

## Research Note

### IDENTIFICATION OF SOME FLAVOR CONSTITUENTS OF *CHIRONJA*<sup>1, 2</sup>

The study of the components of the essential oils of citrus fruits is of major importance to the citrus industry. There is no literature concerning the composition of the essential oils of chironjas produced in Puerto Rico.

The separation and identification of the chemical compounds reported in this work were possible through the application of gas chromatography (GC) and mass spectrometry (MS). The fruits used were harvested at the Corozal Substation during the 1974 season. Samples of 1 to 1.5 kg of the edible portion of the fresh fruits (seed and albedo removed) were mixed with 3 l of distilled water in a blender. The mixture was distilled under reduced pressure for 4 h. The droplets containing the essential oils were collected over hexane. Six distillates were pooled and the hexane removed by flushing N over the distillate at room temperature to yield less than 1 ml of pale yellow oily extract. Anhydrous sodium sulfate was used to remove traces of water.<sup>3, 4</sup>

The separation of the flavor components through gas chromatography was performed in two steps. A 70  $\mu$ l extract sample was injected into a Barber Coleman Series 5000<sup>5</sup> gas chromatograph equipped with a thermal conductivity detector system using a 6 ft  $\times$   $\frac{3}{8}$  in o.d. stainless steel column packed with 20% SE-30 on 60-80 mesh Chromosorb W. The He flow rate was 50 ml/min. The temperature was programmed from 100° to 180°C at 1°C/min.

Twenty-four fractions were collected in glass capillary U tubes cooled in Dry Ice-acetone mixture. Each fraction was further separated by gas

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<sup>3</sup> Kemp, T. R., Stoltz, L. P., Smith, W. T., Jr., and Chaplin, C. E., The composition of the essential oil of leaves of strawberry cultivar "Citation," Proc. Amer. Soc. Hort. Sci. 93, 334-9, 1968.

<sup>4</sup> Kemp, T. R., Knavel, D. E., and Stoltz, L. P., Volatile Cucumis melo components: Identification of additional compounds and effects of storage conditions. Phytochem. 12, 2921-4, 1973.

<sup>5</sup> Trade names are used in this publication solely for the purpose of providing specific information. Mention of a trade name does not constitute a guarantee or warranty of equipment or materials by the Agricultural Experiment Station of the University of Puerto Rico or an endorsement over other equipment or materials not mentioned.

chromatography on 10% DEGS 6 ft  $\times$   $\frac{1}{4}$  in o.d. column operated isothermally at 110°, 120° and 150°C for specific fractions. Some of the fractions (corresponding to a large peak from the SE-30 column) were resolved into several individual peaks on the DEGS column.

Mass spectra were obtained on nine of the individual components resulting from the second step using a Perkin-Elmer Hitachi RMU 6 E double focusing mass spectrometer with an ionizing energy of 70 ev and 100  $\mu$ A with an oven temperature of 200°C.

The identity of the following seven of the nine components subjected to mass spectral analysis was based on the molecular ion, the base peak, fragmentation pattern, and comparison with reference spectra<sup>6</sup>: n-Octanol, d-Limonene,  $\alpha$ -Elemene,  $\beta$ -Elemene, Caryophyllene, Nootkatone, Methyl 9, 12, 15-Octadecatrienoate. Tentative identification of the additional eight components was performed by gas chromatographic comparison of their retention times with authentic standards supplied by Warner-Jenkinson Co.:  $\alpha$ -Pinene,  $\beta$ -Pinene, Myrcene, Citronellal,  $\alpha$ -Terpineol, Linalool, Nerol, Geraniol. Comparison of a chironja oil chromatogram with and without each added standard was also used to establish the tentative identification of unknown components.

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<sup>6</sup> Stenhagen, E., Abrahamsson, S., and McLafferty, F. W., Registry of Mass Spectral Data, Vols. 2 and 3, Wiley-Interscience, New York, 1974.