on treatment with ice water gave a solid. It was recrystallized from 100 mL of isopropyl ether to give 3.6 g (50%) of product: mp 72–75.5 °C. IR (KBr) 3390 (NH), 2985, 1706 (C=O) cm^{-1}; NMR ($CDCl_3$) δ 1.7 [s, 6 H, $>C(CH_3)_2$], 2.9 (d, NCH_3) and 3.22 (m, 4 H, $—SCH_2CH_2S—$), 6.32 (br, s, 1 H, NH). Anal. Calcd for $C_8H_{14}N_2O_2S_2$: C, 41.02; H, 6.03. Found: C, 40.95; H, 6.33.

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Registry No. 1, 20599-47-7; 2, 59375-77-8; 3, 625-48-9; 4, 18942-89-7; 5, 59375-83-6; 6, 55391-27-0; 7, 55391-28-1; 8, 93502-98-8; 9, 93502-99-9; 10, 59375-80-3; 11, 59375-65-4; 12, 59375-66-5; 13, 59375-61-0; 14, 59375-67-6; 15, 93503-00-5; 16, 55391-34-9; 17, 59375-71-2; 18, 59375-53-0; 19, 59379-68-9; 20, 59379-67-8; 21, 60029-68-7; 22, 93503-05-0; 23, 59375-72-3; 24, 59375-74-5; 25, 59375-57-4; 26, 59375-58-5; 27, 59375-56-3; 28, 93503-01-6; 29, 93503-02-7; 30, 93503-03-8; 31, 59375-69-8; 32, 59375-54-1; 33, 59375-70-1; 34, 59375-68-7; 35, 59375-82-5; 36, 59375-62-1; methanedithiol, 6725-64-0; 2-acetoxy-1-nitropropane, 3156-76-1; 2,2,5-trimethyl-4-oximino-1,3-dithiolane, 93503-04-9; 2,2-dimercaptopropane, 1687-47-4; ethanediol, 107-21-1; 2,2-propanedithiol, 1687-47-4; disodium *cis*-ethenedithiolate, 17934-70-2; dimercaptomaleonitrile, 41999-83-1.

Supplementary Material Available: Experimental details for compounds 3–7, 9–12, 21, 25–27, and 35–38 and Table VI listing the physical properties of compounds 17, 19, 20, 22–24, 27–34 (11 pages). Ordering information is given on any current masthead page.

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Volatile Components of Wheat Leaves (and Stems): Possible Insect Attractants

Ron G. Buttery,* Cheng-ji Xu,[1] and Louisa C. Ling

The volatile compounds associated with the leaves (and stems) of the wheat plant were isolated by using Tenax adsorbent trapping and analyzed by capillary GC–MS. The amount of volatiles isolated was of the order of 10–50 ppb of the leaves (and stems). A total of 25 volatile compounds were identified. The major volatile compounds include (*Z*)-3-hexenyl acetate, (*Z*)-3-hexanol, (*E*)-β-ocimene, (*E*)-2-hexenal, and caryophyllene. Unusual compounds include α-copaene, α-farnesene, and linalool oxides.

Some of the authors had previously studied the volatiles of several other major agricultural crops including red clover, corn, and oats [cf. Buttery et al. (1982, 1984) and Buttery and Ling (1984)]. The present work reports a similar study on the volatiles of whole, essentially intact, wheat leaves (and stems).

A study on the volatiles of macerated wheat leaves was recently reported by Hamilton-Kemp and Andersen (1984). The compounds identified by those authors, though, were typical enzyme-catalyzed oxidation products and quite different from the compounds found in undamaged intact leaves (and stems).

As with the volatiles of other plants, it is felt that a knowledge of the volatiles of wheat leaves may be useful in the understanding (and the eventual control) of the attraction of insect pests to wheat and other cereal crops.

Western Regional Research Center, Agricultural Research Service, U.S. Department of Agriculture, Albany, California 94710.
[1]Visiting scientist from Chengdu Institute of Biology, Chinese Academy of Sciences, Chengdu, Sichuan, China.

EXPERIMENTAL SECTION

Materials. Wheat (*Triticum aestivum*, Nebraska HRW, Scout 66) was grown during 1984 on experimental fields in Berkeley, CA. The leaves (and stems) were harvested when the plant were ca. 15–20 cm high by cutting the whole plant off near the base, just above the roots, with a sharp knife. Fresh lots were planted at intervals over about 6 months, and a total of six different batches of samples was obtained for analysis. With plants of this size the stem consists mostly of folded leaves.

Isolation of Volatiles Using Tenax Traps. The method used was essentially the same as that described previously for corn (Buttery and Ling, 1984). The freshly obtained whole leaves (with stems; 500 g) were placed in a 12-L flask. Care was taken not to damage the leaves (and stems), and they were placed in the flask within an hour after harvesting. Air drawn from outside the laboratory (and purified by passage through activated charcoal) was

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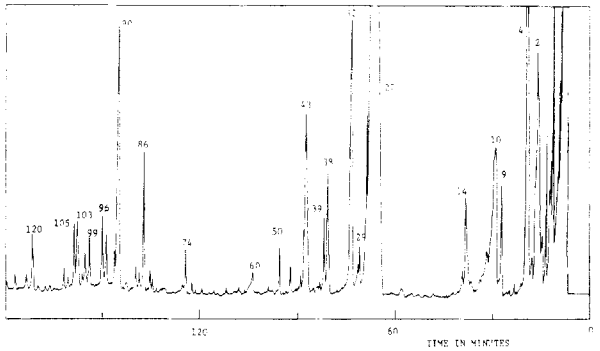

Figure 1. Capillary GLC analysis of Tenax-trapped wheat leaf (and stem) volatiles using the Silicone OV-3 coated, 150 m × 0.66 i.d. Pyrex capillary described in the text.

led into the flask via a Teflon tube. The air passed over the leaves and left the flask through a Tenax trap (10 g; 14 × 2.2 cm). The flow of air was 1 L/min and was continued for 24 h. The trapped volatiles were eluted from the trap with freshly distilled diethyl ether (containing ca. 1 ppm of Ethyl Antioxidant 330). The extracts from two isolations were combined and then concentrated to a small volume (ca. 5 μL). The combined concentrate was used in a single (splitless) injection for the capillary GC–MS analysis.

Capillary Gas–Liquid Chromatography–Mass Spectral (GC–MS) Analysis. Two main capillary GLC columns were used at different times. One was a 150 m long by 0.66 mm i.d. Pyrex glass capillary, wall coated with Carbowax 20-M. The other column was a Pyrex capillary of the same dimensions but wall coated with Silicone OV-3 (Figure 1). The Carbowax capillary was held at 60 °C for the first 40 min after the injection and then temperature programmed at 1 °C/min to 170 °C and held at this upper limit. The Silicone capillary was temperature programmed at 1 °C/min from 20 to 170 °C and held at the upper limit. Separate GC–MS runs were made on the concentrates from six different batches of wheat leaves (and stems). Coupling to the mass spectrometer (a modified Consolidated 21-620 cycloidal type instrument) was done with a single-stage silicone rubber membrane separator. Electron ionization voltage was 70 eV.

Authentic chemical samples were obtained from commercial sources, synthesized by establishing methods, or isolated by using liquid adsorption chromatography and GLC from known essentual oils [(*E*)-β-ocimene from Opopanax oil; caryophyllene, (*E*)-β-farnesene, α- and γ-muurolene, and δ-cadinene from hop oil; α-cubebene and α-copaene from cubeb oil; α-farnesene from ylang-ylang oil; cf. Buttery et al. (1984)]. Identities of all authentic compounds were verified by spectral (IR and MS) means by comparison with published data [e.g., Heller and Milne (1978)].

RESULTS AND DISCUSSION

The amount of total volatile material isolated (based on GLC peak areas) was of the order of 10–50 ppb of the leaves (and stems). Separate capillary GC–MS analyses were carried out on six different batches of wheat leaves (and stems) harvested at intervals during the first 6 months of 1984. This was to ensure that the qualitative analysis was consistent and that some idea could be obtained on the quantitative variation. The main study was concentrated on wheat leaves ca. 15–20 cm high.

The volatiles identified are listed in Table I together with the range of relative concentrations found in the different samples. Compounds were considered identified if their mass spectra and GLC Kovats' indices were consistent with those of authentic samples analyzed on the same instruments. Major mass spectral ions found are the same as those listed in a recent report by some of the authors (Buttery and Ling, 1984).

Table I. Volatile Components of Wheat Leaves (and Stems)

peak[a] no.	compound[b]	Kovats' GLC index[c] Carb. 20-M	Sil. OV-3	rel %
	Aliphatic Aldehydes and Ketones			
1	2-pentanone	990	705	0–1
4a	(*Z*)-3-hexenal	1100	820	1
7	2-methyl-2-pentenal	1130	850	0.2–2
9	(*E*)-2-hexenal	1190	880	2–12
44	nonanal	1390	1120	0.5–1
74	2-undecanone	1600	1310	0.5–2
	Aliphatic Alcohols and Esters			
1a	1-penten-3-ol	1140	710	0–1
2	2-methylbutanol	1180	810	0–5
2	3-methylbutanol	1180	810	
27	(*Z*)-3-hexenyl acetate	1310	1020	40–51
10	(*Z*)-3-hexenol	1370	930	6–18
39	octanol	1530	1110	0–2
	Terpenoids			
29	(*Z*)-β-ocimene	1230	1030	0.5
32	(*E*)-β-ocimene	1250	1050	6–12
38	linalool oxide A [2-methyl-2-vinyl-5-(2-hydroxy-2-propyl)tetrahydrofuran]	1440	1110	0.4–3
43	linalool	1545	1120	0.3–5
60	linalool oxide C (2,6,6-trimethyl-2-vinyl-5-hydroxytetrahydropyran)	1720	1230	0.5
84	α-cubebene	1420	1360	0.1–0.5
86	α-copaene	1460	1390	0.6–3
90	caryophyllene	1570	1435	4–11
96	(*E*)-β-farnesene	1650	1450	0.5–2
99	γ-muurolene	1655	1470	0.2–1
103	α-muurolene	1700	1510	0.2–1
105	α-farnesene	1735	1520	0.2–1
108	δ-cadinene	1730	1540	0.5

[a] Peak number in Figure 1. [b] Mass spectrum and Kovat's GLC retention index are consistent with that of an authentic sample. [c] Kovat's GLC index for the Pyrex Carbowax 20-M capillary GLC column and Silicone OV-3 capillary GLC column as indicated.

As with oat leaves (Buttery et al., 1982), (*Z*)-3-hexenyl acetate, (*Z*)-3-hexenol, and (*E*)-β-ocimene were the major volatile components. The commonly occurring caryophyllene was also the major sesquiterpene. Several other sesquiterpenes (listed in Table I) were also identified. A few additional sesquiterpenes were detected in minor amounts in some samples but were not consistently present throughout (these are not listed). (*E*)-β-Farnesene was found previously in both alfalfa and red clover leaves [cf. Buttery et al. (1984)]. It is a known alarm pheromone of some aphids (Wohlers, 1981). α-Farnesene, also identified in the wheat leaves, is known to occur in the skins of apples, pears, and quinces (Murray, 1969). Both α- and β-farnesenes are somewhat less stable than most of the cyclic sesquiterpenes and therefore may not be as quantitatively recoverable at the ppb concentrations found in wheat and other crop leaves. Care was taken in handling the wheat leaves (and stems) that they were not damaged after removal from the plant. To minimize damage, the leaves were not removed from the stem, the whole plant (above the roots) being placed in the 12-L flask for the isolation of the volatiles. With such samples there is some unavoidable damage, though, at the point where the stem was cut from the base of the plant. Very little of the usual aldehydes, etc., that result from tissue damage were ob-

served, however, and we feel that the severed area contributes only a minor amount to the total volatiles. There seems no other practical way of obtaining the volatiles from the leaves without introducing other artifacts from the soil, environment, plastic containers, etc.

The volatile compounds in macerated wheat leaves reported by Hamilton-Kemp and Andersen (1984) are quite different from those found in the present work. As discussed in previous work [e.g., Buttery and Ling (1984)], damage to the plant material (maceration would cause extreme damage) gives rise to considerable oxidative enzyme activity that breaks down the plant lipid and carotenoid components to a relatively large amount of volatile aliphatic aldehyde and alcohol compounds that are not present in the intact plant [cf. Schwimmer (1981)]. Such enzyme-produced volatiles can completely obscure the original volatiles that may be 100 times less in concentration. The enzyme action might also destroy (e.g., oxidise) some of the volatiles present in the intact plant. To fully understand the attraction of insect pests to their specific host plants, it would seem of primary importance to identify the compounds emitted by the intact plant. Volatiles emitted by damaged plants may also be important in the attraction of certain insect pests.

Comparison with Oat and Barley Leaf Volatiles. The major volatiles found in the wheat leaves are qualitatively the same as those found by Buttery et al. (1982) in both oat and barley leaves (by Tenax trapping). The method using Tenax trapping is not well suited to good quantitative comparison but is satisfactory for semiquantitative information [cf. Dressler (1979)]. (*Z*)-3-Hexenyl acetate is the most predominant volatile in all three cereal leaves. It does appear to be, however, much more predominant in oat leaves than in either barley or wheat.

Registry No. 2-Pentanone, 107-87-9; (*Z*)-3-hexenal, 6789-80-6; 2-methyl-2-pentenal, 623-36-9; (*E*)-2-hexenal, 6728-26-3; nonanal, 124-19-6; 2-undecanone, 112-12-9; 1-penten-3-ol, 616-25-1; 2-methylbutanol, 137-32-6; 3-methylbutanol, 123-51-3; (*Z*)-3-hexenyl acetate, 3681-71-8; (*Z*)-3-hexenol, 928-96-1; octanol, 111-87-5; (*Z*)-B-ocimene, 3338-55-4; (*E*)-B-ocimene, 3779-61-1; linalool oxide A, 34995-77-2; linalool, 78-70-6; linalool oxide C, 39028-58-5; α-cubebene, 17699-14-8; α-copaene, 3856-25-5; caryophyllene, 87-44-5; (*E*)-β-farnesene, 18794-84-8; γ-muurolene, 30021-74-0; α-muurolene, 10208-80-7; α-farnesene, 502-61-4; δ-cadinene, 483-76-1.

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Quality Changes in Lobster (*Panulirus polyphagus*) Muscle during Storage in Ice

Riaz Fatima and R. B. Qadri*

Studies have been made on organoleptic, chemical, and microbiological changes in lobster (*Panulirus polyphagus*) tail muscle stored in ice up to 15 days simulating commercial practice. These changes were correlated with taste panel evaluations of the sensory quality. Significant correlation coefficients were obtained between the mean organoleptic response and the various objectively measured changes during the storage period. The merits of these changes as objective indices of quality, particularly in relation to taste panel assessment, are discussed.

The export of lobster is an important part of the seafood industry of Pakistan. In 1980, 16 tons of lobster valued at Rs. 1.93 millions in foreign exchange were exported in frozen, dried, and live forms ("Handbook of Fisheries Statistics of Pakistan", 1980). Great potential, however, exists for increasing its export in present forms as well as in canned and chilled forms.

For proper utilization attention must be given to the fact that lobster is highly perishable. This necessitates measures to be taken immediately after capture to prevent deterioration in quality. Quality problems are aggravated by the variability of raw material due to the influence of environment, food, season, etc., and by the biochemical reactions that take place after death and that are influenced by the physiological conditions at the time of catch.

Pakistan Council of Scientific and Industrial Research Laboratories, Karachi—39, Pakistan.

There are several reports of the general pattern of spoilage and storage stability of crustaceans. Loss of quality and subsequent spoilage of crustaceans and other seafoods are caused primarily by tissue enzymes and microbial activities. Various tests have been proposed to determine the quality and expected storage life of the raw product. Included are acid-soluble orthophosphate, trimethylamine nitrogen, amino nitrogen, extract release volume, pH, and bacterial count. At present, however, organoleptic measurements of quality are used for purchasing and grading seafoods at various stages during their storage life (Baily et al., 1956; Vanderzant and Nickelson, 1971; Cobb and Vanderzant, 1975; Farooqi et al., 1978). Different species of prawn and scampi (*Nephrops norvegicus*) showed that flesh from crustaceans spoiled at a faster rate than that from cod and other teleosts under similiar conditions (Fieger and Novak, 1961; Vyncke, 1968; Walker et al., 1970). Simidu (1961) reported that the higher rate of spoilage is due to a high proportion of amino

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