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IDENTIFICATION OF SOME VOLATILE COMPOUNDS FROM *CITRULLUS VULGARIS*

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Abstract—Volatile compounds from watermelon (*Citrullus vulgaris*), obtained by vacuum steam distillation—extraction of the fruit at 60–70° in a water-recycling apparatus, were separated by gas chromatography and subjected to spectral analyses. Evidence was obtained for the following new constituents, hexanal, *trans*-2-heptenal, *trans*-2-octenal, nonanal, *trans*-2-nonenal, *trans,cis*-2,6-nonadienal, nonan-1-ol, *trans*-2-nonen-1-ol, *cis*-3-nonen-1-ol, *trans,cis*-2,6-nonadien-1-ol, *trans*-2-decenal, *trans*-2-undecenal, geranial and β-ionone.

INTRODUCTION

Earlier, a group of unusual C_9 aldehydes and alcohols was isolated from muskmelon (*Cucumis melo* L.) fruit and identified[1, 2]. Included among these compounds were *cis*-6-nonenal which has a flavor reminiscent of that of melon or green melon and a nonadienol which is reminiscent of watermelon or watermelon rind. The latter compound was isolated from water melon (*Citrullus vulgaris* Schrad) fruit in sufficient quantity to allow its characterization as *cis,cis*-3,6-nonadien-1-ol[2]. Subsequently, cucumber (*Cucumis sativus* L.) essence was shown to contain all of the C9 aldehydes and alcohols found in muskmelon essence and evidence was obtained for two additional C_9 aldehydes, namely 3-nonenal and 3,6-nonadienal[3]. These aldehydes and alcohols are thought to arise from the cleavage of the 9–10 double bonds of oleic, linoleic, 9,15-*iso*-linoleic and linolenic acids[2, 4, 5]. (A group of C_{16} and C_{17} aldehydes also structurally related to oleic, linoleic and linolenic acids has recently been identified in cucumber essence[6]).

As part of an investigation of the interrelationships of the volatile constituents of the major cucurbits (water melon, muskmelon and cucumber), we have now analysed a concentrate of volatile compounds prepared from watermelon fruit.

RESULTS AND DISCUSSION

An essence or concentrate of volatile compounds was prepared from the fruit by reduced pressure steam distillation–extraction. Initial GLC fractionation of the essence was accomplished using an SE-30 column. Fractions were rechromatographed on a DEGS or a Carbowax column and purified compounds were submitted to spectral analyses. A list of compounds identified and evidence for identification are presented in Table 1. The notation R_t indicates that the identity of a component was confirmed by comparison of its GLC R_t and spectra with those of an authentic compound. The identification of compounds by the notation MS or MS, IR alone is tentative and is based on comparison with published information.

From Table 1 it can be seen that ten C_9 aldehydes and alcohols have been isolated from watermelon and identified. In addition, GLC peaks were observed with the same RR_t as the two C_9 compounds isolated previously from cucumber and tentatively identified as 3-nonenal

Table 1 Volatile compounds isolated from watermelon

Compound	Evidence
Hexanal	MS[10]
trans-2-Hexenal	MS, IR, R_t
trans-2-Heptenal	MS[10]
trans-2-Octenal	MS[10]
Nonanal	MS, R_t
trans-2-Nonenal	MS, IR, R_t
cis-6-Nonenal	MS, IR, R_t
trans,cis-2,6-Nonadienal	MS, IR, R_t
Nonan-1-ol	MS, R_t
trans-2-Nonen-1-ol	MS, IR, R_t
cis-3-Nonen-1-ol	MS, IR, R_t
cis-6-Nonen-1-ol	MS, IR, R_t
trans,cis-2,6-Nonadien-1-ol	MS, IR, R_t
cis,cis-3,6-Nonadien-1-ol	MS, IR, NMR[2]
trans-2-Decenal	MS[10]
trans-2-Undecenal	MS[11]
Geranial	MS[10]
β-Ionone	MS, R_t

and 3,6-nonadienal. Two C_9 alcohols, namely 3,6-nonadien-1-ol and 3-nonen-1-ol were major constituents of the watermelon essence. In comparison, the aldehydes 2,6-nonadienal and 2-nonenal predominated in cucumber essence whereas all C_9 compounds were minor constituents of muskmelon essence. MS evidence was obtained for a group of 2-alkenals with chain lengths from C_6 through C_{11} in watermelon essence. Several 2-alkenals have also been isolated from cucumber[7] and these compounds are thought to arise from lipid oxidation.

Katayama and Kaneko have studied water melon volatiles earlier[8], identifying them solely by comparisons of GLC Rt Among the compounds listed in Table 1, *trans*-2-hexenal is the only compound reported by these investigators.

EXPERIMENTAL

Watermelons were purchased at a market and stored frozen at −23° prior to steam distillation-extraction. (Initially, all analyses were performed on an essence prepared from frozen watermelon; however, GLC R_t data were used subsequently to show that all compounds studied were also present in an essence prepared from fresh watermelon.) Rinds and seeds were removed and 1·6 kg of flesh was macerated with 2 l of dist H_2O The macerate was subjected to steam distillation–extraction in a water-recycling apparatus at 60–70° under red pres as described previously[1–9]. GLC separation of the essence was initially effected on a 1·8 m × 6 mm o.d stainless steel column packed with 20% SE-30 on Chromosorb W The column temp was programmed from 100–180° at 1°/min Fractions collected from the SE-30 column were rechromatographed on a 1·8 m × 6 mm o.d stainless steel column packed with 10% diethyleneglycol succinate (DEGS) or 20% Carbowax 20 M on Chromosorb W Resulting subfractions were collected and submitted to spectral analyses

MS were recorded on double focusing instrument operated at 70 eV IR spectra were obtained using a NaCl microcell and a mirror beam condenser, spectral grade CS_2 was used as solvent Reference compounds were obtained from commercial suppliers or from other laboratories A reference sample of *cis*-3-nonen-1-ol was obtained from muskmelon essence

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