higher-moisture equivalent than the true value. Duplicate samples are taken from the sieved soil.

Fit each of the special centrifuge cups with a small piece of filter paper. In each cup place 32 grams to 34 grams of the sieved sample of air-dry soil, depending upon the moisture content, so as to make 30 grams of an oven-dried sample. It is not necessary to make the samples smooth when placed in the cups.

Place the 16 centrifuge cups in a tin cakepan and add about 500 cc. of tap water. This amount should completely cover the surface of the soil in the cups. The samples are then allowed to stand in the saturated condition from 18 to 24 hours. A variation of 6 hours makes little or no difference in the moisture equivalent.

Tilt the cakepans so as to allow drainage for a period of 15 to 30 minutes. Place the cups in the special centrifuge head and bring them up to a speed of 2,440 r.p.m. (1,000 x gravity) within 5 minutes. Centrifuge for an additional 30 minutes at this speed. Remove the samples from the centrifuge cups and filter papers. This is done by inverting the cups on a clean sheet of paper and then tilting the paper so as to slide the sample into the weighing cans.

Place the lids on the weighing cans and weigh them. After weighing, remove lids and place in the oven and dry for 20 to 24 hours at 110° C. Re-weigh and calculate the percentage of moisture on a dry basis. This is the moisture equivalent. Duplicates should check within 0.9 percent.

A Study of the Accumulation of Chlorides and Their Effect on Beet-Sugar Factory Operations

HEBER C. CUTLER AND R H. WOOLLEY 1

The following discussion deals with an investigation of beetsugar manufacturing operations made for the purpose of determining some of the causes for the variations in yields and losses observed at different factories when operating under apparently identical conditions.

The non-sugars always present in sugar beets show wide differences in both composition and quantity, which depend on the agricultural methods used and on the geographical location of the fields in which the beets are grown. In a general way it may be said that

Chemists, Utah-Idaho Sugar Company.

beets grown in soils that are overly rich in soluble salts suffer in quality regardless of how satisfactory the quantity may be. Beet roots grown in these salt-rich soils, themselves become strongly charged with the salts, especially those of sodium and potassium since the compounds of these metals usually found in soils are much more soluble than are the salts of calcium, magnesium, iron, etc.

The processing plant, from the records of which most of the data gathered in this investigation were taken, is largely supplied with beets grown in salty soils. Analyses show that they contain a high percentage of chlorides, principally sodium chloride or common salt (table 1). These chlorides are, of course, readily extracted along with the sugar in the diffusion battery and so find their way into the diffusion juice. Since the final diffusion juice is made up only of the juice from the beets and the diffusion water, it follows that all chlorides found in the diffusion juice must come from these two sources. In the case of the plant under consideration it happens there is insufficient condensed water to supply all the demands and it is necessary to use raw water to supply the battery.

| | Period 1 | | Teriod 2 | | Ferlod 3 | |
|--------------------------|------------------------------|--------------------|------------------------------|-----------------|-----------------------------|----------------------|
| - | Chiorides on 100 D. S. | Percent Age | Chlorides on 100 D. S. | Percent- êgo | Chloride on 100 D. S. | B Percent- age |
| Juice from beets | _· · | | | | | |
| (63 percent) | .0804 | 82.0 | .0971 | 86.9 | .0711 | 84.5 |
| Diffusion water | | | | | | |
| (37 percent) | .0201 | 18.0 | .0249 | 13.1 | _0222 | 15.5 |
| Diffusion juice | | | | | | |
| (100 percent) | 0176 | | .7040 | | .5300 | |
| Sweet water | .7690 | | .7440 | | ,4940 | |
| Thin juice | .7923 | | .6940 | | .5430 | |
| Increase | | 48.7 | | 50.2 | | 12.1 |
| White massecuite | .6210 | 60.3 | | | | |
| Intermediate massecuite | 1.1427 | 53.6 | | | · | |
| Low raw massecuite | 1.3100 | 79.2 | | | | |
| High green sirup | 1.1349 | | | | | |
| Intermediate green sirup | 1.3200 | | | | | |
| Molasses | 2,0770 | 91.5 | 1.018 | 93.8 | 2.020 | 95.2 |
| Low raw sugar (unwashed) | | | | | | |
| Low raw sugar (washed) | .0730* | | .388 | | .206 | |
| Low raw remelt | .3844 | | .620 | | .350 | |

| | Тя | Եյե | 1. |
|--|----|-----|----|
|--|----|-----|----|

*Excessive.

The experimental work in connection with this investigation was performed during three separate periods, each of about the same duration and involving about the same quantities of beets.

The first period covers the entire campaign of 1940 which was relatively short. The second covers the first 6 weeks of the 1941 campaign. During these two periods, which are comparable, the entire output of low raw sugar, which had been remelted with diffusion juice, was returned to the first carbonators. During the last period a change was made in that the low raw sugar remelted with thin juice instead of diffusion juice was introduced into the green sirup for boiling to intermediate massecuite. These are the periods referred to in tables 1 and 2.

Table 2 .--- Chlorides in factory products.

Period No. 1 Period No. 2 Period No. 3

All figures in percentages

| Beets 63 | | | |
|---|------------------------------|------------------------------|------------------------------|
| Water 37 | 100.0 | 100.0 | 100.0 |
| Thin juice (increase) | 48.7 | 50.2 | 12.1 |
| Remelt | 20.8 | 16.G | None |
| | | | |
| Thin juice | 27.9 | 33.6 | 12.1 |
| Elimination to thin juice | 8.5 | 6.2 | 4.8 |
| Elimination in molasses | 91.5 | 93.8 | 95.2 |
| Elimination of non-sugars in the beets | 33.80 | 38.66 | 40.92 |
| Purity thick juice | 89.9 | 89.9 | 90.35 |
| Elimination in molasses Elimination of non-sugars in the beets | 27.9 8.5 91.5 33.80 | 33.6 6.2 03.8 38.66 | 12.1 4.8 95.2 40.92 |

The quantities of chlorides introduced into the process by the beets and the water were found to be approximately the same during all three periods (table 1). The actual amounts of chlorides as CI and calculated to 100-dry substance are as follows:

| | | <u> </u> | |
|-------------------------------------|------------------------------|------------------------------|------------------------------|
| | Period 1 | Period 2 | Period 3 |
| Juice from beets Diffusion water | Dercentage .0804 .0301 | percentage .0971 .0249 | percentage .0711 .0222 |

Considering the combined chlorides from both sources as 100 percent, the relative amounts from each are as follows:

| | Period 1 | Period 2 | Period 3 |
|-----------------------------|----------|----------|----------|
| Percentage of CI from beets | 82.0 | 86.9 | 84.5 |
| Percentage of CI from water | 18.0 | 13.1 | 15.5 |

The total chlorides when calculated on the dry substance in the diffusion juice were then found to be for period 1, .6176 percent; period 2, .7040 percent; period 3, .5300 percent.

These chlorides, which represent a high percentage of the nonsugars in the juice, are not removed by the lime defecating processes in common use, but are carried in solution with the juice from the carbonators. Also, since it is common practice to return the first wash water from the filter presses to the juice and to use the **last** washings in the lime slacker, it will be seen that no chlorides are eliminated except for about 3.00 percent which were found to remain in the filter press cake (table 3). The remaining 97.00 percent of original chlorides present in the diffusion juice are carried on into the processing operations where no further elimination takes place. Not only is there no elimination, but on the contrary there will he a building up of the original chlorides in the evaporator thin juice as a result of returning the filter wash water to the juice. This condition is still further aggravated when the remelted low raw sugar is added to the diffusion juice as is shown in tables 1 and 2.

This build-up of the chlorides was clearly shown by analysis of the thin juice which indicated there had been an increase of 48.7 percent chlorides for the first period and 50.2 percent for the second period while low raw remelt was being returned whereas in the third period when no remelt was added the increase of chlorides was only 12.1 percent and an accompanying gain of juice purity of 0.45 percent was realized. This increase in purity in itself represents a gain of 0.13 pound of granulated sugar equivalent per ton of beets. A further gain was realized in that the quantity of intermediate massecuite was increased from 15.3 percent on the weight of beets during the first two periods to 19.5 percent on beets during the third period. In turn, this reduced the amount of raw fillmass from 8.88 percent on beets during the first two periods to 8.64 percent on beets during the third period. Consequently less molasses was produced and a reduction of 2 percent in the sugar content was made, also, the corresponding reduction in sugar lost in the molasses was found to amount to .285 percent on the weight of the beets for the third period.

Table 3.-Filter-press washings.

Test No. 1

| Table No. 1 | Brix | Chlorides on 100 brix | Percentage |
|--------------------------|------------------|---------------------------------|------------|
| Juice to presses | 14.2 | .4788 | 100.0 |
| First wash to juice | 12.7 | .4805 | · |
| Second wash to lime milk | 11.8 | .4600 | |
| Third wash to lime milk | 11.2 | .4910 | |
| Fourth wash to lime milk | 10.0 | .4900 | |
| Fifth wash to lime milk | 1,4 | .3286 | |
| Lime cake (on D S) | | .0305 | 6.3 |
| | Test No. Brix | . 2 Chlorides on 100 brix | Percentage |
| | | | |
| Juice to presses | 10.2 | .5400 | 100.0 |
| First wash to juice | 8.5 | .6400 | |
| Second wash to lime milk | 7.0 | .7100 | |
| Third wash to lime milk | 4.9 | .7800 | |
| Fourth wash to lime milk | 2.5 | .0000 | 1 -4 |

1.7

.5900 (combined)

3.20

.0166

Fifth wash to lime milk

Lime cake (on D S)

580

That definite gains were realized by adding the low raw-remelt to the green sirup rather than to the diffusion juice is indicated by referring to the factory records for the periods being discussed. These show an actual increase of 5.7 pounds of granulated sugar produced from each ton of beets.

The contention of the authors that the presence of large amounts of chlorides has a definitely detrimental effect on the operation of a beet-sugar factory is borne out by making a few comparisons between the conditions in the plant discussed above and those in another plant where the battery water is exceptionally pure and where the beets being cut are of about the same purity and sugar content but practically free from chlorides. Calling the plant where high chlorides prevail No. 1, and the plant where chlorides are low No. 2, the following tabulation will make the differences apparent.

| | Plant No. 1 High CI | Plant No. 2 Low CI |
|--|------------------------|-------------------------------|
| Percentage purity of thick juice. Percentage elimination of non-sugars in beets. Chlorides in thick juice on 100 brix. | 37.7 _6764 | 92.2 45.3 .0346 5.20 |

The quantity of non-sugars in the beets is approximately the same in both cases, but they do not respond to the defecation process to the same extent and since analyses show that chlorides form a large proportion of the non-sugars at plant No. 1 and a relatively small proportion at plant No. 2 it would seem safe to assume that chloride difference is responsible, to a considerable degree, for the variation in the results attained.

"We conclude from this investigation that beet-sugar factories operating in regions where relatively high chloride concentrations occur, whether in the beets or process water, plant operations should be regulated so as to avoid as far as possible return of the secondary products to juice end of the house.

Appendix

The methods used for the determination of chlorides were the accepted volumetric methods outlined by Scott in his "Standard Methods of Chemical Analysis second edition.

In all cases Mohr's silver-chromate method was used without clarification where the solutions were sufficiently light in color, otherwise the solutions were clarified by alumina cream and filtered. As a check against the above method, Vohlhard's silver thiocyanate-ferric alum method was used occasionally.

The authors' thanks are due in particular to Wendell Holmes, student chemist, and to the factory-operating superintendents for their assistance and cooperation during this study.