

oxidized by permanganate, gave the respective tri-basic acids XXX and XXXI. It was not possible to degrade III to XXI, nor to convert XXI into III, although many attempts were made. Nor

was it possible to degrade the tribasic acid XXX into XXXI. Many new compounds, incidental to these investigations, have been described.

MINNEAPOLIS, MINNESOTA RECEIVED NOVEMBER 4, 1942

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, SCHOOL OF TROPICAL MEDICINE OF THE UNIVERSITY OF PUERTO RICO, UNDER THE AUSPICES OF COLUMBIA UNIVERSITY]

## Puerto Rican Fatty Oils. II. The Characteristics and Composition of *Guanábana* Seed Oil

BY CONRADO F. ASENJO AND JOSÉ A. GOYCO

The Puerto Rican variety of *guanábana* (*Annona muricata* L.) is an ovoid or somewhat heart-shaped fruit, 15 to 20 cm. long and 9 to 12 cm. broad. The skin is green with isolated curved fleshy spinules; its pulp white and juicy and pleasantly subacid. Imbedded in the pulp is found a large number of seeds, dark brown in color and oblong-elliptic in shape, measuring about 1.6 cm. in length and 1 cm. in breadth. Thirty-three per cent. of the seed is husk and 67% kernel. The common English name of the fruit is soursop.

Locally, the pulp of the fruit has been used for a very long time in the preparation of a deliciously refreshing drink, which is now being canned for commercial purposes. For this reason large amounts of seeds are easily available for study. These seeds are a fairly promising source of oil and yield by acetone extraction 23.86%. The percentage obtained by hot expression (110°) is somewhat lower. This paper presents the results obtained from the study of a sample of hot expressed oil which was kindly supplied to us by the Division of Investigation and Industrial Development of the Department of Agriculture and Commerce of Puerto Rico.

With the exception of a short report from the Food Testing Laboratory of Surinam,<sup>1</sup> in which the iodine, saponification, and Maumené numbers were recorded for a sample of *Annona muricata* L. seed oil, no previous chemical study of this oil could be found in the literature. The present investigation is therefore concerned with the determination of the characteristics and with the isolation, identification, and quantitative estimation of the acids present in the expressed oil.

**Physical and Chemical Examination.**—The oil was yellowish brown in color; the sample was

odorless and had a bland taste. Its chemical and physical properties were determined by the usual procedures<sup>2</sup> with the results given in Table I.

TABLE I  
CHEMICAL AND PHYSICAL CHARACTERISTICS OF EXPRESSED  
*Guanábana* SEED OIL

|   |        |
|---|--------|
| Specific gravity 25°/25°                | 0.9178 |
| Refractive index 20°                    | 1.4709 |
| Iodine number (Hanus)                   | 87.79  |
| Saponification no.                      | 197.0  |
| Acid value                              | 2.29   |
| Acetyl value                            | 12.56  |
| Reichert–Meissl no.                     | 0.81   |
| Polenske no.                            | .56    |
| Unsaponifiable residue, %               | 1.02   |
| Soluble acids, %                        | 0.37   |
| Insoluble acids, %                      | 91.90  |
| Saturated acids, % (corrected)          | 22.02  |
| Unsaturated acids, % (corrected)        | 70.02  |
| Iodine no. of unsaturated acids         | 105.1  |
| Saponification no. of unsaturated acids | 202.1  |

The iodine number shows little unsaturation, and the Reichert–Meissl and Polenske numbers show small amounts of glycerides of volatile acids.

**Examination of the Unsaturated Acids of the Expressed Oil.**—Bromination of the unsaturated acids in the usual manner showed the absence of linolenic acid. Tetrabromostearic acid melting at 114° was obtained, showing the presence of linoleic acid. The percentages of the linoleic and oleic acids were calculated using the iodine number of the unsaturated acid.<sup>3</sup>

**Examination of the Saturated Acids of the Expressed Oil.**—The saturated acids were esterified by the Twitchell method, as modified by Hilditch,<sup>4</sup> and the mixed methyl esters obtained

(2) Association of Official Agricultural Chemists, "Methods of Analysis," Washington, D. C., 4th ed., 1935, pp. 404–429.

(3) J. Lewkowitzsch, "Chemical Technology and Analysis of Oils, Fats and Waxes," 6th ed., Macmillan and Co., Ltd., London, 1921, p. 574.

(4) T. P. Hilditch, "The Chemical Composition of Natural Fats," John Wiley and Sons, New York, N. Y., 1941, pp. 371–372

(1) Anonymous, *Olieu en Vellen*, No. 30, 387–389 (1920), through *Chem. Abs.*, 14, 1230 (1920).

(42 g.) fractionally distilled *in vacuo* with the results given in Table II.

TABLE II

RESULTS OF ANALYSES OF FRACTIONS OBTAINED BY DISTILLING METHYL ESTERS OF SATURATED ACIDS OF EXPRESSED *Guanábana* SEED OIL

| Fractions                        | 1       | 2       | 3       | 4       |
|----------------------------------|---------|---------|---------|---------|
| Temperature, °C.                 | 155-165 | 165-171 | 171-179 | 179-184 |
| Pressure, mm.                    | 3.5     | 3.5     | 3.5     | 4.0     |
| Iodine no.                       | 2.55    | 4.26    | 6.42    | 10.51   |
| Sap. no. esters satd. acids      | 208.9   | 203.4   | 200.0   | 195.9   |
| Esters unsat. acids, %           | 2.55    | 4.26    | 6.42    | 10.51   |
| Esters satd. acids, %            | 97.45   | 95.74   | 93.58   | 89.49   |
| Mean mol. wt. esters satd. acids | 268.0   | 275.2   | 279.8   | 285.8   |
| Composition of methyl esters     |         |         |         |         |
| Myristic                         | 7.47    | ...     | ...     | ...     |
| Palmitic                         | 84.76   | 75.05   | 58.81   | 38.05   |
| Stearic                          | ...     | 15.80   | 30.10   | 47.00   |

There was practically no residue after the vacuum distillation. The iodine and saponification numbers of the different fractions were determined and the mean molecular weights of the esters calculated according to the Baughman and Jamieson<sup>5</sup> method. These results are recorded in Table II. To confirm the data in Table II the acids were isolated from the different fractions and fractionally crystallized from 95% ethyl alcohol.

**Myristic Acid.**—An acid melting at 54.6° was obtained from fraction 1. An anilide prepared from this same acid had a melting point of 84.0°; mix melting point, 83.5°. These two melting points, corresponding very closely to those of myristic acid and its anilide, were considered evidence of myristic acid.

**Palmitic Acid.**—Fractions 1, 2, 3 and 4 gave an acid melting at 62.9°. The anilide prepared

(5) Baughman and Jamieson, *THIS JOURNAL*, **42**, 157 (1920).

from this acid had a melting point of 90.0°; mix melting point, 89.8°, which two melting points corresponded very closely to those of palmitic acid and its anilide and were considered evidence of palmitic acid.

**Stearic Acid.**—Fractions 2, 3 and 4 yielded an acid melting at 69.1°. The anilide prepared from this acid had a melting point of 93°; mix melting point, 92.8°. As these two melting points corresponded very closely to those of stearic acid and its anilide, they were considered evidence of stearic acid.

TABLE III

UNSATURATED AND SATURATED ACIDS IN EXPRESSED *Guanábana* SEED OIL

| Acids    | In unsatd. acids, % | In satd. acids, % | In original oil, % | Glycerides in oil, % |
|----------|---------------------|-------------------|--------------------|----------------------|
| Linoleic | 16.63               | ...               | 11.64              | 12.11                |
| Oleic    | 83.37               | ...               | 58.38              | 61.01                |
| Myristic | ...                 | 1.45              | 0.32               | 0.34                 |
| Palmitic | ...                 | 73.55             | 16.20              | 16.99                |
| Stearic  | ...                 | 25.00             | 5.51               | 5.76                 |

**Acknowledgment.**—This work was supported by a grant from the Department of Agriculture and Commerce of Puerto Rico. Our thanks are due to Dr. D. H. Cook, head of the Department of Chemistry, for his constant encouragement and coöperation.

### Summary

The characteristics and composition of hot expressed *guanábana* (*Annona muricata* L.) seed oil have been determined.

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RECEIVED OCTOBER 19, 1942

[CONTRIBUTION FROM THE WESTINGHOUSE RESEARCH LABORATORIES]

## Ionization and Dissociation by Electron Impact: Butene-1

By D. P. STEVENSON<sup>1</sup>

In this communication the results of a mass-spectrographic investigation of the ionization and dissociation of butene-1 by electron impact are reported. The instrument and general technique have been described in an earlier article.<sup>2</sup> The sample of butene-1 was a Phillips Petroleum Company product which was given to us by the Gulf Oil Company Research Laboratories.

(1) Westinghouse Research Fellow, 1940-1942. Present address: Shell Development Company, Emeryville, California.

(2) D. P. Stevenson and J. A. Hipple, *THIS JOURNAL*, **64**, 1588 (1942).

TABLE I

APPEARANCE POTENTIALS OF VARIOUS IONS IN THE MASS SPECTRUM OF BUTENE-1

| Ion (X <sup>+</sup> )                      | A(X <sup>+</sup> ), e. v. | Process   |
|--|---------------------------|---|
| C <sub>4</sub> H <sub>9</sub> <sup>+</sup> | 9.65 ± 0.1                | 1-C <sub>4</sub> H <sub>9</sub> → C <sub>4</sub> H <sub>9</sub> <sup>+</sup> + e <sup>-</sup> |
| C <sub>4</sub> H <sub>7</sub> <sup>+</sup> | 11.07 ± .1                | C <sub>4</sub> H <sub>7</sub> <sup>+</sup> + H + e <sup>-</sup>                               |
| C <sub>3</sub> H <sub>9</sub> <sup>+</sup> | 11.65 ± .1                | C <sub>3</sub> H <sub>9</sub> <sup>+</sup> + CH <sub>3</sub> + e <sup>-</sup>                 |
| C <sub>3</sub> H <sub>7</sub> <sup>+</sup> | 11.7 ± .2                 | C <sub>3</sub> H <sub>7</sub> <sup>+</sup> + CH <sub>3</sub> + e <sup>-</sup>                 |
| C <sub>3</sub> H <sub>5</sub> <sup>+</sup> | 13.8 ± .2                 | C <sub>3</sub> H <sub>5</sub> <sup>+</sup> + ? + e <sup>-</sup>                               |
| C <sub>2</sub> H <sub>9</sub> <sup>+</sup> | 11.97 ± .1                | C <sub>2</sub> H <sub>9</sub> <sup>+</sup> + C <sub>2</sub> H <sub>3</sub> + e <sup>-</sup>   |
| C <sub>2</sub> H <sub>7</sub> <sup>+</sup> | 13.6 ± .3                 | C <sub>2</sub> H <sub>7</sub> <sup>+</sup> + C <sub>2</sub> H <sub>3</sub> + e <sup>-</sup>   |
| CH <sub>3</sub> <sup>+</sup>               | < 20                      | CH <sub>3</sub> <sup>+</sup> + ? + e <sup>-</sup>   |