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## Acetic Acid, a Major Volatile Constituent of Brown Sugar: Its Origin and Measurement

Mary A. Godshall\* and Anthony J. DeLuca, II

Acetic acid was identified as the major volatile constituent of commercial brown sugars. These sugars contained levels ranging from 31 to 827 ppm. The source of the acetic acid was found to be bacterial action in recycled sweet waters containing low levels of sucrose. Sugars manufactured without these waters had low to no detectable acetic acid levels. The method of analysis used—direct gas chromatography of sugars on the Dupuy inlet—provided a rapid, semiquantitative result. A high level of variability in some of the sugars was attributed to uneven distribution of acetic acid in these sugars. Other volatile constituents due to bacterial action were methanol, ethanol, acetaldehyde, and diacetyl.

Commercial brown sugar, a product of sugar refining that is known as "soft" sugar in the industry, is characterized by a unique odor and flavor. During an investigation of the volatiles responsible for this odor and flavor, it was noted that acetic acid was found in all samples and was present in a much higher concentration than any other volatile constituent (Godshall and Roberts, 1980).

Since the sugars being analyzed were from different sources around the world, representing various processing methods, it was of interest to determine acetic acid levels and sources.

One possibility for the origin of acetic acid was thermal or chemical degradation of sucrose during the manufacture of brown sugar. This seemed unlikely in view of the relatively mild conditions that normally exist in refining. A second obvious possibility was microbiological degradation of sucrose. This required that some processing parameter exist in common in all the refineries producing the brown sugar so that refineries in such diverse places as Canada, Australia, England, and Louisiana could produce brown sugars with high acetic acid levels.

Traditional brown sugars fall into two basic categories, "boiled" and "coated", which represent fundamentally distinct methods of production. A boiled soft sugar is crystallized from a dark refined syrup. The color of the sugar reflects the syrup from which it was crystallized. A coated sugar is a refined sugar that has been sprayed with a thin film of highly colored syrup or molasses to give it the characteristic color.

A survey of processes in use during the manufacture of brown sugar showed one process in common: all of the producers made use of bone char to decolorize various process liquors, and the water used to wash residual sugar off bone char prior to its regeneration was recycled for the manufacture of brown sugar. Other clarification and decolorization processes varied, but this procedure was used by all manufacturers of these sugars. This "sweet water" or "char washing" contains from 5 to 20% sucrose as well as other nutrients and seemed to be a possible source for the microbiological activity that could lead to acetic acid production. In addition, water used to wash sugar out of some vessels was also recycled.

A third type of colored sugar, known as "turbinado" sugar, is also available in American markets. This type of sugar is essentially a raw sugar that has been subjected

Table I. Description of Sugars Used in Acetic Acid Determination

type of sugar	no. of samples	code
light or medium brown, boiled	8	A
dark brown, boiled	6	B
light brown, coated	3	C
dark brown, coated	3	D
turbinado	3	E
specialty sugars		
amorphous, processed with sweet water	2	F
amorphous, processed without sweet water	1	G
light brown, boiled, processed without sweet water	1	H
demerara	1	I

to a cleanup procedure consisting of centrifugation in the presence of syrup or water, resulting in pale brown to golden crystals.

The analysis of underivatized acetic acid by gas chromatography presents many problems due to the acid's highly polar nature, which can lead to peak tailing and ghosting (Trombella and Ribeiro, 1980). A method was needed that would require minimum sample preparation and avoid the necessity of any type of solvent extraction. We decided to investigate the Dupuy external inlet (Legendre et al., 1979) for rapid quantitation of acetic acid. This inlet was developed for direct gas chromatography of volatile constituents in foods and had already been successfully used to identify volatile constituents in molasses (Godshall et al., 1980).

### EXPERIMENTAL SECTION

**Source of Brown Sugars.** Twenty-five brown sugars and three turbinado sugars were contributed by refineries from the United States, Canada, England, and Australia. The sugars analyzed are categorized in Table I. All were of cane sugar origin. Low-purity (i.e., low in sucrose) sweet waters were contributed by a U.S. refinery. These consisted of water from char washing and water from vessel washing. Samples were preserved and chilled after sampling so that no further deterioration or microbiological action would occur.

**Identification of Acetic Acid.** The identification of acetic acid in brown sugar was confirmed by combined GC/MS using the Dupuy inlet. The conditions of mass spectrometry have been described previously (Godshall et al., 1980). The identity of acetic acid was confirmed by MS with some initial difficulty. The Dupuy inlet was equipped with a Na<sub>2</sub>SO<sub>4</sub> condenser through which the

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