

Compounds Identified in Commercial Blackberry Essence

A commercial blackberry essence was extracted with ethyl chloride and the extract was analyzed by combined gas chromatography-mass spectrometry. Compounds identified were: 3,4-di-

methoxyallylbenzene, 3,4,5-trimethoxyallylbenzene, eugenol, α -ionone, 2-phenethylformate, and 2-phenethyl alcohol.

The identity of 16 volatile compounds in a commercial blackberry essence has been reported (Scanlan *et al.*, 1970). More recently, Houchen *et al.* (1972) demonstrated that *p*-cymen-8-ol was converted to 1-methyl-4-isopropenylbenzene by a heat-induced dehydration reaction. The identification of several volatile components not previously identified in commercial blackberry essence is presented herein.

EXPERIMENTAL SECTION

The commercial essence, procedures for essence extraction, gas chromatographic (gc) separation of the extract, and the trapping of fractions were described previously (Scanlan *et al.*, 1970). Several of the fractions were further separated for tandem gc-mass spectral (ms) examination as follows: column, 12 ft \times $\frac{1}{8}$ in. o.d. stainless steel packed with 2.5% Carbowax 20M on 80/100 mesh AW Chromosorb G; column temperature, 140° isothermal; injector, 220°, detector, 230°; flow rate, 30 ml of He per min at 25°.

The gc was fitted with an effluent splitter which directed 17% of the effluent to the flame ionization detector and the remainder to the ms. The heated connection between the gc and the ms was fitted with a microvolume switching valve (Carle Instruments Inc., Fullerton, Calif.) and a membrane-type separator. The valve allowed diversion of the large solvent peak from the ion source of the ms.

Mass spectra were obtained using an Atlas CH-4 ms. Scans were made from *m/e* 25 to *m/e* 250 in 4.5 sec. The ms operating conditions were: filament current, 20 μ A; electron voltage, 70 eV; accelerating voltage, 3.0 kV; and analyzer pressure, 1.5×10^{-6} mm.

Samples subjected to infrared analysis (ir) were trapped from the Carbowax 20M column as described previously (Houchen *et al.*, 1972) and analyzed as a thin film between two NaCl plates on a Beckman IR-5 infrared spectrophotometer equipped with a 5X beam condenser.

RESULTS AND DISCUSSION

Compounds not previously identified in commercial blackberry essence are listed in Table I. A positive sign (+) for gc retention indicates that the relative retention time was within 5% of the values for the corresponding known compound. A + for ms indicates that the ms fragmentation pattern agreed either with a mass spectrum of the known obtained in our laboratory or with a published spectrum. A + for ir means that the ir spectrum matched

Table I. Compounds Identified in a Commercial Blackberry Essence

Compound	Identification based on		
	gc retention	ms	ir
3,4-Dimethoxyallylbenzene	+	+	+
3,4,5-Trimethoxyallylbenzene		+	+
Eugenol	+	+	+
α -Ionone	+	+	
2-Phenethylformate	+	+	
2-Phenethylalcohol	+	+	

the ir spectrum of the corresponding known compound run in our laboratory.

The presence of a dimethoxyallylbenzene in commercial blackberry essence was indicated earlier by Scanlan *et al.* (1970); however, a determination of the positions of substituents was only possible after comparison of their spectrum with the spectrum of known 3,4-dimethoxyallylbenzene. A component with an apparent molecular weight of 168 possessing a strong blackberry-like odor was not identified, but further work will be directed toward the identification of this component.

ACKNOWLEDGMENT

The authors gratefully acknowledge the helpfulness of Merlin Fischer, Kerr Fruit Concentrates, Inc., in providing the blackberry essence.

LITERATURE CITED

- Houchen, M., Scanlan, R. A., Libbey, L. M., Bills, D. D., *J. Agr. Food Chem.* **20**, 170 (1972).
 Scanlan, R. A., Bills, D. D., Libbey, L. M., *J. Agr. Food Chem.* **18**, 744 (1970).

Michael P. Gulan
 Marilyn Houchen Veek
 Richard A. Scanlan*
 Leonard M. Libbey

Department of Food Science and Technology
 Oregon State University
 Corvallis, Oregon 97331

Received for review December 5, 1972. Accepted February 23, 1973. Technical Paper No. 3482, Oregon Agricultural Experiment Station, Oregon State University, Corvallis, Oregon 97331.