

orange. Subtract the ml. of barium chloride used in a blank test from the ml. used in the titration and calculate the result.

5. Precautions.—It is of the greatest importance to follow the order and manner of introducing the reagents in order to obtain consistent and reliable results. Adding a portion of the barium chloride before the silver nitrate is introduced produces a sharper and brighter end point. The sodium-chloride solution makes the end point perceptible, in that there is a quicker and definite change of color from milky-brown to bright orange.

Note: The most distinct end point is obtained if the volume of barium chloride used is not more than 10 ml. If titration requires more than this volume, with the No. I barium-chloride solution, use a more dilute sample.

#### Literature Cited

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4. Kahler, H. L., Betz, W. H., and L. D. *Determination of Sulfate by Tetrahydroxyquinone Method*. Philadelphia, Pa.
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## Some Regional Effects on Beet-Sugar Quality<sup>1</sup>

CHARLES A. FORT<sup>2</sup>

Our 1940 report on beet sugars included tables of average data for different regional groups of factories. The grouping used was based on juice purities combined with general geographical location. Ten groups were made so that the individual factory could compare its product with the average of a relatively small number of factories of corresponding location and similar juice purities. They were not intended for comparing the group averages themselves. Therefore, for the study reported in this paper, with the specific object of comparing the sugars of beets from different producing areas, it was found more satisfactory and simpler to group the data representing

<sup>1</sup>Agricultural Chemical Research Division, Contribution No. 55.

<sup>2</sup>Chemist, Agricultural Chemical Research Division, Bureau of Agricultural Chemistry and Engineering, U. S. Department of Agriculture, Washington, D. C.

This paper was presented by J. C. Keane, Assistant General Superintendent, Utah-Idaho Sugar Company.

the product of individual factories according to the four natural producing areas, namely, the West, the intermountain region, the central dry plains, and the East. Figure 1 shows the distribution of sugar-beet factories and the dividing lines between the areas.

It should be kept clearly in mind that in this discussion we are to deal principally with averages. This means that in each case some sugars were low and others high in the particular factor under consideration. As the averages represent rather large numbers of determinations and sugars, it is felt that the differences in characteristics indicated by the averages are true trends and significant. As both non-Steffen and Steffen sugars showed the same trends in spite of certain differences in non-sugar composition, equal weight was given to the two types in the calculations. Separate averages were prepared for non-Steffen and Steffen factories in each area, and the mean of these averages is what is submitted. This is equivalent to there being an equal number of the two types of factories in each area, which seems to be a fair basis for our considerations.

The degree of variation in ash content within the groups, as well as the average trend of ash determinations on sugars from different regions, is shown in table 1. Here are given the lowest and highest of the 5-year averages (1936-40) for individual factories and the mean of averages for entire groups within this period. There is some overlapping between the different groups as regards maximum

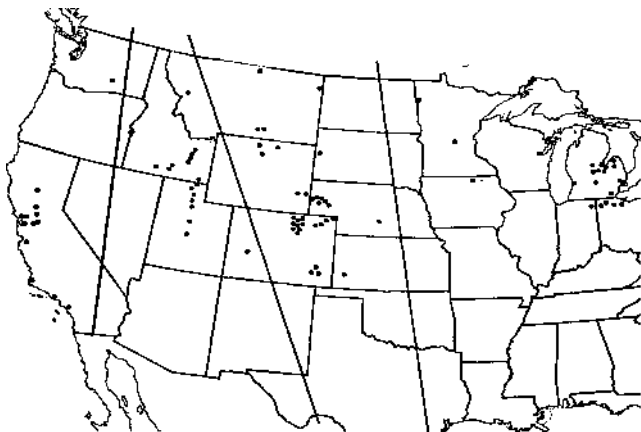


Figure 1.—The distribution and grouping of beet-sugar factories.

and minimum ash values, but the high-ash tendencies of the intermountain and dry plains areas stand out clearly. Unquestionably this is due to the nature of the impurities in the juices handled and not to differences in processing technique.

Table 1.—Extremes, averages and distribution of the 5-year means of the ash of the sugars of different regions.

	West	Intermountain	Central plains	East
	p.p.m	p.p.m.	p.p.m.	p.p.m.
Minimum average	54	00	72	73
Maximum average	114	170	225	165
Grand average	78	120	124	112
Percentages of sugars with ash				
	percentage	percent;] go	percentage	percentage
Below 75 p.p.m.	58	0	5	9
75-100 p.p.m.		7	18	35
100-125 p.p.m.	17	40	41	39
125-150 p.p.m.	0	40	14	9
Above 150 p.p.m.	0	7		8

In table 2 we repeat the averages just shown for 1936-40 and also give the averages for the previous 5 years, 1931-35, and the general average. The averages for the first 5 years (1931-35) probably give the more nearly correct picture of the natural differences between the beets of the different areas; these differences have persisted year after year, especially in the case of the Steffen factories. However, in recent years improved operations seem to have changed the results of comparisons between ash content of sugars representing different groups of the non-Steffen sugar factories. Beet sugars from the central dry plains give an average ash below those of the sugars from, the intermountain region and the East, although still much higher than that of sugars from the West. In spite of this discrepancy in the case of non-Steffen sugars from the central plains in recent years, the complete data indicate strongly that the contrasts shown by the general averages represent the true tendencies of the ash contents of the beets. Further evidence that this conclusion is valid is given by the analyses of a few thick-juice and molasses samples from non-Steffen factories of the four areas. These particular tests were preliminary and few in number but are included in the table. On the thick juices the ash is expressed as percentages on solids and on the molasses as percentages on beets, having been calculated from percentages on molasses, making use of the amounts of molasses produced. The probability that the higher ash of the sugars of the mountain and central areas is due to the natural composition of the beets is evident.

It is to be kept in mind always that superior sugar boiling and sugar washing and the use of special chemicals may produce low-ash sugars even from high-ash sirups. The washing technique is an especially vital factor for, as has been demonstrated by other workers,

Table 2.—A. Averages of ash in sugars.

	West	Intermountain	Central plains	Bast
	p.p.m	p.p.m.	p.p.m.	p.p.m.
1931-35 inclusive	106	220	237	144
1936-40 inclusive	78	129	124	112
Grand average 10 years	92	174	180	128
Molasses ash, percentage beets	0.31	0.46	0.56	0.43
Thick-juice ash, percentage solids	2.18	2.30	2.87	

## B. Averages of white massecuite purities.

1931-35 inclusive	89.6	80.2	88.0	88.0
1936-40 inclusive	92.6	91.8	91.3	91.0
Grand average	90.8	90.5	89.6	89.5

the ash concentration is very high in the outer shell of the sugar crystals as compared with the inner portions. Thus the ash of sugar is much affected by the quantity of surface material removed in washing. It is because of these variables that it is so difficult to judge the composition of the beets on the basis of the composition of the sugars. It is felt that the extensive study that we have made of the accumulated data that the averages presented do indicate differences in sugar-ash composition which are primarily due to differences in the beets, but that the results of individual factories cannot be so interpreted for the reasons just discussed. It has been clearly evident in experiments which we have made that with uniform conditions of sugar boiling and washing the concentration of ash and its elements in the sirup determines the ash composition of the sugar produced, but wide variations are possible when the boiling and washing operations are variable.

Going now to the components of the ash we will first examine the averages for the sulfate radical, table 3. Here, even more so than in the case of total ash, we feel that the average for the early 5-year period better reflects the natural composition of the beets, but the trend is still evident even to date. We refer to the relatively high-sulfate content of the sugars and presumably of the beets of the two central areas being highest in the central plains. This condition is seen both in the concentration of sulfates in the sugar and the proportion of sulfates on ash. Also, the data on thick juices and molasses agree with this conclusion.

While not strictly a phase of this subject, it may be of interest to note that the proportion of sulfate in ash is higher in the sugars than in sirups or molasses. This agrees with the known fact that sulfates are taken up by the sugar to a greater extent than certain other minerals thereby increasing the proportional sulfate content.

Table 3.—Sulfate (SOs) content of sugars.

	West		Internountain		Central plains		East	
	p.p.m.	percent- age on ash	p.p.m.	percent- age on ash	p.p.m.	percent- age on ash	p.p.m.	percent- age on ash
1931-35 inclusive	3	2.8	28	12.7	42	17.7	5	3.5
1936-40 inclusive	8	10.2	13	10.1	17	13.7	10	8.9
Grand average	6	6.5	20	11.5	29	16.1	8	6.3
Molasses SOs	*0.012	3.9	0.018	3.9	0.031	5.5	0.015	3.5
Thick juice SOa	**0.069	3.2	0.114	4.9	0.119	4.1		

\*Quantity expressed as percentage SOa on beets.

\*\*Quantity expressed as percentage SOs on solids.

In the case of chlorides, table 4, the trend is again the same; the chloride content is highest in the two central areas so far as concentration in sugar is concerned. The proportions on ash, however, are high in the far west and very low in the east, and intermediate in the central areas. Figures for the thick juices and molasses confirm the relatively high amounts of chlorides in sugars from the central areas. Chlorides are known to be taken up by the sugar to a lesser degree than sulfates and this is indicated in the data for the central areas by the lower proportion of chlorides in sugar ash; however, it is not shown by the values for the other sections. Considering the scanty tests available on the thick juices and molasses, such a discrepancy is not surprising.

Table 4.—Chloride (Cl) content of sugars.

	West		Internountain		Central plains		East	
	p.p.m.	percent- age on ash	p.p.m.	percent- age on ash	p.p.m.	percent- age on ash	p.p.m.	percent- age on ash
1931-35 inclusive	13	12.3	16	7.3				
1936-40 inclusive	7	9.0	12	9.3				
Grand average	10	10.5	14	8.0				
Molasses	*0.007	2.3	0.072	15.6	0.069			
Thick juice	**0.061	2.8	0.029	9.1	0.264			

\*Quantities in molasses expressed as percentage on beets.

\*\*Quantities in thick juice expressed as percentage on solids.

Potash, table 5, is also relatively high in the sugars from central areas, and there are similar differences in the case of soda, table 6. However, the amounts of soda can not be interpreted as representing beet composition because of the addition of soda ash in processing. The use of 1 pound of soda ash per ton of beets may double the natural soda content and greatly alter the ratio of potash to soda and the proportion of soda in the ash. The low ratios of potash to soda (see table 7) in the west is apparently due to the greater use of soda ash as seen by the averages for the past 5 years.

Table 5.—Potash (IvaO) in sugars.

	West		Inter mountain		Central plains		East	
	p.p.m.	percent- age on ash	p.p.m.	percent- age on ash	p.p.m.	percent- age on ash	p.p.m.	percent- age on ash
1931-35 inclusive	31	29.3	84	38.2	91	38.4	56	38.9
1936-40 inclusive	21	26.9	46	35.7	41	33.1	40	35.7
Grand average	26	28.3	65	37.8	66	36.7	48	37.5
Molasses	*0.100	32.2	0.193	41.9	0.234	41.8	0.197	45.8
Thick juice	**1.015	46.5	1.266	55.0	1.462	50.9		

\*Potash in molasses expressed as percentage on beets.

•Potash in thick juice expressed as percentage on solids,

Table 6.—Soda (NasO) in sugars.

	West		Intermountain		Central plains		East	
	p.p.m.	percent- age on ash	p.p.m.	percent- age on ash	p.p.m.	percent- age on ash	p.p.m.	percent- age on ash
1931-35 inclusive		15.1						
1936-40 inclusive		15.4						
Grand average		15.2						7.0
Molasses	*0.	19.0			0.144			16.5
Thick juice	0.286	13.1			0.341			

\*Soda on molasses expressed as percentage on beets,

•Soda in thick juice as percentage on solids.

Table 7.—Ratio of potash to soda in sugars and soda ash used.

	West	Intermountain	Central plains
1931-35 inclusive	1.9	4.2	3.8
1936-40 inclusive	1.8	4.6	3.4
Grand average	1.9	4.3	3.7
Molasses	1.7	2.2	1.6
Thick juice	3.5	6.4	4.3
Soda ash used			
1936-40 inclusive	*1.01	0.22	0.17
Kequivalent Na <sub>2</sub> O, percentage beets	0.028	0.007	0.005

\*Pounds per ton beets.

Sulfites and lime data which are given in tables 8 and 9 are presented to more or less complete the picture for the principal mineral constituents in sugars. The sulfites are, of course, derived from the sulfuring used in processing and therefore sulfite content is of no direct interest, but it is necessarily taken into account in some calculations which we wish to present. The lime content of sugar is entirely unrelated to the lime content of the beets, being dependent on the amounts of soda ash used and the amounts of inherently soluble lime salts formed in processing. The amount of lime salts left when no soda ash is used is a factor of beet composition as well as of oper-

Table 8.—Sulfites (SO<sub>2</sub>) in sugars.

	West		Intermountain		Central plains		East	
	p.p.m.	percent- age on ash	p.p.in.	percent- age on ash	p.p.m.	percent- age on ash	p.p.m.	percent- age on ash
1931-35 inclusive	8	7.5	30	13.6	30	12.6	12	8.3
1936-40 inclusive	5	6.4	14	10.8	13	10.5	8	7.1
Grand average	7	7.6	22	12.6	22	12.2	10	7.8
Molasses	•0.002	0.6	0.004	0.9	0.014	2.5	0.003	0.7
Thick juice	**0.052	2.4	0.111	4.8	0.115	4.0		

•Sulfites in molasses expressed as percentage on beets.

••Sulfites in thick juice expressed as percentage on solids.

Table 9.—Lime (CaO)

	West		Intermountain		Central plains		East	
	p.p.m.	percent- age on ash	p.p.m.	percent- age on ash	p.p.m.	percent- age on ash	p.p.m.	percent- age on ash
1931-35 inclusive	7	6.6	14	6.4	11	4.6	12	8.3
1936-40 inclusive	7	9.0	7	5.4	6	6.5	9	8.0
Grand average	7	7.6	10	5.7	8	4.4	10	7.8
Molasses	•0.004	1.3	0.006	1.3	0.005	0.9	0.009	2.1
Thick juice	••0.079	3.6	0.031	1.3	0.044	1.5		••

•Lime in molasses expressed as percentage on beets.

••Lime in thick juice expressed as percentage on solids.

Table 10.—

A.—Potash equivalent of total combined mineral-acid radicals and of total combined organic-acid radicals i)

	West		Intermountain		Central plains		East	
	Inor- ganic p.p.m.	Or- ganic p.p.m.	Inor- ganic p.p.m.	Or- ganic p.p.m.	Inor- ganic p.p.m.	Or- ganic p.p.m.	Inor- ganic p.p.m.	Or- ganic p.p.m.
1931-35 inclusive	33	35	97 *	39	110	36	26	63
1936-40 inclusive	29	22	53	20	53	15	29	38
Grand average	31	28	76	29	81	26	28	50
Molasses	•0.026	0.170	0.123	0.211	0.149	0.313	0.038	0.282
Thick juice	••0.239	1.343	0.574	1.042	0.659	1.306		

•Potash equivalents on molasses expressed as percentage beets.

••Potash equivalents on thick juices expressed as percentage solids.

B.—Proportion of total bases (K<sub>2</sub>O equivalent) combined organically.

	West		Intermountain		Central plains		East	
	percentage		percentage		percentage		percentage	
1931-35 inclusive	51.5		28.7		24.9		70.6	
1936-40 inclusive	43.7		26.9		22.5		57.1	
Grand average	48.2		27.4		24.2		64.0	
Molasses	86.6		63.3		67.8			
Thick juice	84.9		64.5		67.9			

ating technique. Taking into consideration the lime content of the sugars, or the lime in the first liquor, and the amounts of soda ash used, it is indicated that the natural amounts of lime salts are greater in the west and east than elsewhere.

Lime and sulfites are both taken up by the sugar selectively, as is indicated by the increased percentages on sugar ash as compared with the thick juices and molasses. For example, while the lime percentage on ash of molasses is generally below 2 percent, on ash of sugars it is around 6 percent; potash correspondingly drops from about 42 percent on molasses ash to approximately 37 percent on sugar ash. This occurs in spite of the fact that the concentration of potash in the thick juices or first liquor is tremendously high as compared with the lime concentration.

Finally we come to a consideration of the ash alkalinities and the indicated organically combined bases. As ash alkalinities were not directly determined prior to the last season, it is necessary to estimate same on the basis of the ash analyses. For this purpose the sum of the ionic-equivalent amounts of the bases (potash, soda and lime) was found and from this was subtracted the sum of the ionic-equivalent amounts of the mineral-acid radicals (sulfate, chloride and sulfite). The difference represents an estimate of the ionic-equivalent amount of combined organic-acid radicals. It seems best for the purposes of this paper to calculate this derived value to potash, and similarly the total, mineral, ionic equivalents to potash. In short, we will assume for the sake of convenience that the only base present is potash; then we can give a comparison of the amounts of bases combined with mineral acids and those combined with organic acids on a uniform basis. Table 10 represents the calculated amounts of potash combined in these two ways and the proportions of total bases (expressed as potash) combined with organic-acid radicals.

We have previously seen that sulfates, chlorides, and potash are relatively high in the beets and sugars of the two central areas; we now see that the condition in respect to organic salts is the reverse, especially on the proportion basis. This is equally evident in the thick-juice arid molasses analyses.

Having seen the general trends indicated by the gross averages, it has seemed desirable to make sure that such extensive averaging has not led to conclusions which are not evident in the individual sugars. From the averages of the sugars from the individual factories for the past 5 years were selected the sugars of the two factories having the lowest ash in each region, one Steffen and one non-Steffen; similarly in each region were selected the two factories with the highest ash. In each case the data for the Steffen and non-Steffen sugars were averaged.



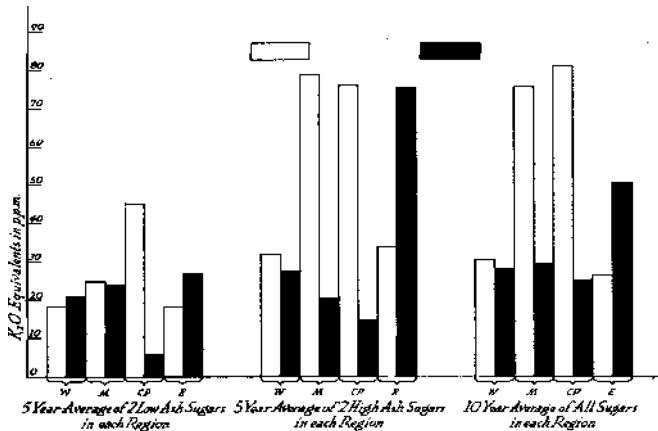


Figure 2.—The comparison of the potash equivalents for the total combined mineral-acid radicals and organic-acid radicals.

Figure 2 shows graphically the calculated potash equivalent of the inorganic-acid radicals and likewise the potash equivalent of the combined organic-acid radicals for these low-ash and high-ash sugars and for the 10-year grand averages which have been previously presented. In each group of figures it will be noted that in the West the inorganically and organically combined potashes are nearly equal. In the two central areas the inorganic potash is very high compared with the organic potash. Only in the low-ash sugars for the intermountain area are the two types of salts near equality. In the eastern area the organically combined bases exceed the mineral salts by large amounts.

Viewed a little differently, it may be noted that the difference between low and high ash is largely due to increased mineral components in all areas except the East where increases in organically combined bases predominate. Again the mineral contents of the western and eastern sugars are nearly equal in all three comparisons, and the differences in ash are due to the larger amounts of organically combined bases in the JBast.

The picture presented by this figure 2 summarizes the observations to which we have called attention, namely: (1) The tendency to higher-ash sugars in the intermountain and central plains areas is due to relatively higher concentrations of sulfates and chlorides;

(2) the low-ash tendency of the west coast sugars is due to low concentrations of both inorganic and organic salts, and (3) the moderately high ash of the eastern sugars is due to high concentrations of organic salts and in spite of low values for inorganic salts. In addition, from portions of the tables presented earlier, we may say that the differences in the salt components of the sugars are probably rather directly related to the composition of the corresponding beet non-sugars. The high and low ashes in a particular region may, however, be partly due to (1) differences in the amount of raw sugars on percentage of juice solids that are recirculated, (2) sugar-boiling conditions, and (3) effectiveness of washing in removing external surface of the sugar crystals which are high in ash. A more comprehensive study is in progress on white massecuites and molasses in hope that general relations may be found which will indicate whether at each particular factory the quantity of sugar ash is affected more by beet composition or by the thoroughness of refining operations.

## The Measurement of Color and Turbidity in Granulated Beet Sugars

R. A. MCGINNIS AND E. E. MORSE<sup>1</sup>

In the early days of beet-sugar manufacturing, the measurement of white-sugar color and turbidity did not present serious problems. As long as the product was one which was white to visual inspection, all requirements were satisfied. The last 20 years, however, have been marked by steady and successful efforts to improve various sugar-quality factors, until today minute differences which are totally invisible excepting to the highly trained eye are regarded of great importance. Unfortunately, in the case of color and turbidity at least, the skill of the research workers and instrument designers has not been adequate to keep pace with the requirements.

The result is that scarcely any two beet-sugar companies use the same method. Turbidity is seldom measured. As far as color is concerned there are innumerable methods in use. The most official method is probably that of Keane and Brice, since this is used by the Carbohydrate Division of the United States Department of Agriculture in their annual survey of the Nation's beet sugars. However, their measurements cannot be duplicated or checked by anyone else.

<sup>1</sup>Spreckels Sugar Company. This paper was presented by P. W. Alston, General Chemist.