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Technical Note

Volatile Aroma Constituents of Noble Muscadine Grapes

ABSTRACT

The volatile components of Noble muscadine grapes were analysed with a combined gas chromatograph–mass spectrometer. Over twenty-five peaks were separated, of which fifteen were identified. A gas chromatogram of the grape extract is presented, along with a Table of compounds and their associated mass spectral data.

INTRODUCTION

Vitis rotundifolia Michx. (muscadine) grapes are the principal grape cultivars grown in the southeastern part of the United States. These grapes grow singly as berries or in small clusters. They are often thick skinned and are usually resistant to Pierce's disease. Their disease resistance gives them the potential of being grown in many hot and humid areas where the cultivation of more susceptible varieties, such as *V. vinifera* and *V. labrusca* is not possible. Several muscadine varieties are found and these vary in colour, flavour, texture and size. One of the unique characteristics of these grapes is their intense aroma. Consequently, they are widely used locally to produce juices, jellies, preserves and wines. They are also known to furnish their characteristic aroma to blends predominating in neutral flavoured *V. vinifera* wines.

Investigations directed towards the determination of the aroma compounds of muscadine grapes have been carried out in the past (Kepner & Webb, 1956; Berry *et al.*, 1979*a*, *b*; Flora & Nakayama, 1981;

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TABLE 1
Volatile Constituents of Muscadines Previously Reported

Kepner & Webb (1956)

Methyl alcohol
Ethyl alcohol
n-Butyl alcohol
Isoamyl alcohol
1-Hexanol
2-Phenylethanol
Acetaldehyde
Isobutyraldehyde
Acetal
Biacetyl
1-Hexanal
Ethyl acetate
Caproate ester
Caprylate ester
Caprate ester
Laurate ester
Methyl ethyl ketone

Berry *et al.* (1979*a*)

Methanol
Ethanol
Butanol
2-Methylbutanol
Hexanol
trans-2-Hexen-1-ol
2-Phenylethanol
trans-2-Hexenal
Ethyl acetate
Ethyl propionate
Propyl acetate
Butyl acetate
Benzyl acetate
Ethyl caprate
d-Limonene

Welch *et al.* (1982)

Ethyl acetate
Isobutyl alcohol
Isoamyl acetate
Ethyl crotonate
Isoamyl alcohol
1,8-Cineole
Isopropyl hexanoate
Hexyl acetate
Acetoin
Ethyl heptanoate
cis-3-Hexen-1-ol
Benzaldehyde
1-Octanol
2-Fenchyl alcohol
Hexadecane
Ethyl decanoate
1,2-Dimethoxy-1-phenylethane
Borneol
Ethyl phenylethylacetate
Geraniol
Benzyl alcohol
2-Methoxy-1-phenyl-2-hydroxyethane
Butyl acetate
Diethyl carbonate
Butanol
1,4-Cineole
Limonene
Ethyl hexanoate
Styrene
p-Cymene
2-Heptanol
Hexanol
Ethyl 3-hydroxy-butyrate
Linalool
Terpinen-4-ol
Methyl benzoate
p-Allylanisole
α-terpineol
Benzyl acetate
2-Phenylethyl acetate
Ethyl laurate
2-Phenylethanol

Welch *et al.*, 1982). The compounds identified by some of these workers are shown in Table 1. In these, volatile compounds were isolated from grape samples after they were either passed through flash evaporators (Kepner & Webb, 1956), or steam distilled (Berry *et al.*, 1979*a*; Flora & Nakayama, 1981; Welch *et al.*, 1982). While the muscadine grape varieties used by Kepner and Webb, Flora and Nakayama and Welch *et al.* were not specified in their reports, the volatiles present in Hunt and Higgins varieties were investigated by Berry and his co-workers.

One of the most promising muscadine varieties for commercial grape juice production is the Noble. The red juice obtained from this grape, prior to heat treatment, is as flavourful as many commercial single-strength grape juices. More research is, however, needed in order to prevent, or retard, the flavour and colour changes that accompany its pasteurization (Bates, 1982; Flora, 1976, 1977). This paper describes the analysis and identification of the volatile compounds isolated from Noble grape juice.

EXPERIMENTAL

Preparation of samples

Grape clusters were harvested at maturity from the campus trial vineyard and berries were manually removed from the stems. The grapes were rinsed several times with water, after which they were crushed on a commercial grape crusher. Harvest parameters were determined on the juice that was cold pressed from these grapes. Juice samples (100 ml) were saturated with sodium chloride, and volatile compounds were extracted with a solvent mixture (25 ml) containing pentane and dichloromethane (7:3) (Miguel *et al.*, 1981). These solvents were over 99% pure and were used without further purification. The extract (8 ml) was filtered after drying over anhydrous sodium sulphate. An odour similar to that of the grape juice was detected in the solvent extract, which was analysed without concentrating by solvent removal.

Analysis of volatile compounds

Volatiles were analysed using a Finnigan model 4510 gas chromatograph–mass spectrometer equipped with an INCOS data system. Samples

TABLE 2
Identity of Volatile Constituents of Noble Grape

Peak no.	*Compound*[a]	*Relative area covered by peak*	*MS Data m/e (relative intensity)*[b]	*Probability of correspondence to published data*
1	1-(2-Butoxyethoxy)-ethanol	0·858	M^+ = 162, 57(100), 45(99), 41(32), 75(20), 87(13), 59(12),72(10), 89,44(9)	0·99
2	Ethyl cyclohexane	0·065	M^+ = 112, 82(100), 43(89), 96(64), 59(53), 40(51), 95,97(29), 44,55,68(22)	0·87
3	3-Ethyl-3-nonen-2-one	0·053	M^+ = 168, 43(100), 97(61), 71(56), 125(30), 40(25), 41(21), 55,83(16), 99(15), 69,107(14)	0·80
4	2,6-bis(1,1-Dimethylethyl)-4-methyl-phenol	0·029	M^+ = 220, 205(100), 220(37), 57(26), 40,206(16), 221(7), 41(6), 44,145(5), 43(4)	0·81
5	4-Hydroxy-3,5-dimethoxy-benzaldehyde	0·034	M^+ = 182, 182(100), 181(43), 40(26), 43,93,183(9), 44,167(7)	0·79
6	1,3-Dimethyl cyclopentane	0·0017	M^+ = 98, 40(100), 55(86), 69(57), 43,44(52), 97(48), 56,57(43), 73,83(29)	0·74
7	Undecanoic acid	0·031	M^+ = 186, 60(100), 43,73(79), 57(67), 40(57)	0·89
8	Dihydro-5-methyl-3-furanone	0·059	M^+ = 100, 100(100), 40(8), 101(7), 43,44(5), 55,57(4), 42,56(3)	0·93
9	Pentyl cyclopropane	0·051	M^+ = 112, 55(100), 57(91), 40(87), 43(83), 69,83(78), 40,97(65), 84(61), 56,58(57)	0·85

10	Hexadecanoic acid	0·636	M^+ = 257, 73(100), 60(92), 57(80), 43(76), 71(51), 129(48), 55(38), 41(33), 85(31), 61(29)	0·97
11	2,4-Octadecenal	1·000	M^+ = 264, 55(100), 69(88), 83(79), 97(67), 43(62), 41(56), 57(53), 56(49), 84(48), 70(45)	0·90
12	Octadecanoic acid	0·108	M^+ = 284, 43(100), 57(89), 60(86), 73(79), 55(55), 71(53), 69(52), 129(48), 41(44), 85(37)	0·76
13	3,4-Dimethyl heptane	0·064	M^+ = 128, 57(100), 71(72), 85(55), 43(45), 40(20), 99(18), 55(13), 69,113,127(12)	0·89
14	1-Hydroxy-2-(hydroxy-methyl)-9,10-anthracenedione	0·055	M^+ = 286, 286(100), 271(81), 189(67), 40(50), 175(45), 69(36), 201(31), 44(29), 43(26), 55,149,272(17)	0·95
15	3,5-Dimethyl octane	0·076	M^+ = 142, 57(100), 71(84), 85(51), 43(47), 40(27), 99(23), 55,113(16), 127(14), 111,141(13)	0·87

[a] Tentative identification by computer search.

[b] Ten most intense peaks.

(200 μl) were injected onto a 30 m × 0·32 mm inside diameter DB5 glass capillary column. GC oven temperature was programmed from 60 °C to 100 °C at the rate of 10 ° a minute and then to 240 °C at 5 ° a minute. The hydrogen carrier gas was used to maintain a pressure of 10 psi on the column. Mass spectrometer conditions were as follows. Ionization voltage, 70 EV; emission current, 0·3 μA; ion acceleration voltage, 5V; and ion source temperature, 140 °C. Identification of the resulting peaks was carried out from their respective mass spectral data, after a computer search, using the National Bureau of Standards' library data, for compounds with identical, or closely related, spectra.

RESULTS AND DISCUSSION

None of the compounds identified in this study (Table 2) had previously been present in muscadine grapes. The chromatogram of the extract (Fig. 1) shows that 2,4-octadecenal was the predominant volatile compound isolated. The respective concentrations of 1-(2-butoxy ethoxy)-ethanol and hexadecanoic acid were relatively high compared with those of the

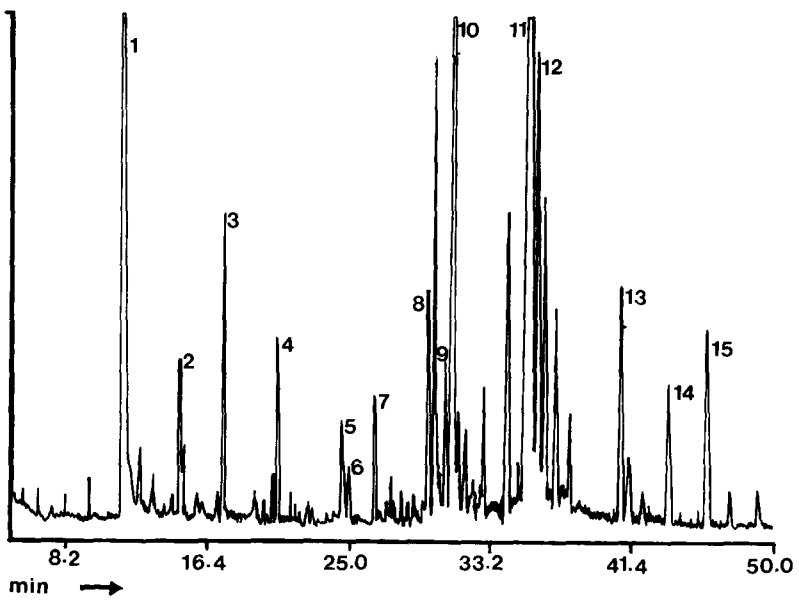

Fig. 1. Gas chromatogram of Noble grape extract. Harvest parameters were: pH, 3·24; total acidity, as tartaric 0·67 %, °Brix, 13·0.

other compounds present in the extract. A minimum of twenty-two other compounds were extracted, many of which were identified. These compounds were not present in controls carried out in the absence of the grape juice. The areas covered by each identified peak on the chromatogram are shown in Table 2.

The difference between the compounds obtained from this investigation and those reported earlier may be attributed to factors such as difference in isolation techniques and the grape variety used. The isolation of volatiles from musts and wines with pentane–dichloromethane solvent was previously used by Miguel *et al.* (1981). Many compounds were isolated with this solvent mixture and these authors observed that this method is superior to that which involves isolation of volatile constituents by steam distillation or headspace analysis of wines. Muscadine juices, especially those obtained from red grapes, undergo rapid colour and flavour changes when heated (Flora 1976; 1977) and preliminary results of an ongoing investigation (O. Lamikanra, unpublished work) indicate significant changes in the volatile compounds isolated from Noble juice that was rapidly heated to 95 °C for less than 5 min. The direct solvent extraction method was therefore chosen, in preference to steam distillation, for this reason. The extent to which the isolation method and varietal differences affect the observed results is not known and the absence of many compounds identified earlier might result from the inability of the solvent to extract these compounds directly from the grape juice. It has, however, been noted in the past (Berry *et al.*, 1979*a*; Flora & Nakayama, 1981; Welch *et al.*, 1982) that many compounds that seemed to be of primary importance to the flavour of muscadine grapes have yet to be isolated and determined. The fact that the odour given off by the solvent extract has some resemblance to the grape juice from which it was extracted suggests that the compounds present in the extract contribute significantly to the aroma of Noble juice.

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