Evidence that the hydrogen sulphide was released during extraction and was not present in the intact fruit was obtained in this study with control fruit. Withdrawal by the procedure of Maier *et al.* [2] of 4–10 ml samples of the interior gas of several fruit and analysis by the above method showed no hydrogen sulphide present.

Thus the measurement of hydrogen sulphide content of freshly extracted citrus juice has potential as an index of quality for determining the degree of freeze damage to fresh citrus fruit. The effect of decreased hydrogen sulphide in headspace gases above freeze-damaged fruit was present several months after freeze damage had occurred.

EXPERIMENTAL

Valencia orange samples were obtained from Mr. Leon Hebb, Citrus Budwood Registration Bureau, Winter Haven, Florida, and from Triangle Grove Service, Winter Haven, Florida, and were judged for degree of freeze damage based on location and visual damage to the trees. Virtually no fruit escaped at least mild freeze damage from the 13 January 1982 freeze (L. Hebb, personal communication). Fruit was washed, halved and the juice expressed by hand from 1–3 fruits for each sample. A 100 ml portion of fresh juice in a 125 ml Erlenmyer flask was used for hydrogen sulphide determination with a gas chromatograph equipped with a flame photometric detector as described earlier [1, 3]. Headspace samples (10 ml) were withdrawn in the period from 2 to 60 min after extraction. Average values for 5–8 determinations for each sample are reported in Table 1.

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VOLATILE COMPOUNDS FROM *TRITICUM AESTIVUM*

THOMAS R. HAMILTON-KEMP and ROGER A. ANDERSEN*

Department of Horticulture, University of Kentucky, Lexington, KY 40546, U.S.A.; *Agricultural Research Service, U.S. Department of Agriculture and Department of Agronomy, University of Kentucky, Lexington, KY 40546, U.S.A.

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Key Word Index—*Triticum aestivum*; Gramineae; wheat; volatiles; C_9 aldehydes and alcohols.

Abstract—Volatile compounds were isolated from aerial parts (foliage and culms) of wheat plants by reduced pressure steam distillation-extraction and identified by gas chromatography-mass spectrometry and co-chromatography with authentic compounds. Infrared spectra were also obtained on some constituents. Compounds identified included nonanal and related unsaturated C_9 aldehydes and alcohols as major components and some additional aldehydes, alcohols and a ketone.

INTRODUCTION

Volatile compounds have been extensively investigated in the edible portions of plants [1] but not as thoroughly in the remaining aerial parts of most major crops. It is known that volatile compounds are involved in plant–parasite interactions [2–6] and greater attention is now being focused on identification of members of this class of compounds in crop plants based on their possible functions in insect and pathogen interactions with plants. As part of an investigation of the volatile compounds in crop plants we have isolated and identified some volatile compounds from wheat.

RESULTS AND DISCUSSION

Approximately 2 mg of volatile oil was isolated per 1 kg of the aerial parts of wheat plants (foliage and culms). A list of compounds isolated and identified by GC/MS and co-chromatography with authentic compounds is given in Table 1. In some instances where adequate sample was available, IR spectra were also obtained. Among the major compounds found were nonanal and a group of closely related unsaturated C_9 aldehydes and alcohols. C_9 aldehydes and alcohols were isolated and characterized as major and characteristic volatile constituents of members of the cucurbit family in earlier work and appear to contribute to their flavour chemistry [7–9]. It has been shown that nonanal and, subsequently, other volatile compounds including nonanol, octanol and 6-methyl-5-hepten-2-one stimulate germination of fungal spores including those of *Puccinia* species which parasitize wheat plants [10, 11]. Nonanal was a major volatile component and 6-methyl-5-hepten-2-one was present in spore distillates and in airstreams drawn through wheat rust (*Puccinia* species) spores [10]. There have been no reports about effects on spore germination of the unsaturated C_9

Table 1. Volatile compounds from wheat plants

Compound	Evidence*	% Composition in volatile oil
Hexanal	MS, R_t	1.3
trans-2-Hexenal	MS, IR, R_t	11.1
Hexan-1-ol	MS, R_t	0.6
trans-2-Hexen-1-ol	MS, R_t	1.7
Heptanal	MS, R_t	2.2
trans-2-Heptenal	MS, R_t	tr†
Heptan-1-ol	MS, R_t	tr
trans-2-Octenal	MS, R_t	0.5
Octan-1-ol	MS, R_t	0.5
Nonanal	MS, R_t	9.9
trans-2-Nonenal	MS, R_t	2.2
trans,cis-2,6-Nonadienal	MS, IR, R_t	7.7
Nonan-1-ol	MS, R_t	tr
trans-2-Nonen-1-ol	MS, R_t	1.2
cis-3-Nonen-1-ol	MS, R_t	2.5
trans,cis-2,6-Nonadien-1-ol	MS, IR, R_t	5.6
cis,cis-3,6-Nonadien-1-ol‡	MS, R_t	5.0
Decanal	MS, R_t	0.5
trans-2-Decenal	MS, R_t	tr
β-Ionone	MS, R_t	3.6
Benzaldehyde	MS, R_t	tr
Unidentified	—	43.9

*MS, IR, R_t represent mass spectrum, infrared spectrum and gas chromatographic retention time, respectively.

†tr indicates less than 0.5%.

‡Standard obtained from cucurbits.

compounds in the volatile oil from wheat that are structurally related to nonanal and nonanol. Evidence has been obtained that the C_9 compounds isolated, arise in plant tissue from enzymatic cleavage of the 9,10 double bonds of C_{18} fatty acid moieties [12].

Additional aldehydes (including *trans*-2-hexenal, a major component), alcohols, and a ketone (β-ionone) were also identified in wheat. Many volatile compounds representing *ca* 40% of the oil, including higher boiling constituents, remain unidentified due to difficulty of interpretation of spectra or lack of authentic standards. Mass spectral data indicated the presence of alkyl benzenes, for example, xylenes and ethyl toluenes, which appear to be similar to compounds reported in unprocessed rice [13]. One major higher boiling compound (*ca* 3.5% of the oil) yielded a mass spectral fragmentation pattern, IR spectrum (bands at 2700 and 1730 cm^{-1}) and GC elution time which were consistent with pentadecanal; however, an authentic sample was not available for confirmation. Although earlier investigations on wheat grain and flour [14–16] have concentrated on compounds generally more volatile than those identified here, hexanal and heptanal were also identified from these sources.

EXPERIMENTAL

Isolation of volatile compounds. Foliage and culms of winter wheat cultivar 'Authur 71', were harvested during the first week in May, 1983 from the University of Kentucky Experiment Station farm in Lexington and stored at −20°. One kg of plant material was macerated in a Waring blender with 1.8 l. of H_2O. The sample was placed in a 12 l. flask of a water recycling apparatus [17] which was adapted for vacuum steam distillation. Four ml of hexane were placed on top of the H_2O layer in the side arm of the apparatus. The pressure in the system was reduced and steam distillation-extraction was carried out at 60–70° for 3 hr. The hexane layers from eight distillations were combined, dried (Na_2SO_4) and the hexane removed under a stream of N_2 to yield a volatile oil.

Separation and identification. GC of the volatile compounds was carried out initially on a 1.8 m × 4 mm i.d. column packed with 20% SE-30 coated on silanized Chromosorb W. The temp was programmed from 100° to 180° at 1°/min and fractions corresponding to chromatographic peaks were collected from the GC in glass U-shaped tubes cooled in a solid CO_2–Me_2CO bath. These samples were analysed with a GC/MS instrument operated at 70 eV ionization potential using a 30 m × 0.25 mm Carbowax 20M fused silica capillary column. The identification of compounds was confirmed by matching MS data and co-chromatography of plant components and authentic compounds on the Carbowax 20M fused silica capillary column. Quantitation of individual components was carried out using an electronic integrator. IR spectra were obtained with the aid of a NaCl microcell and a mirror beam condenser; spectral grade CS_2 was used as solvent.

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