Odor in Granulated Beet Sugar

R. F. OLSON[1]

Received for publication May 21, 1958

A persistent problem in the manufacture of beet sugar has been the inherent odor of the finished product. This odor, no matter how slight, is a subject of concern and should be dealt with accordingly. Hungerford (1)[2] reported on the odor problem and how difficult it was to obtain precise and reliable information.

The objective was to study the odor in granulated sugar and attempt to find what compounds in granulated sugar correlate with the odor. Odor is very hard to define, yet the odor in granulated sugar has been described as musty, sour, sweet, metallic and so on. We are aware that none of the above odors describes a specific compound.

The procedure included evaluations by an odor panel, whose findings were later correlated with chemical studies of a hydrochloric acid eluate prepared from the test sugars. The hydrochloric acid eluate was made by a procedure which included the use of ion-exchange resin to separate some of the nitrogenous constituents from sugar solutions. Dowex 50 cation exchange resin, H^+ form was used. A 1000 gram sample of sugar was made up to approximately three liters and allowed to flow through 35 ml of the resin at the rate of 25 ml per minute. After the sugar solution had passed through the resin, the column was rinsed with distilled water to remove the sugar. The water eluate was discarded and then 50 ml of five percent hydrochloric acid solution was placed in the column and this eluate was collected for subsequent chemical analysis.

All of the test sugars were evaluated for odor by a panel consisting of five people. In these tests all sugars had at least a slight odor. The sugars were placed in clean, sterile, four-ounce jars and covered with an odorless screw-type cap. The comparisons were made at room temperature, approximately 25 degrees Centigrade. Each jar was gently shaken, the cover lifted slightly and the jar brought near the nose to detect the odor.

The test consisted of nine sugars, with the sugars arranged in groups of three. After evaluating the nine sugars in their respective groups a fourth group was made up containing a sugar from each of the other three groups. A comparison then could be made among the nine sugars. The panelists were asked to ascertain which sugars in each group had the most pronounced and the least pronounced odor. The panel repeated the testing three times over a period of a week.

---

[1] Research Chemist, American Crystal Sugar Company, Rocky Ford, Colorado.

[2] Numbers in parentheses refer to literature cited.

Table 1.—Rating of Sugars.

Group Number	Rating of Odor by Panelists										Numerical Rating
	Panelist 1			Panelist 2			Panelist 3			Panelist 4			Panelist 5			
	Tests			Tests			Tests			Tests			Tests			
	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3	
1	B	A	B	B	B	B	B	A	B	B	B	B	A	B	B	B = 2.73
	A	C	A	C	A	C	A	B	C	A	A	C	B	A	A	A = 1.93
	C	B	C	A	C	A	C	C	A	C	C	A	C	C	C	C = 1.38
2	F	F	E	D	F	E	F	D	D	F	F	F	E	D	E	D = 2.07
	E	E	F	E	D	D	D	E	E	D	D	D	D	E	D	F = 2.00
	D	D	D	F	E	F	E	F	E	E	E	E	F	F	F	E = 1.93
3	G	H	H	H	H	H	(G)	I	H	G	H	G	H	G	G	H = 2.47
	I	G	G	I	(I)	I	(H)	H	I	H	G	G	G	H	H	G = 2.03
	H	I	I	G	(G)	G	(I)	G	G	I	I	I	I	I	I	I = 1.50
4	E	C	C	C	C	C	C	C	C	C	C	(C)	C	G	E	C = 2.67
	C	E	E	G	E	E	G	E	E	E	(E)	(E)	E	E	C	E = 1.97
	G	G	G	E	G	G	E	G	G	G	(G)	(G)	G	C	G	G = 1.36

The first listed sugar in each test had most pronounced odor. Brackets indicate panelist found no difference in odor among the samples. For the numerical rating most pronounced odor 3, intermediate = 2 and least pronounced odor = 1.

Table 1 shows the odor rating and the variation in the evaluation of odor in granulated sugar samples.

After evaluating the sugars according to their odor the task was then to identify by chemical means the constituents in the hydrochloric acid eluate and to ascertain which compounds correlated with odor. The search was begun with the listing of the characteristics of an odoriferous compound. The compound should have a low threshold value; it should be able to be synthesized or be the result of a breakdown of another compound during the processing of sugar beets. Amines have these general characteristics and are either gasses or fairly volatile liquids of moderate molecular weight having a pronounced odor similar to ammonia, but of a less pungent and more fish-like odor. Several of the amines are: methylamine, dimethylamine, trimethylamine, ethylamine, diethylamine, n-butylamine and n-amylamine. After concentrating the hydrochloric acid eluate to a volume suitable for chromatographic studies, the eluate was spotted on Whatman No. 4 paper and allowed to develop by a descending method in an isopropanol solvent. Paper chromatographic studies indicate that methylamine reacted similarly with a compound detected in the hydrochloric acid eluate. The Rf of the unknown coincided with the Rf of the methylamine.

Utilizing the method of Rosenblatt, Hlinka and Epstein (2) further tests were run on the eluates. This method affords the determination of primary amines in the presence of other compounds. With the knowledge gained from paper chromatographic and photo-colorimetric work the compound in the eluate was identified as methylamine.

Table 2.—Rating of Sugars.

Sugar Samples	Odor, Methylamine and Betaine		
	[1] Numerical Rating of Odor by Panelists	P.P.M Methylamine	P.P.M Betaine
1	2.30	0.10	16.5
2	2.50	0.12	19.5
3	1.20	0.07	7.5
4	2.44	0.14	21.5
5	2.28	0.10	12.8
6	1.28	0.10	8.5
7	2.60	0.10	20.0
8	2.00	0.07	13.0
9	1.40	0.09	17.0

[1] Numerical rating for this group of sugars was obtained in the same manner as for the group of sugars reported in Table 1.

Another group of sugars were tested and Table 2 shows the comparison between the odor and chemical evaluations of granulated sugar samples.

From paper chromatographic studies it was found that some of the amino acids were present in the granulated sugar. In the testing odor was not discernable from the individual amino acids. Betaine was also present in appreciable amounts in granulated sugar. Betaine was determined by precipitation as the Reinecke salt. The Reinecke-betaine salt was dissolved in 70 percent acetone and read in a colorimeter at a wavelength of 515 millimicrons using known amounts of betaine hydrochloride as standards.

Table 2 shows the comparison of odor, methylamine and betaine concentrations.

Some preliminary tests indicated the presence of methylamine in the centrifugal wash water and in the cation regenerant solution of an ion-exchange system. Molasses has been found to contain methylamine. Another amine was detected in the distillate resulting from distilling an alkaline solution of betaine.

Whether the methylamine was a result of the degradation of a higher compound or was synthesized during the factory process is a question yet to be resolved. Methylamine may be readily synthesized from formaldehyde and ammonium chloride in the presence of heat. Methylamine as previously stated may be due to the degradation of higher compounds, as is dimethylamine and trimethylamine during the distillation of sugar beet residues which contain betaine.

Summary

The odor in beet sugars was evaluated organoleptically by a panel of five people. Methylamine and betaine were found to be present in these sugars. Methylamine and betaine correlate with odor.

Literature Cited

(1) HUNGERFORD, E. H. 1957. Factors affecting odor in beet sugar. Jour. Amer. Soc. Sugar Beet Tech. IX (5) :377-380.

(2) ROSENBLATT, DAVID H., HLINKA, PETER, and EPSTEIN, JOSEPH. 1955. Use of 1, 2-naphthoquinone-4-sulfonate for the estimation of ethylenimine and primary amines. Analytical Chemistry. Vol. 27, No. 8.