Volatile Compounds and Odors in Grain Sorghum Infested with Common Storage Insects

L. M. SEITZ[1,2] and D. B. SAUER[1]

Cereal Chem. 73(6):744–750

ABSTRACT

Lesser grain borer (*Rhyzopertha dominica*), red flour beetle (*Tribolium castaneum*), rice weevil (*Sitophilus oryzae*), saw-toothed grain beetle (*Oryzaephilus surinamensis*), and rusty grain beetle (*Cryptolestes ferrugineus*) were placed in sorghum (1 kg, 14% mc) for 5, 7, and 10 weeks at 27°C. Infested samples were analyzed for insect numbers, frass, odor, and volatiles. Volatiles from whole grain at 60°C were collected on Tenax absorbent, thermally desorbed, and analyzed by gas chromatography using infrared and mass detectors for component identification. Odor was assessed by sensory panels at the Federal Grain Inspection Service and in our own laboratory. Lesser grain borer caused severe off-odor and red flour beetle caused some off-odor. The other three insects caused little or no objectionable odor, even though infestation was heavy. High concentrations of 2-pentanol and the known aggregation pheromones, dominicalure 1 and 2, were consistently present in samples infested with lesser grain borer. These compounds were only a partial cause of the odor from lesser grain borer. Several metabolites from lesser grain borer not previously reported were tentatively identified. The presence and absence of some previously reported insect pheromones and metabolites are discussed.

It is generally known that insects often damage grain during storage, but little has been published on the effects of insect infestations on changes in grain odor during storage (Pederson 1992, Pomeranz 1992). According to Smith et al (1971), bread made from flour infested with *Tribolium* spp. or *Oryzaephilus surinamensis* (Lin.) exhibited undesirable taste. Quinone compounds were considered most responsible for the problem with *Tribolium* spp. The odor of grain infested with lesser grain borer has been described as "sweetish, musty" (Pederson 1992). From our experiences with infested grain and from discussions with grain handlers and inspectors, the odor of lesser grain borer-infested grain could also be described as acrid or urinous, with the latter being more applicable to grains with severe infestations. Mites, which are often included with insects in discussions of stored grains and cereal products, have been investigated for odors and volatiles in infested grains (Tuma et al 1990). Tridecane is a major compound produced by mites.

The objectives of this study were to: 1) determine which insects are most responsible for causing off-odors in grains, and 2) identify volatile compounds produced by insects that cause or are associated with undesirable odors. This information is needed for development of an objective method for detecting and classifying off-odors in grains.

MATERIALS AND METHODS

Test 1

Five common grain storage insects from cultures were placed on samples of sorghum (1 kg) from a local grain elevator (14% initial mc) for 5, 7, and 10 weeks at 27°C. Each sample was infested with one of the following species: lesser grain borer (LGB), *Rhyzopertha dominica* (F.); red flour beetle (RFB), *Tribolium castaneum* (Herbst); rice weevil (RW), *Sitophilus oryzae* (Lin.); saw-toothed grain beetle (STGB), *Oryzaephilus surinamensis*; and rusty grain beetle (RGB), *Cryptolestes ferrugineus* (Stephens).

Approximately 1,200 g of whole sorghum blended with ≈10 g of a mixture of flour and brewer's yeast (95:5) was placed in each of 15 1-gal jars fitted with screen-type lids. Three jars were infested with each species. For RW, LGB, and RFB inoculations, ≈40 insects were added to each jar. For STGB and RGB inoculations, ≈100 insects were added. The jars were stored in an incubator at 27°C. Moisture content was checked weekly. At 5, 7, and 10 weeks, one jar of each species was removed for determination of insect numbers and frass weights. Samples were frozen before testing for odor evaluation and volatiles analysis.

Odor was assessed by sensory panels at the Federal Grain Inspection Service (FGIS), Board of Appeals and Review, in Kansas City, MO, and in our own laboratory (GMRL). Odor intensity levels were rated from 0 to 3, with the latter representing highest off-odor intensity. Odor descriptors were also assigned (okay [normal], musty, sour, insect, or commercially objectionable foreign odor [COFO]).

Test 2

This test was similar to Test 1, except that 1) it was conducted only with LGB and RFB; 2) samples were stored 3, 5, and 7 weeks; and 3) there was a noninfested control for each storage period. About 1,200 g of sorghum plus flour and yeast (a different lot of sorghum than that used for the first test) was placed in each of 15 1-gal jars. Six jars each were infested with LGB, six with RFB, and three left uninfested. About 40 insects were added to each jar. Jars were then stored at 27°C. At 3, 5, and 7 weeks, one of the noninfested jars and two of the infested jars for each insect species were removed for determination of insect numbers and frass weights. Samples were frozen before testing for odors and volatiles.

Analysis of Volatiles

Volatile compounds in samples from both tests were determined. Each whole-grain sample (30 g) was placed in a 25-ml Tekmar sparger of a Tekmar purge and trap instrument (model LSC 2000) equipped with a sample heater (model 211005) and a capillary interface module (model 2530) (Tekmar Co., Cincinnati, OH). Each grain sample was preheated without gas flow at 60°C for 2 min, and then the volatiles from the heated sample were purged with helium at 40 ml/min onto a Tenax trap (0.29 g, 60–80

[1]U. S. Grain Marketing Research Laboratory, Grain Marketing and Production Research Center. USDA, Agricultural Research Service, 1515 College Avenue, Manhattan, KS 66502. Mention of firm names or trade products does not constitute endorsement by the U.S. Department of Agriculture over others not mentioned.
[2]Corresponding author. E-mail: larry@crunch.usgmrl.ksu.edu

Publication no. C-1996-1004-02R.

mesh) (Tekmar). After a 10-min sample purge time, a 6–10 min dry purge was performed to remove excess moisture from the Tenax trap. The trap was preheated at 175°C and the volatiles were desorbed at 200°C for 4 min. With the capillary interface module, the desorbed volatiles were cryofocused at –130°C (liquid nitrogen) and the cryofocused zone was heated at 200°C for 0.75 min before initiation of the analytical run.

A model 5890 series II gas chromatograph coupled with a model 5965B FTIR detector (IRD), and a model 5970 mass selective detector (MSD), all from Hewlett Packard Co. (Palo Alto, CA), were used to analyze volatiles collected from the grain samples. A Supelcowax 10 column (30 m × 0.32 mm, i.d., 0.25 μm film thickness) from Supelco Inc. (Bellefonte, PA) was used for analysis. Column head pressure was 90.2 kPa (13.1 psi) at 50°C, which gave a helium flow rate of about 2.5 ml/min. Oven temperature was held at 50°C initially for 2 min, and then increased to 230°C at a rate of 10°C/min. The temperature of the gas chromatography (GC) injector zone under the capillary interface module was maintained at 200°C. The effluent from the GC column first passed through the IRD and then into the MSD. The transfer line and flow cell temperatures of the IRD were maintained at 200°C. MSD conditions were: direct transfer line temperature, 230°C; ion source temperature, 250°C; ionization voltage, 70 eV; mass range, mass/charge 33–230 a.m.u.; scan rate, 1.57 scans/sec; and electron multiplier voltage, 2,200. Compounds were identified by computer matching of experimental infrared spectra and mass spectra of compounds with standard spectra in two IR vapor phase libraries (HP 59963A EPA and HP 59964A flavors and fragrances) and in the HP 59943B Wiley PBM MS database, respectively.

RESULTS

Test 1

Insect numbers and odor scores for infested sorghum samples stored 5, 7, and 10 weeks are shown in Table I. Off-odor scores, especially in the "insect" category and those assigned by FGIS panelists, were highest for samples infested with LGB and RFB. Samples infested with RW, STGB, and RGB had low off-odor scores considering the high insect counts at 7 and 10 weeks.

Total ion chromatograms (from the MSD) for samples stored 10 weeks are shown in Figure 1. Most of the peaks in the chromatograms are volatile components in the grain itself.

TABLE I
Number of Insects in and Odor Evaluation of Sorghum Infested for 5, 7, and 10 Weeks with Five Species of Common Grain Storage Insects

			Odor Scores[a]							
			GMRL				FGIS[b]			
Treatment[c]	No. of Insects[d]	Frass[e]	Musty	Insect	Sour	COFO	Musty	Insect	COFO	Grade[f]
RGB-5	100		0.8		0.1		0.2			OK
RGB-7	270		0.2				0.4			OK
RGB-10	350	0.9		0.3						OK
STGB-5	1,721		0.4	0.7			0.4		0.2	OK
STGB-7	3,935		0.4	0.4			0.2	0.6		OK
STGB-10	6,500	4.9								OK
RFB-5	2,274		0.6	0.8			0.4	1.2		SG
RFB-7	1,740			0.4		0.6		2.4		SG
RFB-10	2,100	14.0		0.3		0.8		1.6		SG
RW-5	140		0.5	0.2	0.2		0.2	0.2	0.2	OK
RW-7	1,550		0.4	0.8		0.2	0.6		0.2	OK
RW-10	1,600	10.8		1.5	0.3			0.6		OK
LGB-5	46		0.6	0.4			0.2		0.4	OK
LGB-7	1,292		0.2	1.8	1.0			2.2	0.6	SG
LGB-10	1,300	51.2		2.0	1.5	0.8		3.0		SG

[a] Odor intensities (0–3 scale) are means of assessments from six panelists at Federal Grain Inspection Service (FGIS) and four panelists at Grain Marketing Research Laboratory (GMRL). COFO= commercially objectionable foreign odor.
[b] FGIS panelists did not assign sour odor to any of the samples.
[c] RGB = rusty grain beetle, STGB = saw-toothed grain beetle, RFB = red flour beetle, RW = rice weevil, LGB = lesser grain borer.
[d] Includes insects in all stages of development.
[e] Grams of frass per kilogram of infested grain.
[f] OK = normal odor, SG = sample was downgraded to "sample grade" because of odor.

TABLE II
Number of Insects in and Odor Evaluation of Samples Infested for 3, 5, and 7 Weeks with Red Flour Beetle (RFB) and Lesser Grain Borer (LGB)

			Odor Scores[a]						
			GMRL			FGIS			
Treatment	Weeks	No. of Insects[b]	Musty	Insect	COFO	Musty	Insect	COFO	Grade[c]
None	3	0				0.4			OK
None	5	0							OK
None	7	0						0.2	OK
RFB	3	490	0.5	0.5			0.4		OK
RFB	5	1,134	0.5	1.1			0.5	0.9	SG
RFB	7	1,436	0.5	1.0			0.4	0.2	OK
LGB	3	36	0.1	0.5		0.1			OK
LGB	5	40		0.5				0.8	OK
LGB	7	167		0.9	0.5	0.1		0.1	OK

[a] Odor intensities (0–3 scale) are means of assessments from six panelists at Federal Grain Inspection Service (FGIS) and four panelists at Grain Marketing Research Laboratory (GMRL). COFO= commercially objectionable foreign odor.
[b] Includes insects in all stages of development.
[c] OK = normal odor, SG = sample was downgraded to "sample grade" because of odor.

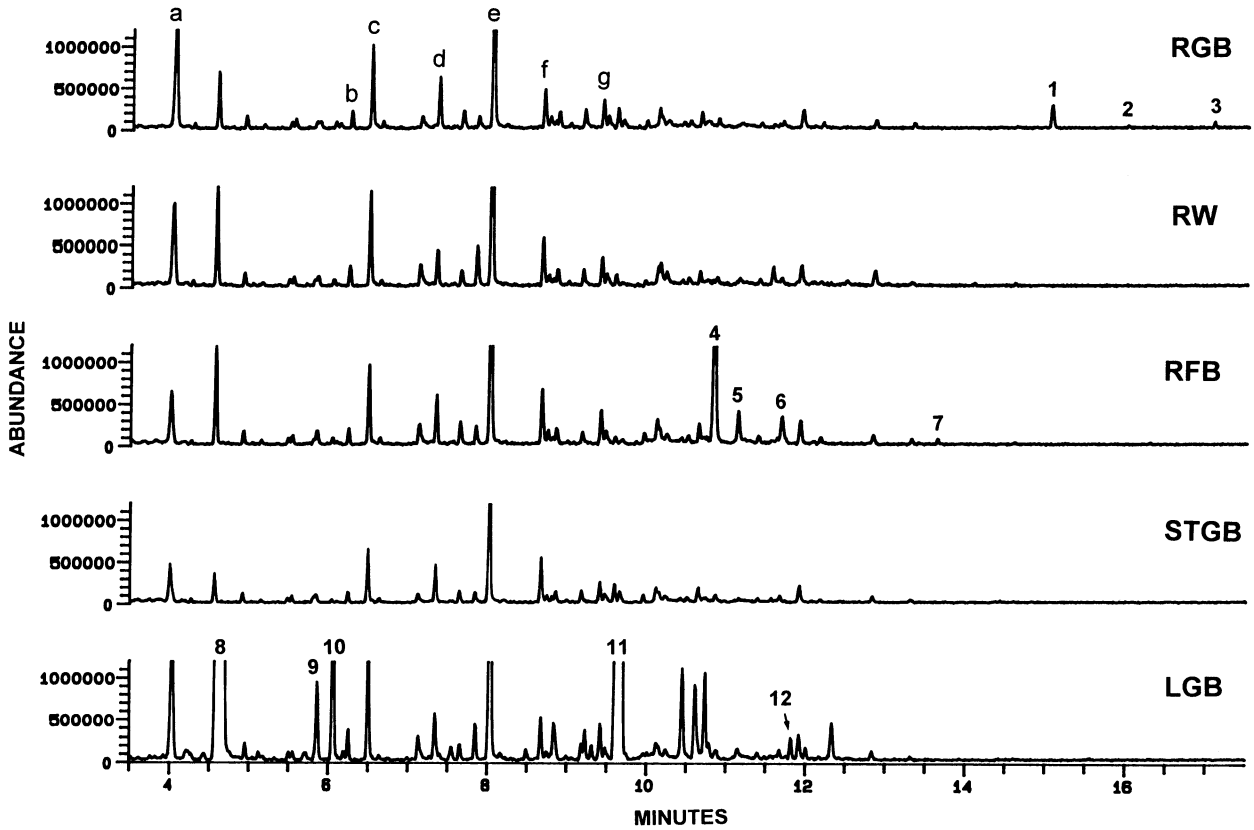

Fig. 1. Total ion chromatograms for samples infested with insects for 10 weeks in Test 1. Chromatograms are labeled by species abbreviations: rusty grain beetle (RGB), rice weevil (RW), red flour beetle (RFB), saw-toothed grain beetle (STGB), lesser grain borer (LGB). Peaks representing insect pheromones and metabolites are identified by number: peaks 1, 2, and 3 are macrolides I, II, and III produced by RGB; peaks 4, 5, 6, and 7 are 1-pentadecene, an apparent diene, 4,8-dimethyldecanal, and another apparent diene, respectively, from RFB; peaks 8, 9, 10, 11, and 12 are 2-pentanol, 3-methyl-2-pentanol, 2-hexanol, dominicalure 1 and 2, and 1-methylbutyl (*E*)-2-methyl-2-hexenoate (the apparent homolog of dominicalure 1 as described in the text), respectively. Some compounds normally in sorghum are marked by letters on the upper chromatogram: a = hexanal, b = 2-pentylfuran, c = 1-pentanol, d = tridecane, e = 1-hexanol, f = nonanal, g = 1-octen-3-ol.

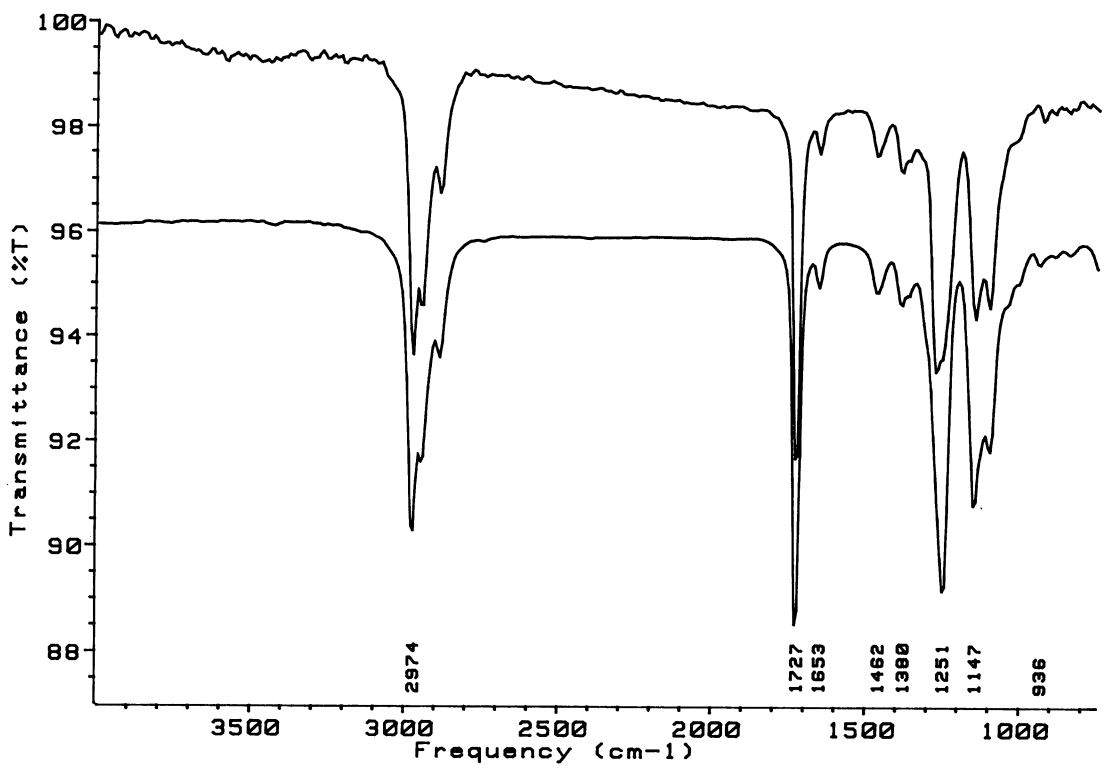

Fig. 2. Vapor phase infrared spectra of dominicalure 1 (upper curve) and apparent homolog (lower curve).

All samples infested with LGB had major components eluting at 9.59 and 9.66 min that exhibited MS and IR spectra consistent with dominicalure 1[(*S*)-(+)-1-methylbutyl (*E*)-2-methyl-2-pentenoate] and dominicalure 2 [(S)-(+)-1-methylbutyl (E)-2,4-dimethyl-2-pentenoate], respectively. These compounds have been identified as aggregation pheromones in male LGB (Williams et al 1981). The major peak at 4.55 min in the LGB sample (Fig. 1) was 2-pentanol, which was quite prevalent even at week 5, and its concentration increased greatly in the 5–7 and 7–10-week intervals. Several other compounds, including 2-hexanol, 3-methyl-2-pentanol, 2-pentanone, and 3-methyl-2-pentanone, were observed in the LGB samples at much lower concentrations than that for 2-pentanol, but they increased greatly from 5 to 10 weeks in a manner similar to that of 2-pentanol. Mass and IR spectra of components giving fairly broad peaks at 15.42 and 15.5 min in the LGB sample infested for 10 weeks, indicate the presence of an unsaturated acid similar to 2-methyl-2-pentenoic or 2-methyl-2-hexenoic acid.

Many more components were produced by LGB than any of the other insects in this test. In addition to components mentioned above, numerous minor components were observed (Fig. 1) that gave infrared and mass spectra which suggested that they were esters, possibly related to the dominicalures. Complete identification of all apparent LGB metabolites will be presented in a subsequent publication, so only spectral data of the component eluting at 10.71 min will be presented here. This component appears to be 1-methylbutyl (*E*)-2-methyl-2-hexenoate, a homolog of dominicalure 1. The IR spectrum of this component is similar to that for dominicalure 1 (Fig. 2). The apparent low intensity molecular ion in the MS for the 10.71 min component was *m/z* 198 compared to *m/z* 184 for dominicalure 1 (Fig. 3), a mass difference of 14, equivalent to one methylene group. The major masses in the spectrum of this component are 14 higher than those in the spectrum from dominicalure 1(*m/z* 111 compared to 97, and 129 compared to 115) (Fig. 3). These intense ions are from the acid moiety of the esters. The IR spectra of dominicalures 1 and 2 were quite similar, with dominicalure 2 showing absorbances at 1,304 and 1,009 cm^{-1} that were only minor shoulders in dominicalure 1.

The large peak at 10.84 min in the sample infested with RFB (Fig. 1) was due to 1-pentadecene, which is known to be produced by RFB (Howard 1987). The RFB aggregation pheromone, 4,8-dimethyldecanal (Suzuki 1980), was detected at 11.69 min. Both compounds were present in all of the RFB samples with concentrations increasing from 5 to 10 weeks. Infrared and mass spectra were fully consistent with these compounds. Two other compounds were detected at 11.14 and 13.65 min in the 10-week samples that appear to be dienes produced by RFB (Howard 1987). There was no MS or IR evidence in any of the RFB samples for the compounds paeonol, 2′-hydroxy-4-methoxypropiophenone, and quinones, which have been reported as metabolites of RFB (Howard et al 1986).

Macrolide RGB pheromones I, II, and III reported by Wong et al (1983) were found in the 5, 7,and 10 week RGB samples (Fig. 1). From extracted ion chromatograms, there was slight evidence for macrolide IV. Infrared and mass spectra, relative ratio of the pheromones, and their retention times in chromatograms were consistent with information reported by Wong et al (1983).

Some of the same or similar macrolides were reported as pheromones from STGB (Pierce et al 1985, 1989; White et al 1989), but we did not find them in any of our STGB samples, even though numbers of STGB were considerably higher than numbers of RGB insects (Table I). Greater insect activity in the STGB samples compared to RGB samples was also indicated by higher frass contents in the STGB samples (Table I). Others have also observed that pheromone production by RGB is more copious than that by STGB (H. D. Pierce, *personal communication*; Pierce et al 1986) . The unsaturated alcohol 1-octen-3-ol was recently reported as an aggregation pheromone produced by STGB (Pierce et al 1989). Any 1-octen-3-ol produced by the insects may have been small in comparison to amount of this compound in the grain itself. The concentration of 1-octen-3-ol was nearly constant during the entire 5–10 week storage period, and its concentrations in the STGB samples were not higher than the concentrations in grain infested with any of the other insects.

The RW aggregation pheromone sitophilure, (R^*,S^*)-5-hydroxy-4-methyl-3-heptanone, reported by Phillips et al (1985) was not found in any of the samples inoculated with RW. No other compounds were observed in the RW samples that could be associated with the RW insects.

Test 2

Insect numbers and odor scores for sorghum stored 3, 5, and 7 weeks are shown in Table II. In contrast to results from Test 1, only RFB samples stored 5 weeks were graded down by FGIS inspectors. The reduced off-odor scores may be due to reduced insect numbers compared to Test 1. It is not clear why this test gave reduced insect numbers. The sorghum used for this test had considerable concentrations of 2,3-butanediol and 3-hydroxy-2-butanone (acetoin) which were not prevalent in the sorghum used for the first test. In Figures 3 and 4, the diastereomers of 2,3-butanediol appear at 10.25 and 10.67 min, and the 3-hydroxy-2-butanone appears at 7.08 min. We do not know, however, whether these compounds affected the insect development in the grain.

Dominicalure 1 and 2 were again found in all samples infested with LGB. A total ion chromatogram for an LGB sample stored 3 weeks is shown in Figure 4. Dominicalure 1 and 2 were eluted at 9.38 and 9.34 min, respectively. The level of 2-pentanol (at 4.72 min) was low at 3 weeks, but higher at 5 and 7 weeks. It tended to increase greatly as storage time and numbers of LGB increased. The peak at 10.31 min, adjacent to one of the diastereomers of 2,3-butanediol, is the homolog of dominicalure (see above).

Like the results from test 1, the RFB aggregation pheromone, 4,8-dimethyldecanal, and 1-pentadecene were present in all samples infested with RFB (Fig. 5). Also, as in Test 1, the RFB metabolites paeonol, 2′-hydroxy-4-methoxypropiophenone, and the quinones described by Howard et al (1986) were not found in any of the RFB samples.

DISCUSSION

In the first test, LGB produced more severe off-odor than any of the other four species. RFB caused moderate to slight off-odor that was distinctly different from the LGB odor. However, RW, STGB, and RGB produced little or no undesirable odors, even in severely infested grain.

Relatively high concentrations of 2-pentanol and the known LGB pheromones (dominicalure 1 and 2) were observed in samples infested with LGB, but sensory analyses of spiked samples showed that these compounds were not the main cause of off-odor. When these compounds were added to sorghum samples at concentrations of 0.9 and 3.8 ppm for each compound, odor of samples with the added compounds were not notably different from control samples of the same sorghum, as judged by five inspectors from FGIS and four panelists from GMRL.

Production of LGB metabolites was much more complex than was previously indicated in the literature. It is not clear whether the 2-pentanol found in the samples was produced directly by the borers or came from hydrolysis of the dominicalures. In the 10-week sample, which had the most intense off-odor and the highest concentration of 2-pentanol, there was evidence for free unsaturated acid that could have come from hydrolysis of the dominicalures or possibly been produced directly by the insect. The amount of the free acid could have been greatly underestimated by the headspace analysis procedure, because the acid may adhere much more tightly to the grain than an alcohol such as 2-pentanol.

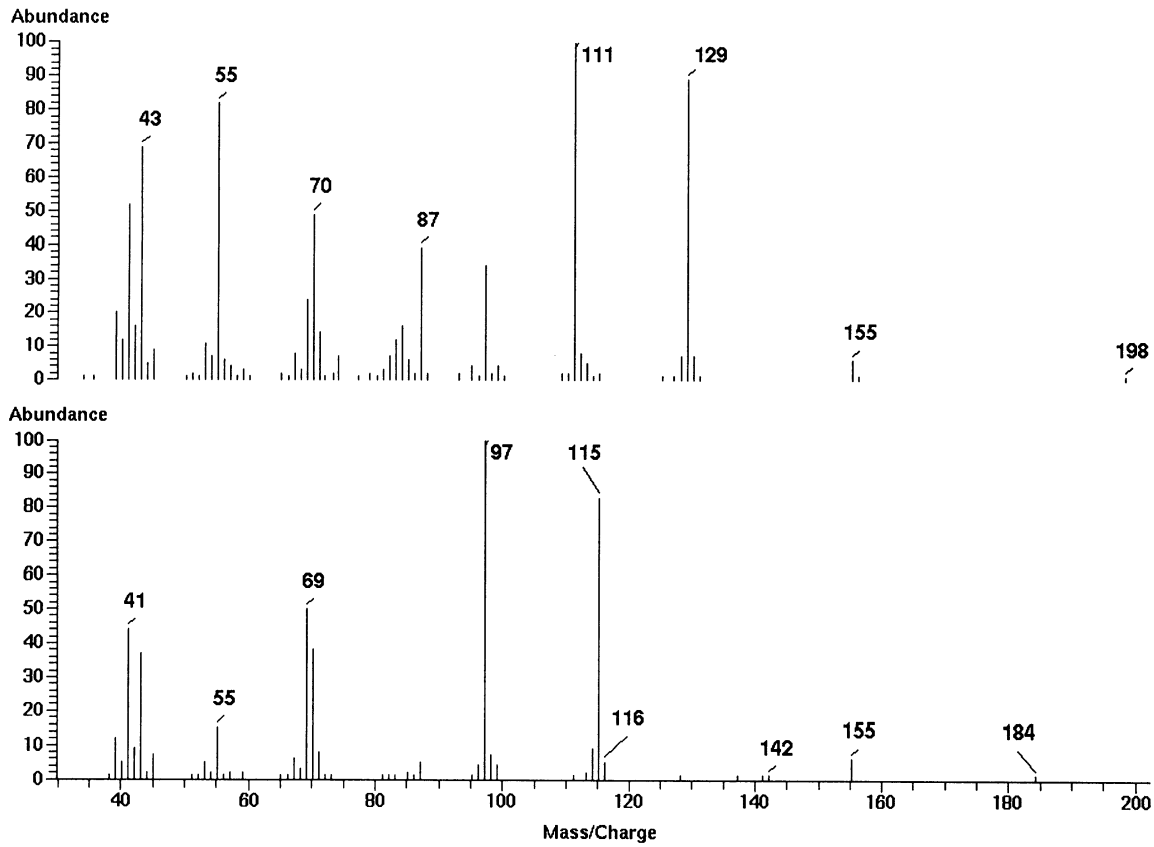

Fig. 3. Mass spectra of dominicalure 1 (lower spectrum) and apparent homolog (upper spectrum).

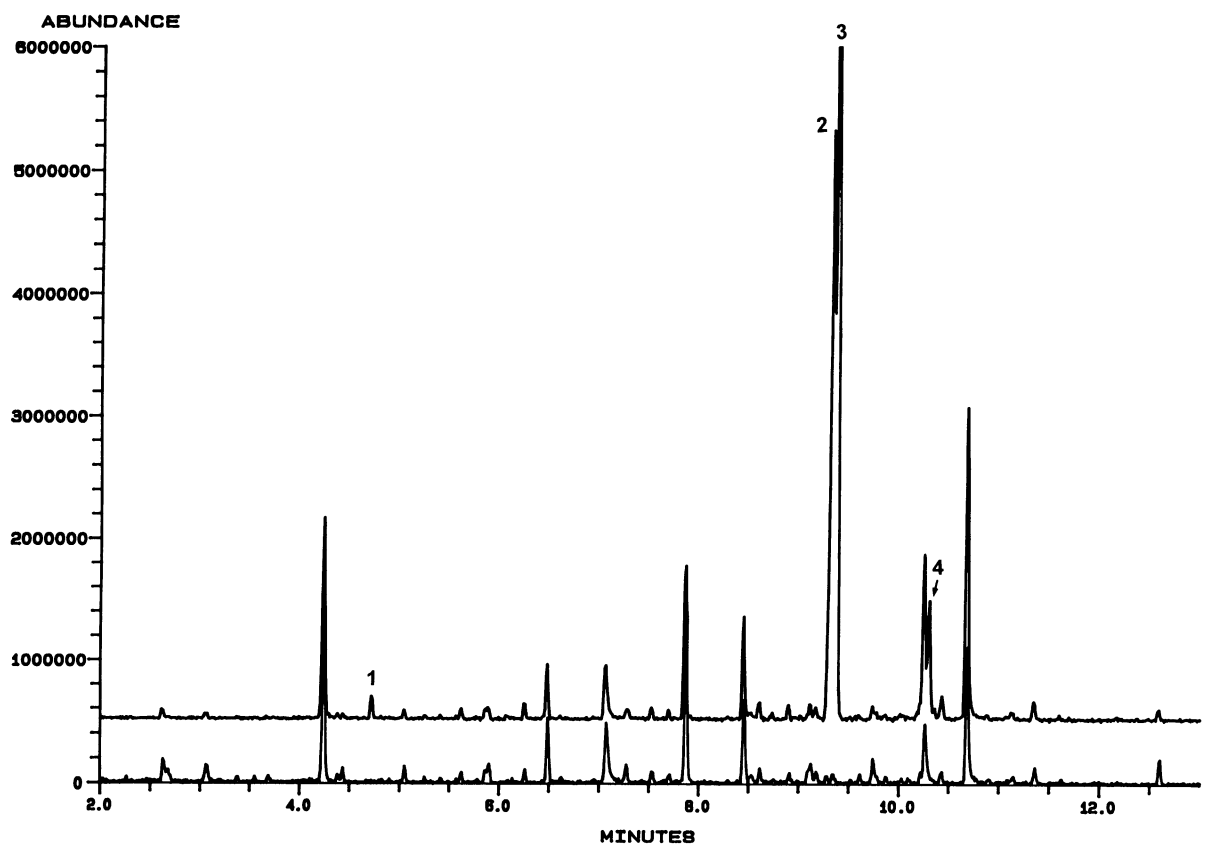

Fig. 4. Total ion chromatograms from lesser grain borer (LGB)-infested (upper) and noninfested (lower) sorghum stored three weeks in Test 2. Numbered peaks are: 1 = 2-pentanol; 2 = dominicalure 2; 3 = dominicalure 1; and 4 = apparent homolog of dominicalure 1.

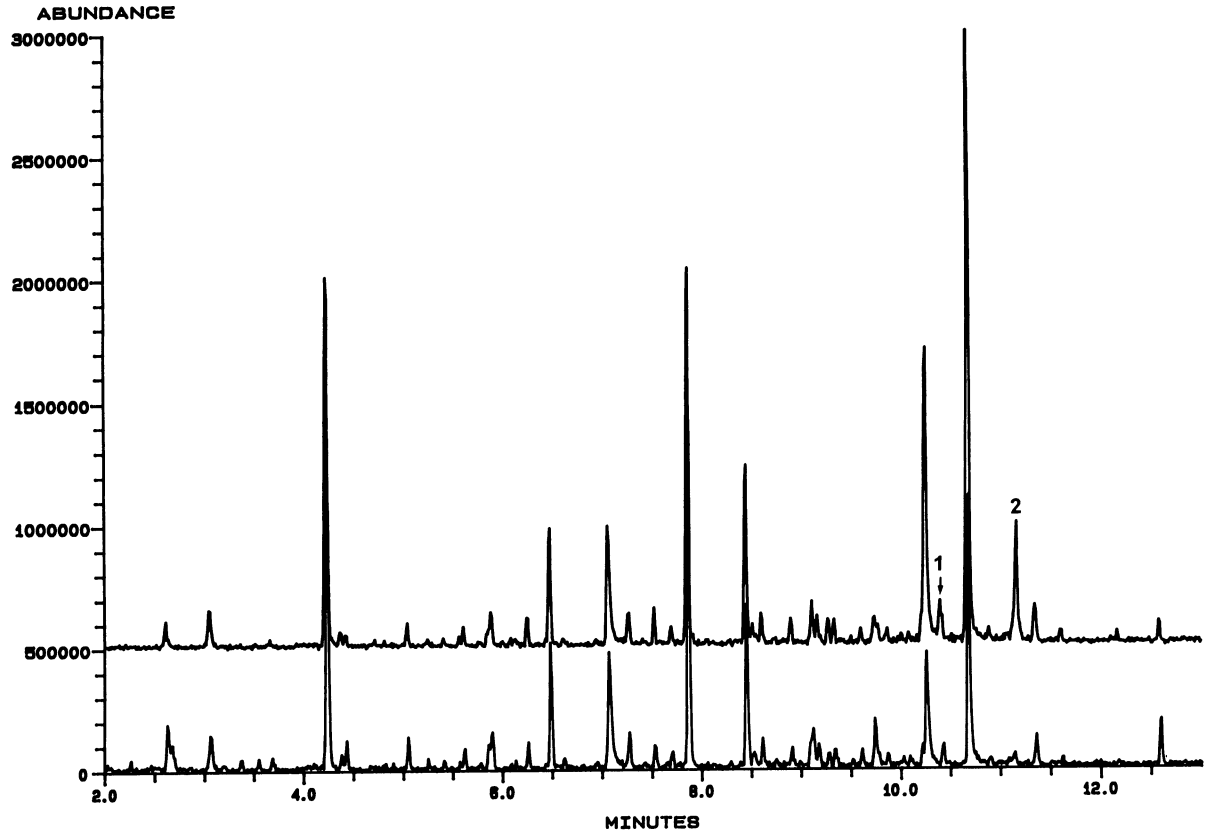

Fig. 5. Total ion chromatograms from red flour beetle (RFB)-infested (upper) and noninfested (lower) sorghum stored three weeks in Test 2. Numbered peaks are: 1 = 1-pentadecene; 2 = 4,8-dimethyldecanal.

It is possible that any unsaturated free acids that may be present, or that some of the other unidentified metabolites mentioned above are contributing to the off-odor associated with the LGB insects.

Because the RGB samples did not have off-odors, the observed macrolide RGB metabolites were apparently not odorous, at least not at the low concentrations in our samples. To some individuals, these macrolides have a pleasant aroma with a cherry note (H. D. Pierce, *personal communication*). As discussed above, some known insect pheromones were not found in this study. Either these compounds were produced at such low levels that they could not be detected, or the analysis procedure was not appropriate for detecting them. The latter case could possibly explain why the rather polar quinone compounds known to be produced by RFB were not detected. Alternatively, the RFB may not have been disturbed enough to cause production of the quinone compounds.

In commercial samples received from official inspectors, we have observed some of the insect pheromones and metabolites mentioned above in wheat, corn, and sorghum samples that had insect-type odors (Seitz and Sauer 1991, Seitz and Sauer 1994). Samples with strong LGB-type of odor contained elevated concentrations of 2-pentanol. If 2-pentanol concentration was high, lower levels of 2-pentanone and 2-hexanol were usually present, which was consistent with the observations in this study. 1-Pentadecene was associated with insect odors in commercial samples, but 4,8-dimethyldecanal was not found. Two compounds found to be associated with insects in commercial grain samples, linalool and an unidentified sesquiterpene (Seitz and Sauer 1994), were not observed in this study.

ACKNOWLEDGMENTS

We thank Harold Mohr, Kevin Fay, and Leon Hendricks for technical assistance. We gratefully acknowledge Ralph Howard for helpful discussions on insect metabolites.

LITERATURE CITED

HOWARD, R. W. 1987. Chemosystematic studies of the Triboliini (Coleoptera: Tenebrionidae): Phylogenetic Inferences from the defensive chemicals of eight *Tribolium* spp., *Palorus ratzeburgi* (Wissmann), and *Latheticus oryzae* Waterhouse. Ann. Entomol. Soc. Am. 80:398-405.

HOWARD, R. W., JURENKA, R. A., and BLOMQUIST, G. J. 1986. Prostaglandin synthetase inhibitors in the defensive secretion of the red flour beetle Tribolium Castaneum (Herbst) (Coleoptera: Tenebrionidae). Insect Biochem. 16:757-760.

PEDERSON, J. R. 1992. Insects: Identification, damage, and detection. Pages 435-489 in: Storage of Cereal Grains and Their Products. D. B. Sauer, ed. Am. Assoc. Cereal Chem: St. Paul, MN.

PHILLIPS, J. K., WALGENBACH, C. A., KLEIN, J. A., BURKHOLDER, W. E., SCHMUFF, N. R., and FALES, H. M. 1985. (R^*,S^*)-5-Hydroxy-4-methyl-3-heptanone: Male-produced aggregation pheromone of *Sitophilus oryzae* (L.) and *S. zeamais* Motsch. J. Chem. Ecol. 11:1263-1274.

PIERCE, A. M., PIERCE, H. D., Jr., BORDEN, J. H., and OEHLSCHLAGER, A. C. 1989. Production dynamics of cucujolide pheromones and identification of 1-octen-3-ol as a new aggregation pheromone for *Oryzaephilus surinamensis* and *O. mercator* (Coleoptera: Cucujidae). Environ. Entomol. 18:747-755.

PIERCE, A. M., PIERCE, H. D., Jr., BORDEN, J. H., and OEHLSCHLAGER, A. C. 1986. Enhanced production of aggregation pheromones in four stored-product coleopterans feeding on methoprene-treated oats. Experientia 42:164-165.

PIERCE, A. M., PIERCE, H. D., Jr., OEHLSCHLAGER, A. C., and BORDEN, J. H. 1985. Macrolide aggregation pheromones in *Oryzaephilus surinamensis* and *Oryzaephilus mercator* (Coleoptera: Cucujidae). J. Agric. Food Chem. 33:848-852.

POMERANZ, Y. 1992. Biochemical, functional, and nutritive changes during storage. Pages 55-141 in: Storage of Cereal Grains and Their Products. D. B. Sauer, ed. Am. Assoc. Cereal Chem: St. Paul, MN.

SEITZ, L. M., and SAUER, D. B. 1991. Volatile compounds associated with objectionable odors in commercial grain samples. Cereal Foods World 36:707.

SEITZ, L. M. and SAUER, D. B. 1994. Detection of specific compounds that indicate off-odors in grains. Cereal Foods World 39:605.

SMITH, L. W., PRATT, J. J., NII, I., and UMINA, A. P. 1971. Baking and taste properties of bread made from hard wheat flour infested with species of *Tribolium*, *Tenebrio*, *Trogoderma*, and *Oryzaephilus*. J. Stored Prod. Res. 6:307-316.

SUZUKI, T. 1980. 4,8-Dimethyldecanal: The aggregation pheromone of the flour beetles, *Tribolium castaneum* and *T. confusum* (Coleoptera: Tenebrionidae). Agric. Biol. Chem. 44:2519-2520.

TUMA, D., SINHA, R. N., MUIR, W. E., and ABRAMSON, D. 1990. Odor volatiles associated with mite-infested, bin-stored wheat. J. Chem. Ecol. 16:713-724.

WILLIAMS, H. J., SILVERSTEIN, W. E., BURKHOLDER, W. E., and KHORRAMSHAHI, A. 1981. Dominicalure 1 and 2: Components of aggregation pheromone from male lesser grain borer *Rhyzopertha dominica* (F.) (Coleoptera: Bostrichidae). J. Chem. Ecol. 7:759-780.

WHITE, P. R., CHAMBERS, J., WALTER, C. M., WILKINS, J. P. G., and MILLAR, J. G. 1989. Saw-toothed grain beetle, *Oryzaephilus surinamensis* (L.) (Coleoptera: Silvanidae): Collection. identification, and bioassay of attractive volatiles from beetles and oats. J. Chem. Ecol. 15:999-1013.

WONG, J. W., VERIGIN, V., OEHLSCHLAGER, A. C., BORDEN, J. H., PIERCE, H. D., Jr, PIERCE, A. M., and CHONG, L. 1983. Isolation and identification of two macrolide pheromones from the frass of *Cryptolestes ferrugineus* (Coleoptera: Cucujidae). J. Chem. Ecol. 9:451-474.

[Received March 26, 1996. Accepted July 23, 1996.]