Partial Composition of Sugar Beet Molasses

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The need for more precise knowledge of the constituents of sugar beet molasses has been recognized for many years. The less we know about the composition of molasses and other processing liquors, the more we have to work blindly in developing logical methods for reducing molasses production or in finding further uses for the residues. Only after an accurate picture of molasses composition has been drawn will it be possible to adopt a rational evaluation of the melassigenic importance of various non-sugars. Molasses composition data may be applicable to crystallization studies, to genetic improvement of the beets, and in development of improved processing procedures. Feed formulators, fermenters, and others using molasses will find composition data valuable and new useful byproducts may be discovered.

Nine composite molasses samples have been analyzed for several of their constituents. Six of these samples were from straight house factories. The remaining samples were from factories using the Steffen process. The samples were obtained from Alvarado, Betteravia, Clarksburg, Manteca, and Woodland, California; Brighton, Colorado; Carrollton, Michigan; Moorhead, Minnesota, and Toppenish, Washington. The molasses samples were analyzed for potassium, total nitrogen, ammonia, amide and amino nitrogen, betaine, pyrrolidone carboxylic acid (PCA), lactic, glycolic, malic and citric acids, sulfur, phosphorus, chloride and total milliequivalents of anions. The results are presented on the basis of grams per 100 grams of non-sucrose solids.

Experimental

The molasses samples were diluted to 10 to 20 percent solids and the various determinations were made on aliquots of the diluted samples. Potassium was determined chemically utilizing the formation of insoluble potasium-sodium cobaltinitrite (1)[2]. Total and ammonia nitrogen were determined by conventional AOAC methods (2). It was later found on analyzing solutions containing known amounts of sucrose and potassium nitrate that the total nitrogen method used on these samples recovered

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[2] Numbers in parentheses refer to literature cited.

only 60 to 80 percent of the nitrate present. Based on this finding the total nitrogen determinations on the original molasses may be low by 4%. Acidic nitrogen determinations may be low by 8%. Amide nitrogen was determined by the method of Winton and Winton (3).

The method of Peters and Van Slyke was used to determine amino nitrogen (4). Betaine was determined by the method of Walker and Erlandsen (5). Pyrrolidone carboxylic acid (PCA) was calculated from a corrected nitrogen determination of the PCA fraction separated by ion exchange fractionation from other acidic nitrogen compounds (6). Citrate was determined by the pentabrom-acetone method (7). Lactic and malic acids were determined colorimetrically (8, 9). Glycolic acid was measured by an adaptation of the malic and lactic acid methods (10). Total halogen was determined by AOAC methods (2). Potter's method was used for estimation of total sulfur (11). Allen's method was used for estimation of total phosphorus (12).

The values for sulfur, chlorine, and phosphorus are presented as if they existed in molasses as sulfate, chloride, and phosphate. This assumption will introduce an error in the case of sulfur, since some of the sulfur will occur as sulfite or as a sulfur compound that will release sulfur dioxide on acidification and prolonged heating (13). Milliequivalents of anions were determined on an aliquot of dilute molasses by titration to pH 8.3 with sodium hydroxide solution after the cations were exchanged with hydrogen, using the cation exchange resin Dowex-50 (H).[3] Some acidic compounds such as glutamic and aspartic acids are also removed by the ion exchange resin. A nearly complete analysis of the amino acids from one sample of molasses (14) shows that the distribution of various amino acids in sugar beet molasses is quite different from the average distribution in protein. On the basis of the amino acid composition of sugar beet molasses a factor of 8.4 was calculated to convert amino nitrogen into amino acid content. Total amide is calculated as asparagine, since this amide is most likely to survive alkaline processing treatment, whereas the more common amide in sugar beets, glutamine, readily decomposes to form pyrrolidone carboxylic acid (PCA). Much of this ammonia is lost during the evaporation step in the manufacture of sugar.

Results

Results of the various analyses and calculations are presented in Tables 1 through 4. Table 1 presents a picture of the basic

[3] Mention of a specific product does not constitute endorsement by the Department of Agriculture over others of a similar nature not mentioned.

impurities or non-sucrose solids present in molasses. Table 2 shows the amounts of organic acids in molasses, while Table 3 gives the same data for inorganic acids and also the total milliequivalents of acids. Table 4 shows the average milliequivalents for each acid present in molasses and also the amount due to known organic and inorganic acids.

On the basis of data in Tables 1, 2, 3, and 4, the only apparent differences between the Steffen and straight house molasses are in the lower lactate content and, possibly, a lower potassium content of the Steffen molasses. The large variations in chloride content are of considerable interest, since Wise and Nicholson

Table 1.—Beet Molasses—Basic Constituents (g./100 g. non-sucrose solids).

Factory[1]	Potassium	Betaine	Amino acids[3]	Amide[4]
Al[2]	14.1	13.0	10	1.9
Be	14.6	16.9	11	2.8
Br	18.7	14.8	9	1.5
CaM	21.1	19.5	9	1.7
Cl	12.6	19.0	9	2.0
Ma	20.1	19.2	12	2.5
Mo	15.2	19.0	8	2.0
To[2]	13.5	20.4	11	2.5
Wo[2]	11.5	13.8	7	2.0
Average	15.7	17.3	10	2.1

[1] Abbreviations: Al = Alvarado, Be = Betteravia, Br = Brighton, CaM = Carrollton, Cl = Clarksburg, Ma = Manteca, Mo = Moorhead, To = Toppenish, and Wo = Woodland.

[2] Steffen molasses.

[3] Amino nitrogen times 8.4.

[4] Amide nitrogen calculated as asparagine.

Table 2.—Beet Molasses—Acidic Organic Constituents (g./100 g. non-sucrose solids).

Factory	Lactic	Glycolic	PCA	Citric	Malic
Al	3.3	2.1	6.1	1.5	1.8
Be	5.3	1.3	10.8	.0	1.2
Br	5.3	.8	8.3	2.5	2.6
CaM	5.4	1.7	6.7	.5	1.7
Cl	5.2	1.2	9.4	.5	1.4
Ma	6.6	1.7	13.4	.5	2.1
Mo	5.9	.8	9.0	1.5	2.1
To	2.3	1.3	14.5	1.3	1.9
Wo	3.1	1.9	6.4	1.0	2.2
Average	4.7	1.4	9.4	1.0	1.9

Table 3.—Beet Molasses—Acidic—Constituents.

Factory	Inorganic Anions (g./100 g. Non-Sucrose Solids) Chloride	Sulfate	Phosphate	Total Anions[1] (meg./100 g. Non-Sucrose Solids)
Al	5.4	2.5	0.2	610
Be	6.7	4.0	0.2	560
Br	8.9	8.0	0.3	750
CaM	5.6	6.0	0.2	620
Cl	5.7	5.1	0.2	560
Ma	8.7	3 7	0.3	770
Mo	2.6	4.6	0.4	500
To	1.5	4.9	0.5	470
Wo	5.2	2.8	0.3	520
Average	5.6	4 6	0.3	600

[1] Includes organic and inorganic anions.

Table 4.—Average Milliequivalents of Anions (meg./100 g. non-sucrose solids)

Anions	Milliequivalents	
Lactate	52	
Glycolate	18	
PCA	73	
Citrate	16	
Malate	28	
Total Organic		187
Chloride	158	
Sulfate	96	
Phosphate	10	
Total Inorganic		264
Undetermined Acids		149
Total		600

(15), Moritsugu (16), and Rorabaugh and Norman (17) have pointed out that chloride as the potassium or sodium salt in a sucrose-water--chloride system decreases the rate of sucrose crystallization and increases sucrose solubility. These results have been confirmed in our laboratory by the work of Teranishi, Knowles, and McCready (18). They have shown by experiments with sucrose solutions containing a single impurity, that potassium sulfate had no measurable effect on crystallization rate but lowered the final solubility of sucrose in the mixture. On the other hand either potassium or sodium chloride lowered the rate of crystallization and increased sucrose solubility rate. The principal rate and solubility effects are apparently due to chloride.

The basic constituents form 45 percent of the total determined impurities. The principal unknown substances in this category are unidentified nitrogen compounds. Twenty-nine percent of the determined constituents are acids consisting of 18 percent organic acids and 11 percent inorganic acids. With the exception of nitrate most of the unknown or undetermined acids are probably organic. There are a few percent of raffinose, galactinol, and other essentially neutral compounds present. The known impurities that we have measured quantitatively average 74 percent of the non-sucrose solids and range from 58 to 89 percent for the samples investigated. The amounts of several known constituents of beet molasses such as sodium, raffinose, and nitrate were not measured in this study but a more complete analysis of representative molasses from the 1956 campaign is now under way and the average values for sodium, nitrate, and raffinose in straight house molasses are 3.6, 2.4, and 3.1 percent, respectively. If we are justified in assuming that we would have obtained similar values for the molasses samples reported here, 17 percent of the impurities are still undetermined.

Summary

The average of the total non-sucrose solids determined in the molasses samples examined constitutes 74 percent of the total impurities. Sodium, raffinose, acetate, galactinol, nitrate, and unknown nitrogen compounds are included in the remaining 26 percent. Up to 20 percent of the molasses impurities remain unidentified.

The impurity showing the greatest quantitative variation is chloride, which is of particular importance, since it is also one of the substances known to have an especially deleterious effect on sucrose crystallation.

Steffen molasses probably has a lower potassium and lactate content than straight house molasses although an insufficient number of samples have been examined to verify this.

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