

# Volatile Constituents in Guava

Kenneth L. Stevens, John E. Brekke, and Donald J. Stern

The volatile components have been isolated from guava puree (*Psidium guajava*, L.) and separated by gas-liquid chromatography. The separated constituents were directed into a mass spectrometer via a molecular separator and identifications made from the mass spectral patterns. A total of 22 components have been identified with *cis*-3-hexen-1-ol, hexanol, and hexanal predominating. Several

aromatic compounds have been identified with methyl benzoate,  $\beta$ -phenethyl acetate, and methyl cinnamate comprising the bulk of this class. Some terpenes and terpene alcohols have also been identified. Several constituents have been classified as sesquiterpenes (m.w. = 204), but specific identifications have not been possible, with the exception of caryophyllene.

Although the investigation of the volatile constituents in fruits has been progressing at a phenomenal rate during the past few years, and especially with the advent of GC-MS combinations, relatively little attention has been given to the tropical fruits. The guava (*Psidium guajava*, L.) is an attractive fruit possessing many desirable characteristics. It has a rather delicate pink color and makes excellent juice, jam, jellies, etc. Because of this, the fruit is rapidly assuming greater economic importance. Guava is widely distributed in Hawaii (below 3000 ft), being introduced in the 1790's presumably from tropical America.

One of the first reports concerned with the volatile constituents was by Kunishi and Seale (1961) at the University of Hawaii. They made some preliminary studies on guava and found the puree to contain 49 ppm of oil. The oil, by infrared spectroscopy, contained neither carbonyl nor carbon-carbon double bonds; however it gave positive bromine and permanganate tests. No specific identifications were made.

In 1968 and 1969 a group of Indian workers (Pattabhiraman *et al.*) reported the isolation and partial characterization of the volatile material in guava. They found that the guava oil was not adversely affected by mild heating. Also, the isolated oil was subjected to thin-layer chromatography and a spot, detected by vanilla-sulfuric acid and 2,4-dinitrophenylhydrazine, had the characteristic aroma of guava. The material was also reduced by lithium aluminum hydride. These data, along with the ultraviolet and infrared spectra, suggested to them that the aroma constituent(s) was of the carbonyl and ester type. Although the infrared spectrum of the crude guava oil showed no hydroxyl absorption, a 3,5-dinitrobenzoate derivative was made which showed three spots by thin-layer chromatography. While several components were shown to be present, no specific identifications were made.

## EXPERIMENTAL

**Guava Puree.** Fruits of the Beaumont variety produced in Hawaii were sorted, washed, then comminuted with a Fitzmill,

Western Regional Research Laboratory, Agricultural Research Service, U.S. Department of Agriculture, Albany, Calif. 94710

using a 4-B screen. The milled fruit was passed through a paddle pulper (0.045 screen) to remove seeds, then passed through a paddle finisher (0.020 screen) to remove stone cells. The puree was then placed in 30-lb cans with polyethylene liners and was frozen at  $-23^{\circ}\text{C}$  and air-freighted to Albany, Calif., where it was stored at  $-29^{\circ}\text{C}$  until further use.

**Preparation of Essence.** The puree was thawed and distilled at 22 mm Hg ( $24^{\circ}\text{C}$ ) until 46% by weight had been stripped off. The resultant clear distillate was saturated with sodium chloride and extracted with three portions of ether. After drying the ethereal extract with anhydrous magnesium sulfate, the ether was removed with the aid of a solvent take-off apparatus. A strong guava aroma was associated with the resultant oil.

**Analytical Procedures.** Samples of the oil were chromatographed on a 1000-ft  $\times$  0.03-in. i.d. open-tubular column coated with Carbowax 20 M. The initial temperature was  $60^{\circ}\text{C}$  and programmed to  $200^{\circ}\text{C}$  at approximately  $1.3^{\circ}$  per min. The effluent from the column was directed into a membrane-type molecular separator (Black *et al.*, 1969) with a silicone membrane (type DSRE 2608 silicone polymer). A temperature of  $150^{\circ}\text{C}$  was maintained on the separator at all times. The "cracking pattern" was monitored on an oscilloscope and recorded on a high speed oscillographic recorder. Scans from 20 to 220 mass units were made in 1 sec on an EAI model 300 quadrupole mass spectrometer operating at approximately  $5 \times 10^{-6}$  Torr. The individual mass spectral patterns were then compared with known compounds and assignments made. In each case the assignment was verified by enriching the sample with the suspected compound (purchased from a commercial company) and obtaining an increase in peak height at the appropriate retention time, using a flame ionization detector.

## RESULTS AND DISCUSSION

A chromatogram of the guava oil is shown in Figure 1. The numbers above the peaks refer to the compounds listed in Table I and are those that have been identified. The first major point is the rather high concentration of hexanol (peak No. 9) and peak No. 10, *cis*-3-hexen-1-ol. These two

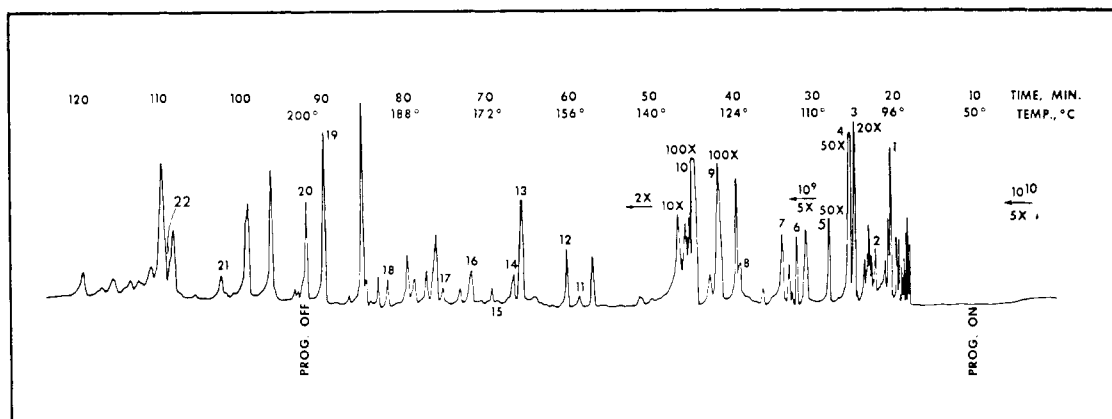


Figure 1. Programmed temperature gas chromatogram of guava puree extract using a 1000-ft  $\times$  0.03-in. i.d. open-tubular column coated with Carbowax 20M

Table I. Compounds Identified in Guava Puree

| Peak No. | Compound                      |
|----------|-------------------------------|
| 1        | ethyl acetate                 |
| 2        | pentanal                      |
| 3        | 2-methyl-1-propanol           |
| 4        | hexanal                       |
| 5        | 1-penten-3-ol                 |
| 6        | limonene                      |
| 7        | 1-pentanol                    |
| 8        | 3-hexen-1-yl acetate          |
| 9        | 1-hexanol                     |
| 10       | 3-hexen-1-ol                  |
| 11       | benzaldehyde                  |
| 12       | 1-octanol                     |
| 13       | caryophyllene                 |
| 14       | methyl benzoate               |
| 15       | 1-nonanol                     |
| 16       | $\alpha$ -terpineol           |
| 17       | citral                        |
| 18       | $\beta$ -phenyl ethyl acetate |
| 19       | $\beta$ -phenyl ethyl alcohol |
| 20       | $\beta$ -ionone               |
| 21       | methyl cinnamate              |
| 22       | cinnamyl acetate              |

compounds are almost ubiquitous to the plant kingdom and their presence in guava puree is in contrast to the findings of previous workers.

Other alcohols are also present but represent smaller amounts, e.g., 2-methyl-1-propanol (peak No. 3), 1-penten-3-ol (peak No. 5), 1-pentanol (peak No. 7), 1-octanol (peak No. 13), and 1-nonanol (peak No. 15) were found. It is interesting to note the presence of the terpene alcohol,  $\alpha$ -terpineol (peak No. 16) as well as the aromatic alcohol,  $\beta$ -phenyl ethyl alcohol (peak No. 19).

Another feature of the guava oil is the large number of aromatic compounds present, in particular the esters. A small amount of benzaldehyde (peak No. 11) is present. The fragrant compounds, methyl benzoate (peak No. 14),  $\beta$ -phenyl ethyl acetate (peak No. 18), methyl cinnamate (peak No. 21), and cinnamyl acetate (peak No. 22), are all present and probably contribute to the overall pleasant fruity aroma of the fruit. Methyl cinnamate has been found in strawberry

(McFadden *et al.*, 1965) while cinnamyl acetate has been reported (Schantz *et al.*, 1962) to be present in Cassia oil (Chinese cinnamon). Although the two aromatic esters are known to be present in some fruits, their distribution seems to be rather limited.

One compound which undoubtedly contributes to the floral aroma is peak No. 20,  $\beta$ -ionone. The low threshold of this material, coupled with its intense violet aroma, make it a strong contender for the most potent material in guava oil.

A small amount of limonene (peak No. 6) and citral (peak No. 17) are also present and make up additional examples of the terpenes found in guava. In addition, several sesquiterpene hydrocarbons have been classified; however only one, caryophyllene (peak No. 13), has been positively identified. Most of the remaining large peaks are sesquiterpenes, yet an assignment of structure was not possible. By smelling these components as they emerged from the gas chromatograph with a nondestructive detector, it was generally agreed that all of them were rather bland and contributed little if anything to the overall aroma.

In general, practically all classes of compounds, with the exception of nitrogen and sulfur, are present which have been found in volatile essence of fruits (Nursten and Williams, 1967). For instance, the alcohols remind one of the high concentration found in grapes, while the terpenes and sesquiterpenes are reminiscent of the citrus family. The aromatic compounds and especially the esters are what one might expect from some of the spices.

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Received for review February 27, 1970. Accepted April 27, 1970. Reference to a company or product name does not imply approval or recommendation of the product by the U.S. Department of Agriculture to the exclusion of others that may be suitable.