Beet Molasses True Purity Predicted From Thin Juice Chloride Content

J. B. STARK[1]

Received for publication August 10, 1967

Statistical evaluation of straight house molasses composition variables of samples collected from U.S. beet sugar factories in 1956 showed a significant positive correlation at the 1% level for chloride content and purity (3)[2] Since chloride content is correlated with molasses purity and is not removed during processing, the chloride content of thin or thick juice should provide a reliable means to predict molasses purity. To gain further information on molasses true purity and composition relationships during a factory campaign, a cooperative study was undertaken with Spreckels Sugar Co.[3] This report discusses results obtained during this study on the relationship of thin juice chloride content and molasses purity. Further data obtained from an extension of this study to samples of thick juice and molasses from Manteca (Spreckels Sugar Co.) in 1962 and thin juice and molasses obtained from Clarksburg (American Crystal Sugar Co.) in 1962 are also included.

Methods

At Western Regional Research Laboratory the following methods were used. Molasses samples in covered 8-ounce glass jars were heated to about 80°C in a hot water bath until all the sugar dissolved. The jars were opened and the contents stirred thoroughly with a heavy glass stirring rod. Samples of 15-20 g were poured into tared beakers and weighed precisely.. The samples were dissolved in hot water and transferred quantitatively to 500 ml volumetric flasks. When cool the contents were made to volume. Aliquots were removed for solids, chloride, reducing, and total sugar determinations.

Chloride was determined using an Aminco-Cotlove automatic chloride titrator[4]. Solids were determined by weighing duplicate aliquots containing 300-600 mg of solids in tared glass weighing

[1] Western Regional Research Laboratory, Agricultural Research Service. U. S. Department of Agriculture, Albany, California—94710.

[2] Numbers in parentheses refer to literature cited.

[3] A memorandum of understanding was negotiated August 30, 1960, between Spreckels Sugar Co., San Francisco, California, and the Western Regional Research Laboratory, for joint evaluation of the effect of chloride on molasses purity.

[4] American Instrument Co., Silver Springs, Maryland.

dishes (50 mm diameter) and evaporating in a forced draft oven at 60°C to a heavy syrup (about 6 hours). The dishes containing the syrups were placed in a vacuum oven at 10 mm Hg or less and the foam formed *in vacuo* dried at 60°C. After 30 hours the foams were dry and the pressure was restored with air, bubbled slowly through sulfuric acid. The dishes were placed in a desiccator, cooled and weighed.

Reducing sugars were determined by pipetting a suitable aliquot (100 ml) into a 200-ml volumetric flask, clarifying with lead acetate and deleading with dry sodium oxalate. Total sugars were determined on aliquots of the clarified, deleaded solution by treating with invertase and using the methyl red end point for the action. Seven drops of invertase (Difco Analytical) were added to the sample and reducing sugars determined on the inverted solution using the Munson-Walker method (1). Porcelain filter crucibles which do not require an asbestos pad were used for weighing the copper oxide precipitates. True purity was calculated from chemical sucrose and oven solids. Raffinose was determined on a few 1960 molasses samples using the paper chromatographic method of Bevenue and Williams (2). The average of these values, 0.13% on molasses, was used to correct the 1960 total sugar values to sucrose content. Factory raffinose values were used in 1962 to correct total sugars to sucrose. Factory RDS and purity determined at the factory for thin and thick juice were used to convert chloride to the non-sucrose solids basis.

Experimental

During 1960, shift samples of thin juice were collected for 14 days from September 21 through October 4 and analyzed at the Manteca factory for chloride, solids (RDS) and sucrose (polarimetric). Daily average values were calculated from the shift sample results. Daily composite molasses samples were also collected and analyzed for chloride at Manteca and for purity and solids at Western Regional Research Laboratory. Samples of thin juice or thick juice were collected during the 1962 campaign (Clarksburg from November 14-27, Manteca from November 18-December 1). Factory values for RDS and purity of thin or thick juice and raffinose in molasses were used in the calculations. Chlorides in the juices and molasses true purity determinations were made at Western Regional Research Laboratory.

Impurities entering the factory during thin juice production on one day do not appear in molasses that day because of the time needed for juice concentration and sugar crystallization and are not completely eliminated for several days. To predict mo-

lasses purities several days production should be averaged and compared with molasses produced for an equivalent time starting one or two days later than the production of thin or thick juice.

Results

Table 1 compares the day to day variations in chloride content of thin and thick juices and molasses. Purity variability for molasses is also shown. The 5-7% higher chloride content of molasses on the impurities basis over thin juice was expected because some non-sugars are removed during processing from thin to thick juice. Fewer impurities are removed during processing from thick juice to molasses so only slight differences are found between these processing liquors.

Table 1.—Molasses purity and chloride content of thin or thick juice and molasses.

	Manteca - 1960			Manteca - 1962			Clarksburg - 1962		
	Cl g/100 g non-sucrose		Molasses	Cl g/100 g non-sucrose		Molasses	Cl g/100 g non-sucrose		Molasses
Days	Thin juice	Molasses	Purity	Thick juice	Molasses	Purity	Thin juice	Molasses	Purity
1	7.06[1]			5.07			3.04		
2	5.67			5.50			3.02		
3	5.64	6.92[2]	64.03	5.56	5.48	65.81	2.95	2.93	64.93
4	6.71	6.71	64.82	4.91	5.39	64.98	2.85	3.30	64.10
5	4.28	6.08	65.11	5.81	5.82	65.14	2.73	3.34	63.10
6	6.01	6.67	64.48	5.85	5.42	64.44	3.25	3.18	63.07
7	6.44	6.08	64.63	5.74	5.37	64.16	3.39	3.24	61.56
8	5.73	6.26	66.32	6.15	5.81	64.82	3.30	3.66	63.95
9	6.17	6.27	64.91	5.80	6.13	64.79	3.74	3.51	61.62
10	5.24	5.78	64.36	5.85	5.90	65.08	3.89	3.89	62.65
11	5.70	6.02	64.76	5.50	6.04	64.38	3.69	3.99	62.98
12	5.29	5.85	64.79	5.40	5.66	63.82	3.92	3.73	61.60
13	6.24	5.58	64.52	5.86	5.52	63.63	3.59	4.03	62.60
14	5.95	5.68	65.32	6.29	5.53	63.89	3.55	3.61	61.34
15		6.03	64.32		5.65	65.13		4.13	63.76
16		6.44	66.16		5.95	64.37		4.04	64.61
Avg	5.87	6.17	64.90	5.66	5.69	64.60	3.35	3.61	62.99

[1] Chloride determinations were made on samples collected during 14 days continuous operations.

[2] Initial molasses samples were collected two days after thin or thick juice collection and continued two days longer.

Table 2 compares molasses purities predicted from chloride content of thin juice, thick juice or molasses with the observed purity. The equation used (3) is: Purity = 0.75 (% Chloride) + 60.34. Predicted purities based on thin juice chloride content show very good agreement with observed values. Because of slightly lower chloride content in thin juice than in molasses the predicted purity values are slightly less than the observed values. Molasses purities predicted from molasses chlorides also show very good agreement with observed values.

Discussion

Molasses purity predictions made from chloride determinations on thin or thick juice should have a number of uses. It is useful to predict changes in molasses purity as changes in chloride

Table 2.—Comparison of predicted and observed molasses true purities.

	Manteca		Clarksburg
	1960	1962	1962
Thin juice chloride	5.87	5.66[1]	3.35
Predicted molasses purity[2]	64.74	64.59	62.85
Observed molasses purity	64.90	64.60	62.90
Difference	—0.16	—0.01	—0.14
Molasses chloride	6.17	5.69	3.61
Predicted molasses purity[2]	64.96	64.61	63.05
Difference	0.06	0.01	0.06

[1] Thick juice chloride.

[2] Predicted purities using the equation Purity = 0.75 (% Cl) + 60.34 where Cl is expressed as g/100 g non-sucrose solids for 14 days consecutive operation.

occur in the beets. For example, a change in molasses purity from the crystallizer may be directly attributed to changes in the chloride content of the beets rather than other changes in the operation. Chloride determinations should also be useful in estimating the efficiency of some factory operations. At present, a factory producing high purity molasses is generally considered to be operating less efficiently than one producing low purity molasses. However, a factory producing 64 purity molasses with a high chloride content may actually be conducting a better operation than one producing 62 purity molasses with a low chloride content.

The geneticist and agronomist might also find purity predictions useful in evaluating sugar production from new varieties of beets or different soil types. A better method than purity determinations is offered for evaluating certain processing characteristics of beets to give a more reliable estimate of sugar recovery and molasses production. Attempts should be made to grow beets that do not take up excessive amount of chloride from highly saline soils.

Conclusions

Chloride determinations of thin or thick juice are useful in predicting changes in molasses purity as changes in chloride occur. Purity predictions may be helpful in evaluating factory efficiency. These predictions also offer a simple method to geneticists and agronomists to evaluate some aspects of the processing value (purity and amount of molasses) from beets grown under different conditions of soil and water salinity.

Acknowledgments

The author is indebted to Dr. R. A. McGinnis, General Chemist, and others from Spreckels Sugar Co. for their cooperation in providing samples and factory analytical data and supervising chloride determination. Mr. Peter Hanzas, Research Chemist, and Mr. Don Jackson, Chemist, American Crystal Sugar Co., provided samples and factory analytical data. Mr. Earl Potter, Chemist, Western Regional Research Laboratory, determined chemical sugars, and Mr. Henry Wright and Mrs. Marion Long, Western Regional Research Laboratory, determined total solids.

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.

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