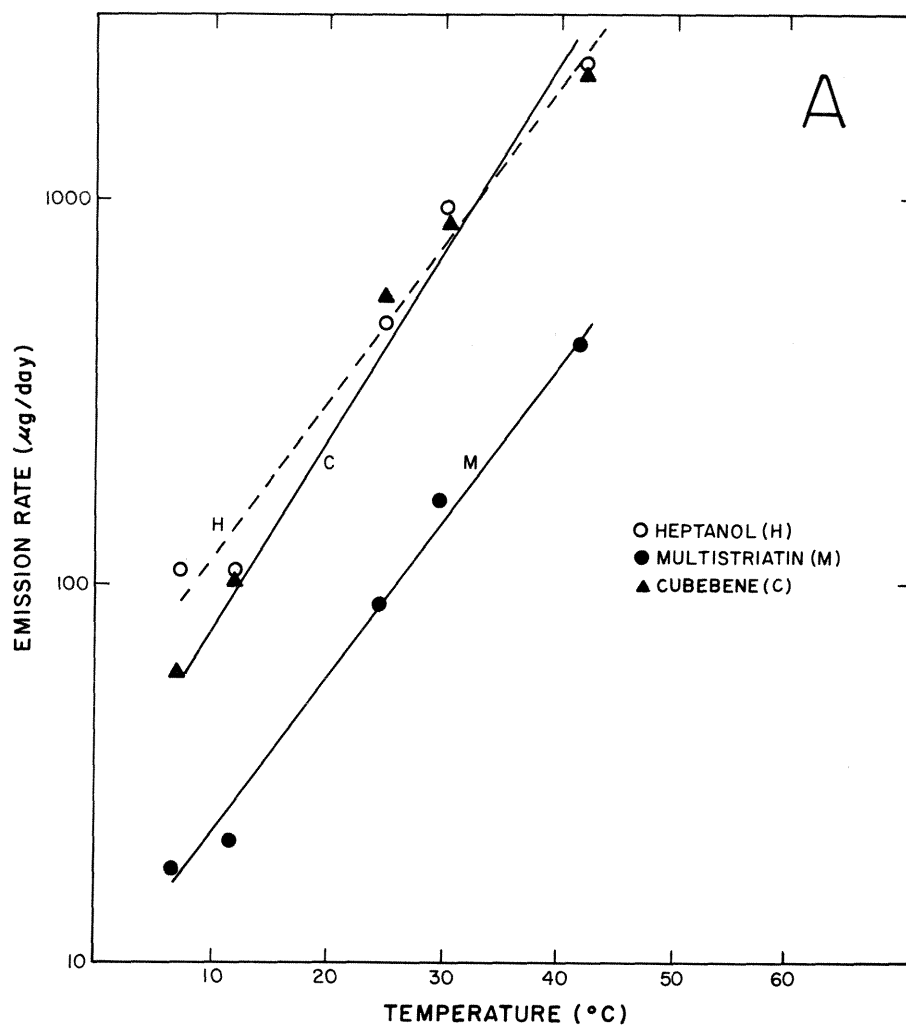
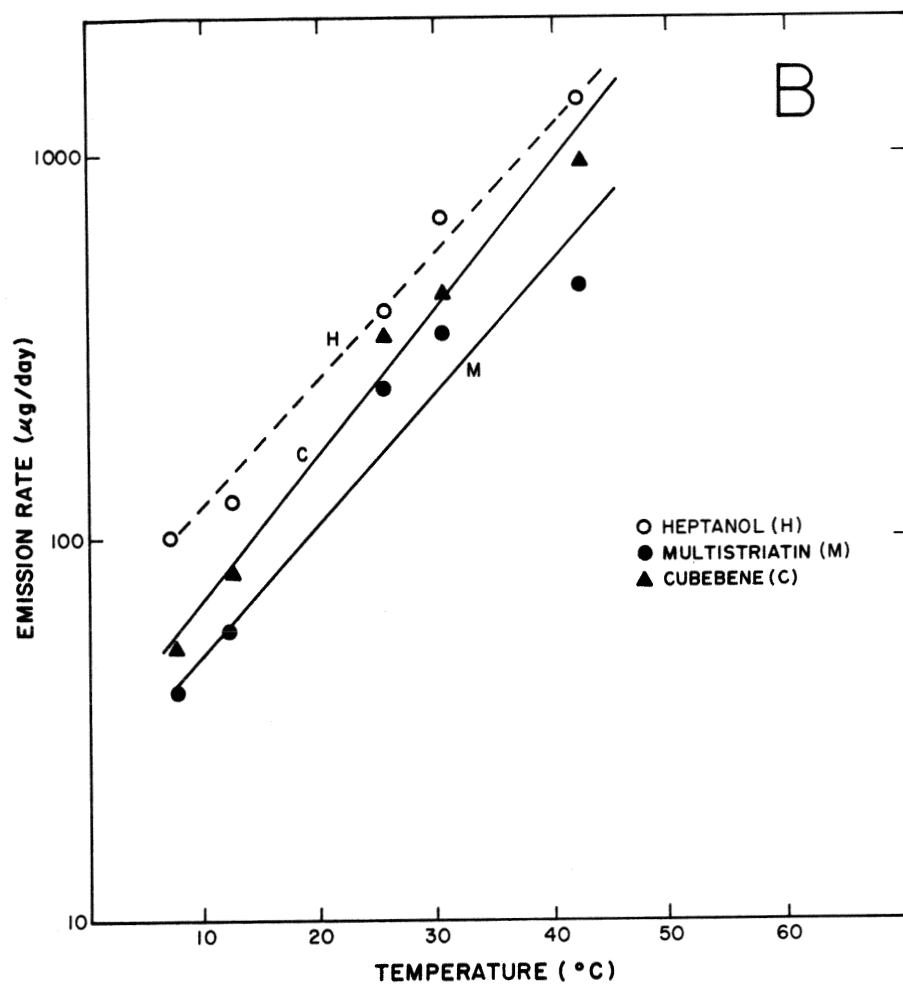


Figure 1.—Effects of temperature on mean release rates of multilure components from controlled release attractant dispensers: (A) Hercon lures; (B) Conrel lures (based on two lures of each type).



Initial Release Rates

For at least a week after initial exposure of the lures (about 7 days for Hercon, 11 days for Conrel) release rates of all components greatly exceeded the specified rates (the lures were designed to release 400:100:800 $\mu\text{g/day}$ of H:M:C) (Table 2). But the release rates fell sharply during those periods. By the 11th day, the emission rate from Conrel lures was about half the initial rate, and the emission rate from Hercon lures, which initially was higher than that from the Conrel lures, declined even

more. The release rates of all components from both lures were near or well below the specified rates after 11 days. However, the high initial rates obviously resulted in a significant percentage of the total pheromone in the baits being emitted during the first 2 weeks of exposure—about one-fourth of the original 49 mg of H in Hercon lures was emitted during that period. In the practical use of lures, these high and rapidly changing rates could introduce bias and variability into

measurements of beetle response, especially in short-term tests of beetle behavior. We expected some initial burst in emission rates due to the accumulation of pheromone on the surface of the lures while they were packaged, but not at the magnitude or duration recorded in this test. Because these initial rates were not typical of the longer-term rates, we deleted the data for the first 2 weeks in evaluating and comparing long-term release rates for the three compounds.

Table 2.—Initial release rates (0–11 days) of multilure components from laboratory-aged Hercon and Conrel lures

Lure and component	Mean ^a release rate ($\mu\text{g/day} \pm \text{SE}$) after exposure for—			
	0 days	3 days	7 days	11 days
Hercon				
Heptanol	2193 $\pm$ 202	683 $\pm$ 54	445 $\pm$ 31	267 $\pm$ 14
Multistriatin	327 $\pm$ 21	123 $\pm$ 9	85 $\pm$ 8	50 $\pm$ 3
Cubebene	1121 $\pm$ 16	833 $\pm$ 15	763 $\pm$ 84	514 $\pm$ 45
Conrel				
Heptanol	1356 $\pm$ 39	986 $\pm$ 95	744 $\pm$ 70	532 $\pm$ 76
Multistriatin	246 $\pm$ 16	219 $\pm$ 25	196 $\pm$ 14	140 $\pm$ 16
Cubebene	862 $\pm$ 91	933 $\pm$ 69	751 $\pm$ 217	469 $\pm$ 58

^aMean values for three of each type of lure.

Release Rates from Laboratory- and Field-Aged Hercon and Conrel Lures

The data from release rate determinations are shown in Figure 2. They indicate a daily emission rate of 1 to 3 percent of the amount of attractants remaining from the previous day. There are some large differences in these curves (between Hercon and Conrel, field- and laboratory-aged); these resulted from a combination of factors including the selection process, weathering-aging conditions, and differences in lures.

Some of the differences in release rate curves between laboratory- and field-aged lures were due to the selection process. As might be expected, the release rates from randomly selected (field-aged) lures were more variable than visually selected (laboratory-aged) lures (Table 3). The selection process also resulted in differences in the dose detected. Careful selection of Conrel lures, in which the attractants are

visible, allowed us to choose lures with higher average release rates than those that were randomly selected from the general lot. But we were unable to select for high release rates from Hercon lures, in which the attractants are impregnated in opaque, laminated plastic. In fact, the average rates of H and M from randomly selected Hercon lures were higher than from carefully selected lures (Table 3).

Table 3.—Mean release rates^a and release-rate ratios for three components of multilure from laboratory- and field-aged Hercon and Conrel lures on given days

Lure and component	Day 14 ($\mu\text{g/day} \pm \text{SE}$)	Ratio	Day 44 ($\mu\text{g/day} \pm \text{SE}$)	Ratio
Lab-aged Hercon				
Heptanol	232 $\pm$ 21 ^b	5.2:1:9.3	66 $\pm$ 7	4.1:1:14
Multistriatin	45 $\pm$ 6		16 $\pm$ 9	
Cubebene	482 $\pm$ 52		219 $\pm$ 8	
Field-aged Hercon				
Heptanol	416 $\pm$ 53	5.7:1:5.8	107 $\pm$ 14 ^c	3.8:1:6.5
Multistriatin	73 $\pm$ 10		28 $\pm$ 4	
Cubebene	426 $\pm$ 28		183 $\pm$ 7	
Lab-aged Conrel				
Heptanol	669 $\pm$ 145 ^b	3.2:1:2.3	243 $\pm$ 18	2.7:1:1.5
Multistriatin	206 $\pm$ 27		91 $\pm$ 18	
Cubebene	471 $\pm$ 18		135 $\pm$ 3	
Field-aged Conrel				
Heptanol	274 $\pm$ 35	2.2:1:2.9	132 $\pm$ 16 ^c	3.4:1:1.7
Multistriatin	126 $\pm$ 21		39 $\pm$ 6	
Cubebene	369 $\pm$ 77		68 $\pm$ 11	

^aMean values for three of each type of lure.

^bRelease rates at day 15.

^cRelease rates at day 42.

The smaller slopes of the field-weathered lures indicate that these lures were depleted more slowly than those aged in the laboratory (Fig. 2), probably for the most part because the average temperature in the field was lower than in the laboratory. However, the air flow around lures, moisture content in the air, and solar radiation may also have influenced depletion rate.

Despite problems introduced by the selection and aging processes, some differences between types of lures are apparent. The slopes of H and M from Conrel lures, whether laboratory-aged or field-aged, were lower than those for Hercon (Fig. 2A, B). This means the average output of these components from the Conrel lures was more constant. The situation was reversed for C because

Hercon lures had a more constant C output (lower slope) (Fig. 2C). These differences in slopes are reflected in the average release rates at 44 days, which is approximately the specified half-life of the lures and a period generally coincident with peak beetle abundance in suppression tests (Lanier et al. 1976). At 44 days, Conrel lures released H and M at higher rates (Fig. 2A, B) than Hercon and C at lower rates (Fig. 2C).

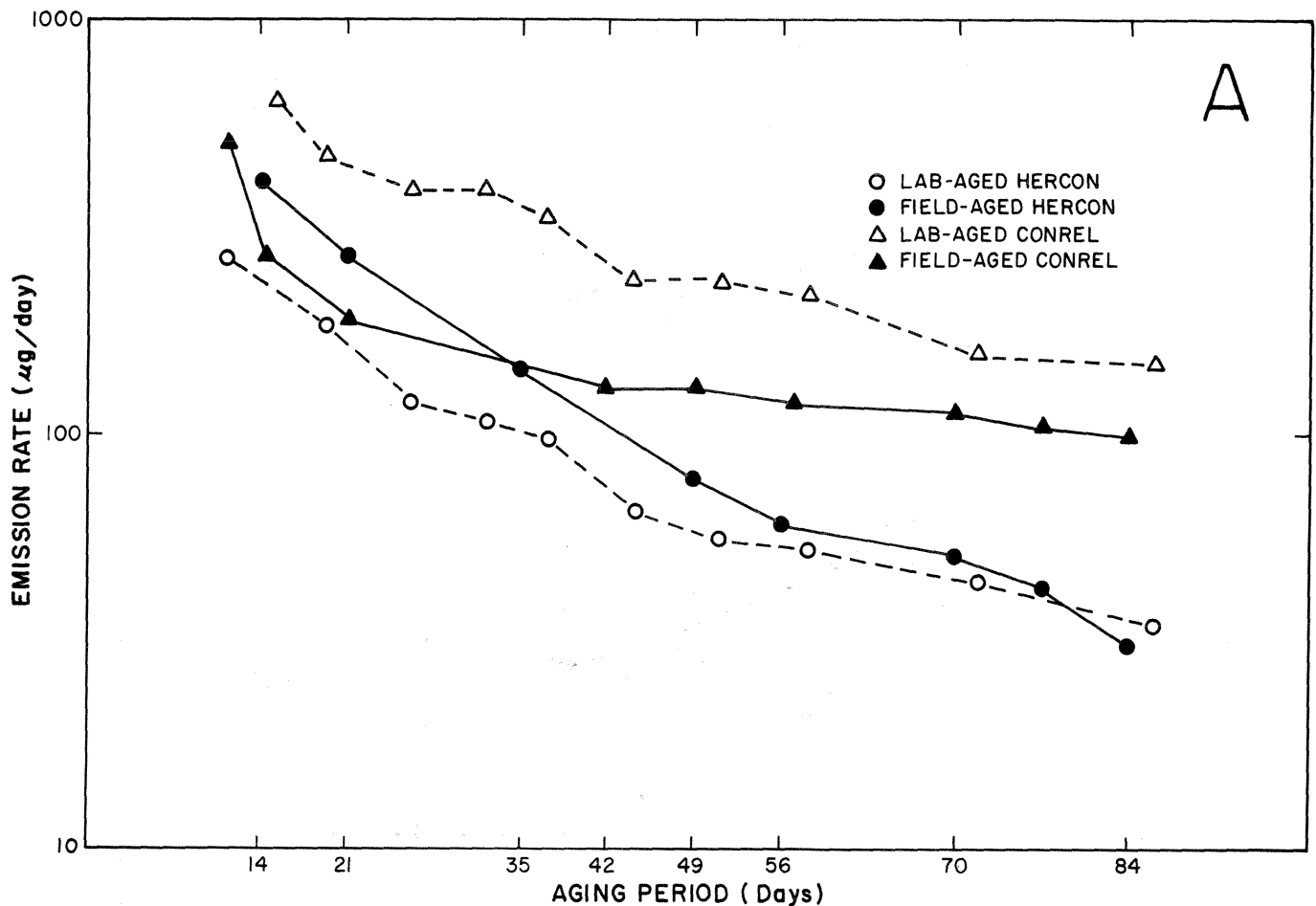
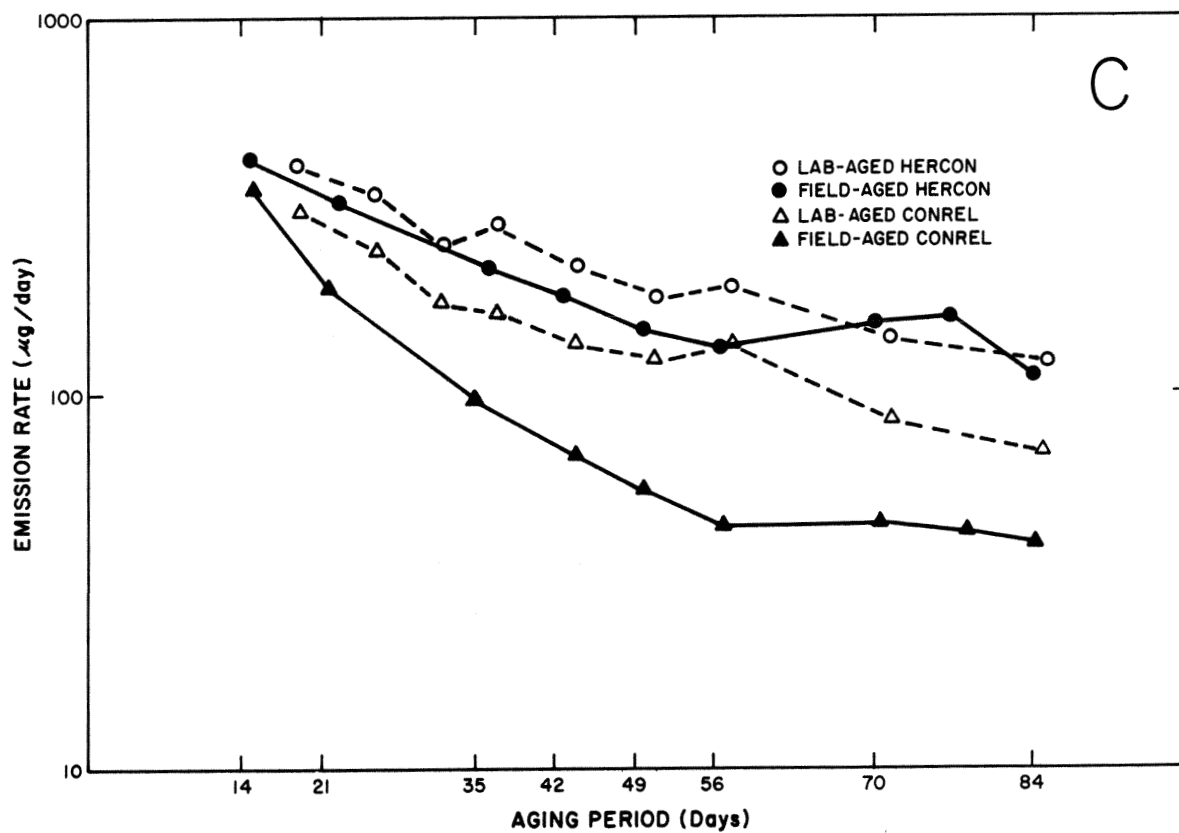
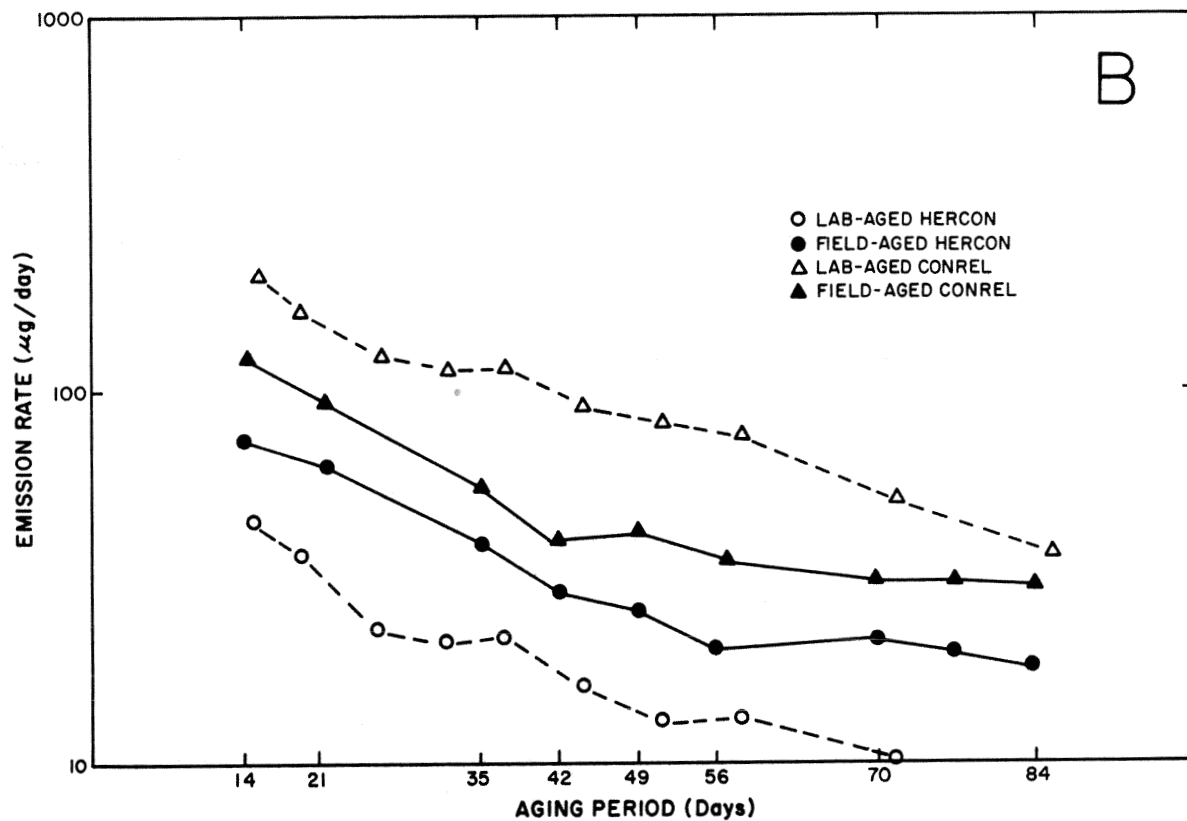


Figure 2.—Mean release rates of multilure components from laboratory- and field-aged Hercon and Conrel lures: (A) for 4-methyl-3-heptanol; (B) for α -multistriatin; (C) for α -cubebene (three lures of each type).



The lures differed not only in relative release rates of the three components but at 44 days (except for laboratory-aged Conrel for H and M), both lures also released doses well below those specified (400:100:800 $\mu\text{g/day}$ H:M:C). We expected this decrease but consider it only moderately important for two reasons. First, the average dose released from either lure can be manipulated easily by changing the size (Hercon) or number of fibers (Conrel). In fact, the specified rates were higher than needed for maximum beetle catch; we have deliberately specified high rates to ensure an adequate rate throughout the trapping period (90 days). Second, above a certain threshold the absolute dose emitted is much less important in attracting beetles than the ratio of components. Attractiveness of the pheromone blend depends heavily on the ratio in which components are emitted—especially the ratio of H to M (Lanier et al. 1977; Cuthbert and Peacock 1978). The lures differed significantly in this respect (Fig. 2, Table 3).

The average ratio in which H to M was emitted from field-aged Conrel increased slightly over time. Although the release rate ratio of H to M for Conrel lures was more stable than that for Hercon lures, the ratio from Hercon lures was closer to that specified at the time of manufacture (400:100:800). For example, the average ratio for field-aged Conrel lures was about 2.2 to 1 ($\frac{274}{126}$) at day 14 and 3.4 to 1 ($\frac{132}{38}$) after 44 days expo-

sure. The average ratio for field-aged Hercon lures was about 5.7 to 1 at 14 days and 3.8 to 1 at day 44 under the same conditions (Table 3). Even though Conrel lures generally had a higher, more stable release rate than Hercon, the ratio of H to M was closer to specifications for Hercon lures—and the Hercon lures caught 50 percent more beetles in concurrent suppression tests.³

However, some lures of either type are likely to have a low, suboptimal ratio of H to M (less than 2 to 1). Although the data show the average ratios usually were greater than 3 to 1, the standard errors indicate that some lures in the general lot would be expected to have much lower, less attractive ratios. Using actual data as an example, the average release rates from field-aged Conrel lures at 14 days indicate a 2.2 to 1 ratio of H to M (Table 3). But in some lures the output of H could be less than 240 ($274 - 35$), and the output of M could exceed 147 ($126 + 21$), which would result in a suboptimal ratio (1.6 to 1). The proportion of suboptimal lures cannot be estimated accurately from these data because the release rates of H and M for a given lure are not necessarily independent.

³The Conrel lures were redesigned to reduce the output of α -multistriatin. As a result, the attractiveness of Conrel lures equaled that of Hercon lures (Peacock, J. W., unpublished).

Conclusions

On the basis of these tests, we have no strong reason to assume that either the Hercon or Conrel lure is better than the other. Although both lures differed significantly in many particulars (dose, ratio, initial rates), overall release-rate characteristics were similar. After an initial burst, both types had relatively constant release rates, depleting at a rate of about 1 to 3 percent per day. The release rates from both types of lures only approximated the specified doses and ratios, but these can be (and were) easily adjusted in subsequent formulations. Most important, the ratio of components released from the lures was relatively constant over the life of the lures and at different temperatures. The measured variation among lures of the same type indicated that most lures emit an acceptable ratio of H to M, but also that some proportion of both lures emits a suboptimal ratio. Both lures were highly temperature-sensitive. And since field temperatures commonly differ by several degrees between locations and even from hour to hour, the differences in release rates due to temperature probably are larger than those due to daily degradation of lures or to the differences between and among the lures.

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Release rates of the three-component aggregation attractant of the smaller European elm bark beetle, *Scolytus multistriatus* (Marshall), from laboratory-aged and field-aged Conrel and Hercon dispensers were monitored for 85 days by GLC analysis of cold-trapped volatiles. Both dispensers had relatively low and constant rates of decay for all three attractant components after an initial burst in emission rates. Within limits, the ratio of components released remained constant over time and at various temperatures.

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Keywords: *Scolytus multistriatus*; pheromone; *Ulmus*; lures; volatiles

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