

The Relation of Beet Molasses Composition to True Purity

Part I. Composition

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Received for publication August 10, 1967

Molasses formation from beet sugar liquors has had the attention of technologists for more than 100 years. Its formation in the factory is dependent on many factors but probably the most important variables are the effect of some non-sugars in decreasing the rate of crystallization and others in increasing the solubility of sucrose in the molasses solution. Since equilibrium between crystallized sugar and sugar in solution is not reached in industrial molasses because of the time necessary to reach this equilibrium, factory molasses contains more sugar than a completely exhausted molasses. Molasses in this paper refers to factory discard molasses.

In an attempt to determine what chemical factors were related to variations in molasses purity a composition study was made of molasses produced in the different beet sugar areas of the United States. Samples were selected from straight house and Steffen factories and analyzed for a large number of compounds and groups of compounds. The analytical methods used and the data obtained are presented in Part I of the paper while the statistical evaluation of the data is presented in Part II.

Experimental

Molasses samples were obtained during the 1956 campaign from a cross section of the beet sugar producing areas of the United States. Straight house samples were obtained from Betteravia, Clarksburg and Manteca, California; Brighton, Eaton and Swink, Colorado; Rupert, Idaho; Carrollton, Michigan; Crookston and Moorhead, Minnesota; Sidney, Montana; Fremont, Ohio; Belle Fourche, South Dakota; West Jordan, Utah; Toppenish, Washington, and Green Bay, Wisconsin. Spring and fall campaign samples were obtained from Betteravia. Swink ordinarily operates as a Steffen house, but the sample included in this study was produced from Kansas beets while the factory was operating as a straight house. Steffen house samples were

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obtained from Alvarado, Oxnard, Spreckles, Tracy and Woodland, California; Loveland, Colorado; Twin Falls, Idaho; Mason City, Iowa; Grand Island and Scottsbluff, Nebraska; Nyssa, Oregon; and Torrington, Wyoming. A spring and fall campaign sample was obtained from Oxnard. A sample of molasses was obtained from Johnstown, Colorado as representative of the barium process and a sample of molasses produced by ion exchange in 1955 was obtained from Layton, Utah.

Samples were collected by factory personnel and shipped to this laboratory for analysis. An attempt was made to obtain composite samples representing at least a month's operation. After receipt of the samples (one to two gallons) they were heated, thoroughly mixed, subdivided into 200 g samples for analysis and stored at 34° F. Samples were removed later from cold storage, heated, stirred to insure uniformity, and a weighed sub-sample taken for analysis.

Total solids were determined in duplicate by taking aliquots from a diluted sample that would give a solid residue of 300-600 mg, placing them in tared glass weighing dishes, 50 mm diameter, and heating them in a forced draft oven at 60° C for about six hours to a thick syrup. The dishes were then placed in a vacuum oven and the pressure reduced until the syrup foamed and filled the dish. After 30 hours drying at 60° C the pressure was restored with air slowly bubbled through sulfuric acid. The dishes were placed in a desiccator and weighed when cool. Since these were beet molasses samples with relatively low reducing sugar content there was a negligible loss in weight from decomposition due to prolonged heating. Cane molasses decomposes to a much greater extent on heating than beet molasses (4,5)².

To obtain representative samples of molasses for sugar analyses 50 g were weighed in a beaker, washed into a liter volumetric flask with water and sufficient 95% alcohol added to make the final volume approximately 80% alcohol. After heating and cooling, the volume was adjusted to the mark and the contents were allowed to stand overnight. A 100 ml aliquot of the clear solution was taken up in a pipet without filtration. Sucrose and reducing substances were determined by the Munson-Walker official method (1) (Secs 6.2 (b), 6.74 (a), 6.77, 6.78 (b) [2], 25.35, 29.39, 29.40 and 42.11). The cuprous oxide was collected and weighed in a porcelain filtering crucible. Sucrose was calculated as 0.95 times the total reducing sugar after invertase inversion, less reducing substances before inversion, less 0.72 times the raffinose content. Sucrose = 0.95 [Total Reducing

²Numbers in parentheses refer to literature cited.

Sugar—(Reducing Sugars +0.72 Raffinose)]. The factor 0.72 for the reducing power of anhydrous raffinose after invertase inversion was determined in this laboratory.

Raffinose was determined separately by paper chromatography using the method of Bevenue and Williams (2).

Milliequivalents of anions, total weight of anions and average equivalent weight of anions were determined by an ion exchange procedure (8). Only those compounds that pass through the cation exchanger and are adsorbed on the anion exchanger and subsequently eluted with ammonium hydroxide are included as anions. Amino acids and a few other amphoteric compounds are retained on the cation exchanger. Such compounds, although having acidic characteristics, are not included in calculations of anions.

Ash was determined on a diluted molasses sample charred in a platinum dish under an infrared heater and then heated in a muffle furnace at 550° C overnight, cooled and weighed.

Alkalinity of the ash was determined on the total sample of ash. The residue in the Pt dish was treated with 25 ml of 0.1 N hydrochloric acid, heated on a steam bath, cooled, and the excess acid titrated with 0.1 N sodium hydroxide to pH 4.0.

Total halides calculated as chloride was determined gravimetrically by the AOAC method (1) (Secs 6.5, 6.6).

Total nitrogen was determined by the AOAC Kjeldahl method (1) (Sec 2.23). The original molasses and ion exchange fractions containing nitrate were treated according to the improved procedure for samples containing nitrate (1) (Sec 2.24).

Determinations of other types of nitrogen were made as follows: nitrate according to the method of Johnson and Ulrich (6), ammonia by the AOAC method (1) (Sec 2.25); amide by the method of Winton and Winton (9), amino nitrogen by the method of Peters and Van Slyke (7), and betaine by the colorimetric method of Focht and Schmidt (3).

Potassium, sodium and calcium were determined by flame photometry with a Model 9200 Beckman³ flame photometer attachment with a Model 4020, medium bore hydrogen-oxygen atomizer-burner for the Beckman Model DU Spectrophotometer. An attempt to determine magnesium by the method was made, but the values were very low and, because of the interference caused by potassium, sodium and calcium, the results were not considered reliable.

³ 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.

A Beckman Model H-2 pH meter was used for the pH determinations. They were made at 25° C on undiluted molasses and on samples diluted to 10% solids.

To obtain a better idea of nitrogen distribution in molasses, an ion exchange separation was made of the classes of nitrogen compounds according to the diagram in Figure 1 and the fractions analyzed for various groups of compounds. Fifty grams of molasses (A) was diluted to 200 ml and passed through a column of 400 ml of Dowex 50-X8, 20-50 mesh, H⁺ form⁴. After loading, the column was washed with distilled water (a) until the effluent pH was 3.2. The load and effluents were combined and made to 1,500 ml (B). Aliquots of B were taken for determinations of milliequivalents of anions. The results were not corrected for large molecules that were adsorbed on therein but not eluted. One liter of B was passed through a column of 400 ml of Duolite A-4, 10-50 mesh, base form⁵. The load was followed by two liters of water wash (b). The combined load and wash effluent was evaporated to 500 ml and aliquots analyzed for "neutral nitrogen". Neutral nitrogen is defined as the nitrogen not retained on Dowex 50 or Duolite A-4 under the conditions of the fractionation.

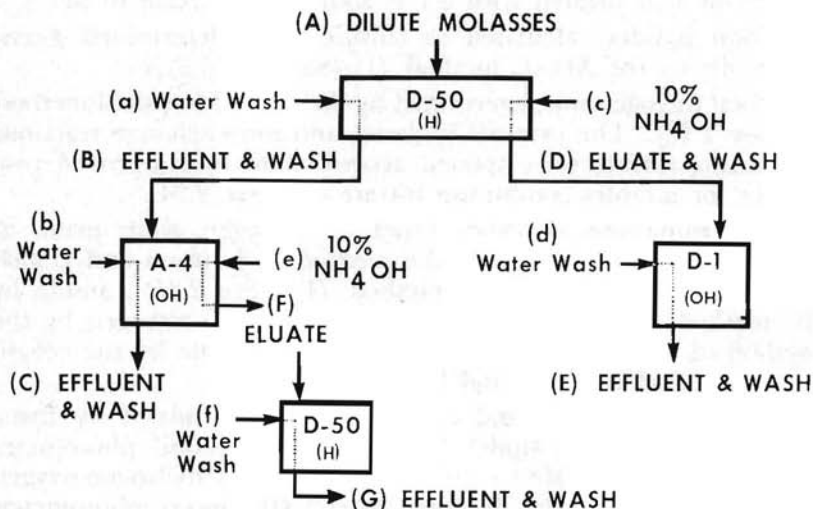


Figure 1.—Procedure for separating molasses nitrogen components.

After the water wash (b) the column of Duolite A-4 was eluted with 3 liters of 10% ammonium hydroxide (e). The ammonia was evaporated from the eluate (F) by boiling. After

⁴ Dow Chemical Co., Midland, Michigan.

⁵ Diamond Alkali Co., Redwood City, California.

the ammonia was removed, the solution was treated with portions of dilute sodium hydroxide to displace ammonium ions. Boiling was continued until all ammonia was removed. This was shown by the absence of ammonia odor or drop in pH after further slight addition of sodium hydroxide and further boiling. The solution (F) was diluted to 200 ml and passed through a column containing 100 ml of Dowex 50, X-8, 50-100 mesh, H⁺ form. The load was followed by a water wash (f) until the effluent volume (G) of load and wash was one liter. Aliquots of this acid solution were titrated in tared beakers with 0.1 N sodium hydroxide. The solution was evaporated and the beakers were weighed. The average equivalent weight of anions was calculated from the weight of sodium salt and equivalents of anions (8). Other aliquots of G were taken for the determination of acidic nitrogen.

The water wash (a) of the first Dowex 50 column was followed by 3 liters of 10% ammonium hydroxide (c). This procedure leaves nearly all metallic ions on the resin. Excess ammonia was evaporated from the eluate (D) and combined ammonia removed with sodium hydroxide as shown with F. The solution was made to two liters and aliquots analyzed for basic and amino nitrogen. A liter aliquot was concentrated to about 200 ml and passed through a column containing 100 ml of Dowex 1-X8, 50-100 mesh, base form. The load was followed by a water wash (d) of 500 ml. The combined load and wash effluent (E) was concentrated to 250 ml and aliquots were taken for "betaine nitrogen" determinations. We assume that nearly all of this fraction is composed of betaine but slight amounts of other basic nitrogen compounds such as choline may be present.

Results and Discussion

Table 1 contains the results from straight house molasses samples. Table 2 shows the results from Steffen molasses samples and also for one sample obtained by the barium process and one by ion exchange.

Molasses purities varied from 59.01 to 67.45 for straight house molasses. The actual amount of sugar lost to molasses is greater for the higher purities than casual inspection would indicate. The ratio of sugar to non-sugar is 2.072 at 67.45 purity but only 1.440 at 59.01 purity. Thus the loss of sugar per unit of non-sugars is almost 44% greater in molasses of 67.45 purity than in molasses of 59.01 purity. The range of purity is less for Steffen molasses with a minimum of 61.23 and a maximum of 65.74. Average purity for both types of molasses is nearly the same.

Table 1.—Molasses composition (straight house)

	Belle Fourche	Betteravia spring	Betteravia fall	Brighton	Carlton	Carroll- ton	Clarks- burg	Crook- ston
Sucrose-g	61.57	60.97	62.50	62.32	67.45	64.51	62.93	61.66
Raffinose-g	1.27	.49	.26	1.88	.23	.82	.66	1.85
Reducing sugars-g	.5	1.3	.8	.4	2.5	1.2	.5	.8
Potassium-g	7.31	4.85	4.19	6.18	3.62	5.47	4.80	4.90
Sodium-g	.89	1.14	1.86	1.77	1.47	.63	1.55	.95
Calcium-g	.11	.31	.21	.09	.11	.55	.36	.17
Ash-g	15.7	11.8	12.0	15.4	9.5	11.8	13.1	11.4
Ash alkalinity-meq	137.8	111.3	130.3	118.8	91.7	134.3	129.2	133.5
K+Na-meq	225.6	174.0	188.3	235.5	156.8	167.5	190.3	166.7
Ca-meq	5.5	15.4	10.5	4.5	5.5	27.4	18.2	8.6
Alkalinity-meq/g ash	8.78	9.43	10.86	7.71	9.65	11.38	9.86	11.71
Chloride-g	1.06	1.18	1.42	2.29	1.46	1.42	1.60	.25
Nitrate-g	1.09	.44	.82	.98	.51	.58	.62	1.18
Anions-meq	192.0	175.0	177.8	203.8	146.7	172.5	185.8	157.2
Anions-eq wt	85.3	88.5	98.6	73.7	91.4	82.7	88.4	103.4
Anions-g	16.4	15.5	17.5	15.0	13.4	14.3	16.4	16.3
Total nitrogen-g	2.18	2.68	2.57	1.95	2.24	2.27	2.48	2.66
Amino nitrogen-g	.36	.59	.47	.37	.47	.37	.47	.54
Amide + ammonia-g	.05	.13	.12	.05	.09	.06	.10	.08
Basic nitrogen-g	1.32	1.74	1.53	1.32	1.55	1.51	1.56	1.61
Acidic nitrogen-g	.78	.83	.84	.61	.68	.67	.87	.94
Betaine fraction N-g	.75	.78	.79	.71	.71	.82	.80	.84
Neutral N-g	.02	.04	.04	.04	.09	.08	.05	.03
Betaine-g	6.10	5.79	5.46	5.53	4.72	6.09	5.97	5.97
Basic solids-g	11.27	13.88	12.66	11.25	12.04	12.21	12.63	12.28
Petaine fraction-g	7.22	7.59	6.22	6.19	5.16	7.39	6.53	6.15
pH undiluted	8.90	6.98	6.88	9.21	6.81	6.75	7.38	7.13
pH diluted 1/10	8.72	6.55	6.68	9.24	6.70	6.49	7.10	7.12

(g/100 g solids unless shown)

Eaton	Fremont	Green Bay	Manteca	Moorhead	Rupert	Sidney	Swink	Toppish	West Jordan	Avg
62.75	63.32	61.21	66.15	62.37	63.29	63.65	67.20	59.01	63.74	63.14
1.27	.47	1.20	1.00	.93	2.03	1.02	2.74	1.27	1.20	1.14
.4	.6	.5	.4	.8	.4	.4	.4	1.0	.4	.7
5.94	5.72	5.83	4.26	5.31	5.68	5.68	4.91	4.57	7.20	5.36
1.34	1.02	.52	2.24	.73	.61	1.44	1.63	1.18	1.06	1.22
.11	.23	.20	.13	.25	.14	.09	.14	.37	.13	.21
14.1	13.0	12.5	13.3	12.0	12.3	14.5	12.8	11.4	15.0	12.9
136.7	146.2	126.0	136.3	139.6	117.1	136.7	106.3	154.0	125.5	128.4
210.4	190.7	172.1	206.2	167.5	172.2	208.1	196.7	168.5	230.7	190.4
5.5	11.5	9.8	6.5	12.7	7.1	4.6	6.9	18.5	6.6	10.3
9.70	11.25	10.08	10.25	11.63	9.52	9.43	8.30	13.51	8.37	10.08
1.27	.91	.85	2.02	.40	.92	1.02	2.17	.37	1.96	1.25
1.29	.49	.70	1.17	1.02	.70	.91	1.06	1.02	1.33	.88
185.2	179.5	178.0	181.6	159.1	158.8	182.3	181.8	162.3	224.3	178.0
81.1	91.6	96.5	80.0	98.5	91.0	81.9	74.7	101.9	78.5	88.2
15.0	16.4	17.2	14.5	15.7	14.5	14.9	13.6	16.5	17.6	15.6
2.30	2.08	2.56	2.21	2.60	2.25	1.95	1.36	2.84	2.28	2.30
.43	.36	.43	.38	.42	.39	.36	.26	.46	.40	.42
.06	.07	.08	.07	.07	.07	.03	.09	.11	.05	.08
1.46	1.31	1.59	1.24	1.67	1.48	1.26	.93	1.63	1.50	1.46
.77	.68	.96	.78	.91	.66	.49	.37	1.10	.52	.75
.74	.74	.91	.62	.70	.69	.73	.45	.87	.79	.75
.02	.05	.03	.07	.12	.10	.11	.16	.17	.21	.08
5.87	5.43	6.73	4.74	7.12	6.22	5.75	3.35	5.88	5.65	5.69
12.85	10.49	13.13	10.45	14.41	12.95	10.52	7.64	13.61	12.00	12.02
6.11	5.88	7.06	5.09	5.73	5.53	5.91	3.59	6.72	6.13	6.12
9.06	8.09	6.79	9.11	7.48	8.15	9.13	8.43	8.12	8.93	
8.98	7.93	6.58	8.23	7.20	8.08	9.02	8.47	8.09	8.82	

Table 2.—Molasses composition

	Steffen house				
	Alvarado	Grand Island	Love-land	Mason City	Nyssa
Sucrose-g	61.55	63.28	62.10	63.66	65.10
Raffinose-g	2.02	1.79	2.46	1.80	2.44
Reducing sugars-g	.3	.5	.4	.5	.5
Potassium-g	3.78	5.71	5.21	4.23	4.51
Sodium-g	1.60	.53	1.33	.77	.91
Calcium-g	.41	.31	.22	.52	.15
Ash-g	11.2	11.5	13.1	10.5	10.5
Ash-alkalinity-meq/100 g	138.1	128.9	140.7	125.1	113.4
K+Na-meq/100 g	166.2	169.1	191.2	141.6	156.2
Ca-meq/100 g	20.6	15.2	11.0	26.1	7.4
Alkalinity-meq/g ash	12.33	11.21	10.74	11.91	10.80
Chloride-g	1.18	.76	.79	.46	.61
Nitrate-g	.55	1.09	1.09	1.00	.93
Anions-meq/100 g	167.6	158.6	180.0	156.9	139.0
Anions-eq wt	94.8	99.6	94.0	104.9	100.4
Anions-g	15.9	15.8	16.9	16.5	14.0
Total nitrogen-g	1.91	2.15	1.89	2.35	2.20
Amino nitrogen-g	.39	.44	.36	.46	.44
Amide :- ammonia N-g	.06	.07	.05	.11	.10
Basic nitrogen-g	1.28	1.17	1.22	1.36	1.28
Acidic nitrogen-g	.65	.68	.62	.79	.71
Betaine fraction N-g	.60	.68	.66	.63	.75
Neutral N-g	.06	.05	.04	.09	.12
Betaine-g	4.38	4.98	4.90	4.97	4.99
Basic solids-g	12.57	8.96	9.72	11.59	11.29
Betaine fraction-g	5.51	5.03	5.32	5.11	5.86
pH undiluted	8.25	8.21	8.35	7.11	6.91
pH diluted 1/10	8.12	8.12	8.39	7.27	6.73

(g/100 g solids unless shown)

Steffen house									Barium process	Ion exchange
Oxnard spring	Oxnard fall	Scotts- bluff	Spreckels	Torring- ton	Tracy	Twin Falls	Wood- land	Average	Johns- town	Layton
65.29	63.23	61.23	63.02	61.75	63.83	64.12	65.74	63.38	71.96	60.10
1.05	1.11	2.76	2.27	4.15	2.26	3.16	1.62	2.22	16.00	2.62
1.3	.6	.4	2.4	.4	.4	.7	1.0	.7	.4	6.2
2.92	3.22	6.32	3.29	5.41	3.80	4.65	4.18	4.40	.75	1.21
2.05	2.27	.67	1.74	1.13	2.02	.86	1.29	1.32	1.04	2.64
.23	.22	.18	.48	.11	.19	.18	.32	.27	.05	.30
10.6	11.7	13.7	11.2	12.8	11.8	10.7	10.2	11.5	3.8	8.5
110.9	122.3	130.2	118.2	117.1	115.8	100.9	121.1	121.7	56.4	127.0
164.0	181.4	191.3	160.0	187.7	185.3	156.3	163.2	170.3	64.6	145.8
11.7	11.0	9.2	24.0	5.3	9.3	9.1	16.0	13.5	2.7	15.0
10.46	10.45	9.50	10.55	9.14	9.81	9.43	11.87	10.63	14.84	14.94
1.08	1.09	1.18	1.66	1.26	2.08	.85	1.33	1.10	.19	.61
.43	.83	.73	1.14	.79	1.02	.65	.87	.86	.07	.20
155.2	179.4	179.1	166.9	172.0	172.7	155.7	192.0	167.3	58.8	161.7
96.1	99.4	87.5	85.3	82.2	82.4	93.4	87.0	92.8	101.1	102.0
14.9	14.8	15.7	14.2	14.1	14.2	14.5	16.7	15.2	5.9	16.5
1.65	1.72	1.78	1.86	1.58	1.98	1.97	1.98	1.93	.1	1.74
.28	.34	.36	.32	.30	.33	.39	.37	.37	.01	.15
.07	.07	.05	.12	.02	.04	.06	.07	.07	.01	.05
1.01	1.06	1.23	.91	.97	1.24	1.36	1.44	1.19	.15	1.16
.51	.59	.48	.58	.33	.61	.62	.67	.60	.04	.65
.46	.52	.70	.44	.65	.65	.71	.73	.63	.08	.78
.10	.12	.08	.12	.15	.20	.09	.20	.11	.03	.07
3.68	3.56	5.40	3.75	4.96	4.79	5.12	4.29	4.60	.96	5.57
8.96	9.26	10.99	7.52	9.82	10.08	11.50	11.96	10.32	1.62	9.68
4.13	3.85	5.73	3.60	5.47	4.76	5.49	5.56	5.03	.51	6.54
7.02	7.09	8.48	6.38	8.78	7.72	6.92	7.26		9.67	6.10
6.81	7.07	8.48	6.15	8.70	7.72	6.72	7.19		10.37	6.03

The raffinose content of straight house molasses averages about half that of Steffen molasses produced in the same area. There is a great overlap in the ranges, however, and eleven of the Steffen molasses had less raffinose than the maximum found in straight house samples. Many other constituents have a slightly higher average value in straight house molasses but no constituent measured in these experiments would enable one to distinguish between the two molasses types. A comparison of the analytical data for Manteca and Tracy molasses shows the close similarity of the two molasses types produced largely from comparable beets. The main difference is in the raffinose content which is concentrated by the Steffen process. Johnstown molasses (barium process) has a very high purity and raffinose content compared to other molasses. Consequently it has low values for other constituents calculated on the dry basis. Raffinose is accumulated in ion exchange molasses to about the same extent as in Steffen molasses. Purity of ion exchange molasses would appear to be lower than for most straight house or Steffen samples and the quantity per ton of beets is much less.

Reducing sugars average the same for both types of molasses but above pH 8 the amount of reducing sugars seems to be closely related to the pH of the molasses. Higher pH molasses have very little reducing sugar. This is probably due to the slow rate of formation of reducing sugars from sucrose at the higher pH values and to the more rapid destruction of glucose and fructose at these values. While sucrose is most stable near pH 9, glucose and fructose are most stable at pH 3-5. In the samples studied the pH varied from slightly acidic (6.38) to moderately basic (9.21). Generally the pH of a diluted sample is lower than that of the original but in some cases a higher value is obtained on dilution.

Potassium ions occur to the largest extent of any metallic ion present in molasses. In some cases part of the sodium present is from sodium carbonate added to maintain higher alkalinity. The calcium content is closely related to pH. Molasses with lower pH values contain more calcium. This would be expected since most calcium salts become more soluble as the pH decreases. Steffen molasses calcium content is about 50% higher than for straight house molasses. Part of this increase is due to recycling certain anions as calcium salts insoluble at the pH of the Steffen treatment but more soluble at the pH of first carbonation. Hence, these calcium salts will increase and the calcium will not be precipitated except by the addition of sodium hydroxide or carbonate.

Ash averages slightly higher for straight house molasses. Ash

alkalinity is a measure of organic acids that are converted to carbonates during ashing. Little difference is shown between the two molasses although there appears to be some preferential accumulation of organic anions over inorganic anions in Steffen molasses. This is demonstrated by the slightly higher ratio of milliequivalents of alkalinity to grams of ash.

Chlorides show the greatest quantitative variation for a major molasses constituent: eight-fold for straight house (0.25%-2.20%) and four-fold for Steffen molasses (0.4%-2.08%). Nitrate content varies about two-fold with little apparent difference between the two molasses types.

Milliequivalents of anions, equivalent weight of anions or weight of anions present show little variation between the two molasses types. Anions as a group average over 40% of the non-sugars.

Basic nitrogen compounds form the major portion of the total nitrogen. Betaine is usually half of this fraction with amino acids being most of the remainder. Pyrrolidone carboxylic acid is the principal acidic nitrogen constituent in most molasses with nitrate second. There is little apparent difference between the two types of molasses.

Johnstown molasses (barium process) with a very high purity has an extremely high raffinose content compared to other molasses and consequently shows low values for other constituents on the dry basis.

Ion exchange molasses has a higher sodium than potassium content. This is probably due to preferential adsorption of potassium on the ion exchanger. This difference appears to be a distinguishing characteristic from other molasses while nitrogen and anion composition are very similar.

Summary

Straight house and Steffen molasses cannot readily be distinguished on the basis of chemical analyses except that the average raffinose content of Steffen molasses is usually twice that for straight house molasses produced in the same area. However, unless this value is below 1% for straight or above 2.5% for Steffen it is difficult to distinguish between molasses samples.

Chloride shows an eight-fold variation, the greatest sample-to-sample variation of any major constituent analyzed. Most constituents have a variation of less than two-fold with betaine showing the least variation for a major non-sugar.

The statistical relationship of sucrose and non-sugar is presented in Part II.

Acknowledgment

The authors are indebted to the following companies for molasses samples: The Amalgamated Sugar Co., Ogden, Utah; American Crystal Sugar Co., Denver, Colorado; The Great Western Sugar Co., Denver, Colorado; Holly Sugar Co. Corp., Colorado Springs, Colorado; Layton Sugar Co., Layton, Utah; Menominee Sugar Co., Green Bay, Wisconsin; Michigan Sugar Co., Saginaw, Michigan; Northern Ohio Sugar Co., Denver, Colorado; Spreckels Sugar Co., San Francisco, California; Union Sugar Division, Consolidated Foods Corp., San Francisco, California; and Utah-Idaho Sugar Co., Salt Lake City, Utah.

The authors wish to thank the following people for many of the analytical determinations: Mr. E. F. Potter, Mrs. M. Long, Mr. H. Wright, and Mr. T. Jaouni.

Literature Cited

- (1) ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS. 1955. Official and tentative methods of analysis of the Association of Official Agricultural Chemists. Ed. 8. Washington, D.C.
 - (2) BEVENUE, A., and K. T. WILLIAMS. 1958. Quantitative determination of raffinose or melibiose by paper chromatography. *Arch. Biochem. Biophys.* 73: 291-295.
 - (3) FOCHT, R. L., F. H. SCHMITT and B. B. DOWLING. 1956. Colorimetric determination of betaine in glutamate process end liquor. *J. Agr. Food Chem.* 4: 546-548.
 - (4) GARDINER, S. D., and F. J. FARMILOE. 1954. Design and operating technique of a vacuum drying oven. Part II. Solids in cane molasses. *Analyst.* 79: 447-453.
 - (5) GARDINER, S. D., and F. J. FARMILOE. 1955. Design and operating technique of a vacuum drying oven. Part III. Solids in (a) mixtures of cane and beet molasses and (b) beet molasses. *Analyst.* 80: 557-561.
 - (6) JOHNSON, C. M., and A. ULRICH. 1950. Determination of nitrate in plant material. *Anal. Chem.* 22: 1526-1529.
 - (7) PETERS, J. P., and D. D. VAN SLYKE. 1932. *Quantitative Clinical Chemistry.* Williams and Wilkins, Baltimore. 1: 162-63, 385-90.
 - (8) STARK, J. B. 1960. Determination of average equivalent weight and total weight of plant acids by ion exchange resins applied to sugar beet molasses. *J. Agr. Food Chem.* 8: 234-236.
 - (9) WINTON, A. L., and K. B. WINTON. 1945. *The analysis of foods.* Wiley, New York. 550.
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