not found in the corresponding residue from the Michigan sirup, and it is very probably an oil used in the kettles to stop foaming.

Conclusion

Investigation of the flavor of maple sirup showed that it depends to a great extent on an unstable phenolic substance which is associated with a crystalline aldehyde melting at 74–76° and similar in odor and properties to vanillin. Maple sirup may contain minute quantities of other aldehydic substances which influence the flavor.

WASHINGTON, D. C.

[CONTRIBUTION FROM THE FOOD RESEARCH DIVISION, BUREAU OF CHEMISTRY AND SOILS, UNITED STATES DEPARTMENT OF AGRICULTURE]

THE ACIDS OF FIGS

BY E. K. NELSON

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No record has been found in the literature regarding the identity of the acids of figs. Bigelow and Dunbar[1] in their review of the literature on fruit acids do not mention the fig. Condit and Creuss[2] state that fresh Kadota figs contain from 19 to 24% of sugar and from 0.1 to 0.44% of acid, calculated as citric.

In an investigation of sour figs and figs affected with internal rot, B. J. Howard, of this Bureau, collected samples of normal and diseased figs near Fresno, California, during the summer of 1927. They included Adriatic "black necks," normal Adriatics, Calimyrna figs affected with internal rot and normal Calimyrnas. Part of these samples, when dried, afforded the material for an investigation of the acids in figs.

Experimental

Approximately two kilograms of each kind of figs was ground to a paste. The paste was disintegrated in 1 gallon of water at 60°, digested on a steam-bath for an hour, diluted with an equal volume of alcohol and strained through a linen bag. The residue was pressed dry, soaked in hot dilute alcohol and re-pressed. The fig extract was concentrated to a sirup in a vacuum pan, the distillate being collected for the determination of volatile acids. After being diluted with water and acidified with sulfuric acid, the sirup was extracted with ether to recover ether soluble acids. Only fig fat and small quantities of stearic and oleic acids were found in this ether extract.

The diluted sirup was then precipitated with a moderate excess of lead subacetate. After filtration, the lead precipitate was suspended in water and a stream of carbon dioxide was allowed to bubble through it for some time in order to break up lead sugar compounds. After it was filtered and washed, the lead precipitate was decomposed with sulfuric acid and the excess of acid was removed with the equivalent quantity of barium hydroxide. The acid solution was evaporated to dryness on the steam-bath

[1] Bigelow and Dunbar, *J. Ind. Eng. Chem.*, **9**, 767 (1917).

[2] Condit and Creuss, Calif. Agr. Expt. Sta., *Bull.*, **436**, 1–45 (1927).

and esterified by being refluxed for five hours with absolute alcohol containing 2.5% of hydrochloric acid. After distilling off the alcohol, the esterification process was repeated. The crude esters, in ether solution, were washed with sodium hydroxide solution, the ether was evaporated and the esters, after being weighed, were subjected to fractional distillation at 10 mm.

The hydrazides prepared from the fractions were used for the identification of the respective acids.

The barium salt of the volatile acids from 2282 g. of Adriatic black necks weighed 15.71 g. It was fractionally crystallized and the barium in each fraction was determined. Fraction 1 contained 53.9% of barium; Fraction 2, 53.89%; Fraction 3, 53.95%; and Fraction 4, 53.87%; calcd. for barium acetate, barium = 53.72%. The volatile acid is therefore acetic acid. No ether soluble acids other than small quantities of stearic and oleic from the fig fat were identified.

The crude ester of the non-volatile acid, 9.75 g., when distilled at 10 mm. at 170–171° afforded 8.1 g. of pure ester. It had the boiling point of triethyl citrate and gave the characteristic hydrazide, melting at 104–106° in its hydrated form and at 147° when seeded and in the anhydrous form.

In the tests with normal Adriatic figs, 1925 g. of fruit yielded 1.11 g. of barium salt of volatile acids, corresponding to 0.27 g. of acetic acid per kilo. The silver salt was analyzed and 64.44% of silver was found; calcd. for silver acetate, 64.45%. The ethyl citrate, boiling at 170–171° at 10 mm., weighed 8.93 g., corresponding to 3.2 g. of citric acid per kilo, and afforded the characteristic hydrazide.

Two kilos of Calimyrna figs affected with internal rot yielded 2.4 g. of the barium salt of volatile acids, corresponding to 0.56 g. of acetic acid per kilo. It was shown to be acetic acid by the analysis of the silver salt (64.15% of silver). From the ester of the non-volatile acids a small quantity was obtained distilling under 170° at 10 mm., and from the hydrazide of this, several times recrystallized, a small quantity of malic acid hydrazide which melted at 177–179° was separated; a mixture of this with pure malic hydrazide showed no lower melting point. The main portion of the ester, 9.51 g., distilled at 170–171°, corresponding to 3.3 g. citric acid per kilo and affording the characteristic hydrazide.

Normal Calimyrnas, 2.066 kilos, afforded 1.15 g. of the barium salt of volatile acids, corresponding to 0.26 g. of acetic acid per kilo. The silver salt contained 64.6% of silver.

In the esters of the non-volatile acids, a small quantity of malic acid was identified by the hydrazide. Nine and five-tenths grams boiling at 170–171° at 10 mm. corresponded to citric acid, 3.5 g. per kilo.

Conclusion

Acetic and citric acids were found in Adriatic figs, and acetic, citric and a small quantity of malic acids were identified in Calimyrna figs.

It was found that Adriatic black neck figs contained more than 10 times as much free acetic acid as the normal Adriatic figs. Less citric acid was found than in the normal figs.

Normal Calimyrna figs contained 0.26 g. per kilo of free acetic acid and 3.5 g. of citric acid, besides a small quantity of malic acid.

In Calimyrna figs affected with internal rot the free acetic acid amounted to 0.56 g. per kilo, and the citric acid amounted to 3.3 g. per kilo.

WASHINGTON, D. C.