### COMPOSITION OF THE SOLUBLE NON-SUGARS IN BEETS AND THEIR ELIMINATION DURING PROCESSING

### By A. R. Nees

Every step in the sugar refining process is directly concerned with, or is incidental to the separation of sugar from the accompanying non-sugars.

In the processing of beets there are two steps which are chiefly concerned with the elimination of non-sugars, - the diffusion battery and first carbonation. Average beets contain about 135 to 140 pounds non-sugar, other than water, per ton. Aproximately 90 to 100 pounds of insoluble matter in form of pulp is removed in the battery and 35 to 40 pounds is carried into the raw juice. The carbonation process removes 12 to 15 pounds. The remainder goes on into the sugar end and eventually passes out with the molasses.

Every sugar technologist since the beginning of the industry, has given attention to both the diffusion and carbonation operations, with a view to increasing the elimination of non-sugars, thus increasing the yield of granulated sugar and decreasing the quantity of molasses.

The chief function of the battery is to separate soluble constituents of the beets from the insoluble.

Some believe that osmosis or "selective diffusion" take place in the battery and that it may be so operated as to improve the purity of the diffusion juice. It is probably true that certain colloidal cellular matter is coagulated by heat and mechanically filtered out in the battery. Beyond this there is not much evidence that soluble non-sugar is removed. On the other hand there is much evidence that, under conditions of high temperature and a long time of retention, hydrolysis or dissolution of certain constituents such as hemi-celluloses and pectin-like compounds occur, thus causing a lowering of purity. The elimination of impurities in the battery is only of importance if something is removed which cannot be removed by lime and carbonation.

So far as the removal of colloidal and soluble non-sugars is concerned the standard practice is, of course, the treatment with milk of lime or calcium saccharate and subsequent removal by carbonation and filtration. Many methods have been proposed to supplant or supplement the lime treatment, but none have proven feasible from an economic standpoint. Many variations in technic have been adopted from time to time with the idea of improving the precipitation and removal of impurities. Their value is uncertain. At times good results are obtained with little effort - at other times poor results are obtained in spite of all one can do.

It was this uncertainty which prompted the inception of the work reported in this paper. It was often noticed that beets from adjoining territories gave different results in the respective factories in which they were processed. This was particularly noticeable in two Colorado districts. The factories are of comparable size, have similar equipment and maintain, as far as possible, the same operating conditions. Yet one with poorer quality beets invariably obtained a higher elimination of non-sugars as measured by the purity of thin juice and molasses production. The average figures for the fiveyear period immediately preceding the tests are shown in Table 1. -299-

### TABLE I

			Sngar Percentage on Beets	Apparent Purity Beets	Apparent Purity Thin Juice	Molasses Percentage on Beets
District	No.	12	15.30	83.9	88.8	1.99

Table II shows similar figures for the year in which the tests were made.

	Sugar Percentage on Beets	Apparent Purity Beets	Apparent Purity Thin Juice	Sugar in Molasses Percentage on Beets	
District No. 1	14.61	84.6	89.0	2.24	
District No. 2	14.61	84.7	87.7	2.27	

The difference in the results obtained by the two factories is obvious and this investigation was undertaken to determine the reason for the difference.

The tests were carried out in accordance with the following procedure: Apparent purities (refractometer dry substance and direct polarization) were run on daily composite samples of pressed juice, diffusion juice and thin juice (second saturation juice) and on colloid-free pressed and diffusion juice. All daily samples were concentrated and composited by weeks for more complete analysis. Corresponding composite samples of molasses were also obtained. Analyses were made on all weekly samples for total nitrogen, proteins, non-protein nitrogen, nitrate nitrogen and lixiviated ash.

Non-protein nitrogen compounds were calculated from the non-protein nitrogen by multiplying by the factor 9.0. The non-nitrogenous organic compounds were determined by difference. This is admittedly an empirical method but it is believed to permit a fair comparison of the composition of the various juices.

Finally all results were reduced to campaign averages. It is these latter figures which will be discussed.

The various non-sugars are shown in Chart I and Table III, expressed as pounds per ton of beets. This method of expression makes it possible to compare not only the different juices from the same district, but to compare those of the two districts, since the beets have the same average sugar content.

First, compare the composition of pressed juice from the two districts. The total non-sugars in No. 1 is 43.4 lbs. per ton of beets and in No. 2, 43.7, practically the same. The inorganic compounds are also the same, being 13.5 pounds per ton of beets. There is a decided difference in nitrogenous compounds. Proteins, non-protein nitrogen compounds and nitrates are all higher in the beets from District No. 2, by 1.2, 1.3 and 0.5 pounds respectively. The non-nitrogenous organic compounds are lower by 2.9 pounds.

TABLE II

Comparison of the non-sugars in pressed juice and diffusion juice is of interest from the standpoint of so-called "battery elimination".

There is a reduction in the total non-sugar in diffusion juice as compared to pressed juice of 8.6 pounds per ton in No. 1 beets and 4.6 lbs. in No. 2 beets. The difference is largely accounted for by an increase in ash and only a slight decrease in non-nitrogenous organic in No. 2 beets, while there is a decrease in ash and a relatively large decrease in non-nitrogenous organic compounds in No. 1. The increase in ash in No. 2 beets is partly due to solids in the battery supply water and the addition of soda ash to the battery, 0.55 pound and 0.22 pound per ton of beets coming from these respective sources.

(Battery supply water for No. 1 factory had the equivalent of 0.06 pound solids per ton of beets and no soda was added to the battery.)

After making these corrections, No. 1 beets still show 0.52 pound per ton greater reduction in ash than No. 2. Apparently there is an elimination of substantially more ash constituents from No. 1 beets in the battery. This point will be discussed later.

The comparison of diffusion juice with thin juice shows the effect of liming and carbonation on the removal of non-sugars. The most obvious thing is that the thin juice from No. 2 beets contains 3.1 pounds more non-sugar than No. 1. When the proper corrections are made for solids in battery supply water and soda ash used in process in both factories, the difference is reduced to 2.4 pounds. This amount of non-sugar will carry 3.6 lbs. of sugar into the molasses at 60 purity or increase the sugar in molasses 0.18% on beets. The reason for this difference in elimination lies in the fact that No. 1 beets contain a relatively large amount of non-nitrogenous compounds which are readily eliminated, while No. 2 beets contain a large amount of nitrogenous compounds which are not eliminated. This results in the production of lower purity thin juice and consequently a higher molasses production from No. 2 beets. The elimination of other types of non-sugar is practically the same for both districts.

Colloid-free pressed and diffusion juices were prepared by treating with colloidal aluminum hydroxide. The alumina hydrosol carries a positive electric charge while the juice colloids are negatively charged. A mutual precipitation occurs when the two types of colloids are brought together. Chart I and Table III show the amount of each type of non-sugar precipitated by alumina hydrosol. The remainder is assumed to be in true solution.

Practically all of the proteins are removed but only a small part of the non-protein nitrogen compounds. Beets No. 1 show 50% greater ash removal in colloidal form than No. 2. Again the most marked difference is in the nonnitrogenous organic compounds. Forty per cent of these compounds are precipitated from the pressed juice of No. 2 beets and 35% from that of No. 1 beets, while only 16% are precipitated from the diffusion juice of No. 2 and 23% from No. 1. It is to be noted too that there is more of these compounds in the colloid-free diffusion juice than in the pressed juice of No. 2 beets, while the reverse is true for No. 1 beets. It is indicated that some of these compounds in No. 2 beets are changed from the colloidal to the true solution state in the battery. Apparently, however, they are removed by lime so that the net effect of this conversion on the purity of thin juice is negligible.

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The composition of non-sugar in molasses is the same as that of thin juice, with the exception of the non-nitrogenous organic, which shows an increase of 2.2 pounds per ton of beets for District No. 1 and 2.3 pounds for No. 2. This increase is the result of the inversion of sugar during the process and is made up of invert sugar and its decomposition products.

Table IV shows the composition of the inorganic compounds. As pointed out before, total inorganic compounds, expressed as lixiviated ash, is the same in the pressed juice from the beets of both districts, and there is less ash elimination from No. 2 beets even after a correction is made for solids in battery supply water and soda ash. The reason becomes apparent when it is noted that there is an increase in alkali compounds from pressed juice to diffusion juice which, of course, carries through to thin juice. These additional potassium and sodium compounds evidently come from the hydrolysis of alkaliorganic compounds in the battery. Whether this hydrolysis is influenced by the method of battery operation or is an inherent characteristic of the beets is not known. It is known, of course, that calcium, magnesium and phosphorus compounds are removed at carbonation. This is clearly indicated by the figures in the table. Practically none of the chlorides are eliminated. Forty-seven per cent of the sulfates are eliminated from No. 1 beets while there is an increase of 25% in the thin juice from No. 2 beets. This latter figure is obscured by an unknown addition from battery supply water.

The association of the inorganic substance in the colloidal phase shows some interesting variations. For example, in No. 1 beets both sodium and potassium are removed with the colloids. In No. 2 beets neither of these is removed, but instead an equivalent amount of magnesium is precipitated. Practically all of the calcium, silica, iron and alumina, and about one-half the phosphorus are shown to exist in colloidal condition.

If one were able to resolve the organic substance into their component parts as easily as the inorganic, a nore complete picture would be obtained and the problem of elimination could be attacked in a more intelligent manner. It is apparent that 80% to 85% of the non-sugars in thin juice are composed of two groups, inorganic and non-protein nitrogenous compounds. It is evident, therefore, that any improved method of juice purification will be successful only in proportion to its ability to remove those substances. Such a method will have to be a radical departure from the present one, probably a supplemental method which will follow the line and carbonation process. The development of such a method is not impossible from a technical standpoint. The economic factors will govern its application.

This discussion has pointed the difference in the relative proportion of the various non-sugar in beets which have the same sugar content and purity. This difference is undoubtedly a function of soil fertility, agricultural nethods and climatic conditions. The contention of many operating men that factory results are influenced by the quality of beets is borne out by these results, although it is doubtful if all operating troubles can be ascribed to this source.

#### Acknowledgment

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## TABLE III

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## POUNDS NON-SUGARS PER TON BEETS

		Non- Protein				Non- Nitrogenous		
	Protein Compounds	Nitrogen Compounds	Nitrates N205	Ammonia NH <sub>Z</sub>	Inorganic	Organic Compounds	Total	Purity
			DISTRI	CT NO. 1				
Pressed Juice	3.42	9.05	0.82	0.17	13.51	16.45	43.43	*86.05
Colloid-free Pressed Juice	0.30	8.20	0.88	0.15	12.15	10.54	33.25	88.97
Diffusion Juice	1.22	8.00	0.74	0.15	12.97	11.78	34.80	88.63
Colloid-free Diffusion Juice	e 0.38	7.93	0.77	0.16	12.25	8.91	30.61	89.63
Thin Juice (2nd Saturation)	0.00	7.50	0.77	0.03	11.75	3.84	23.74	91.98
Molasses	0.00	7.50	0.77	0.03	11.70	6.10	26.20	**61.63
			DISTRI	CT NO. 2				
Pressed Juice	4.63	10.31	1.34	0.26	13.55	13.56	43.65	*85.98
Colloid-free Pressed Juice	0.04	9.84	1.26	.25	12.49	8.30	32.17	89.00
Diffusion Juice	1.66	9.91	1.15	.25	12.97	12.07	39.11	87.43
Colloid-free Diffusion Juice	0.12	9.54	1.20	0.24	13.52	10.17	34.80	88.68
Thin Juice (2nd Saturation)	0.19	8.99	1.27	0.04	12.79	3.48	26.76	90.95
Molasses	0.04	9.07	1.21	. 0.03	12.70	5.80	28.80	**60.39

\*Purity by refractometer dry substance and direct polarization.

\*\*True purity, double enzyme method.

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# TABLE IV

### ASH CONSTITUENTS POUNDS PER TON OF BEETS

	Pressed Juice	Colloid-free Pressed · Juice	Diffusion Juice	Colloid-free Diffusion Juice	Thin Juice	Mólasses
		DISTRICT	NO. L			
Si02 R203 Ca0 MgQ K20 Na20 Cl S03 P205 C02 (Calc <sup>1</sup> d)	0.09 0.19 0.40 1.04 5.03 1.93 1.62 0.69 0.69 2.26	0.02 0.06 0.06 1.00 4.86 1.84 1.67 0.61 0.32 2.11	0.03 0.06 0.15 1.03 5.14 1.92 1.46 0.43 0.53 2.43	0.01 0.09 0.06 0.86 5.04 1.87 1.58 0.43 0.35 2.32	0.02 0.01 0.05 0.02 5.21 2.07 1.55 0.34 0.04 2.74	0.02 0.05 0.12 0.01 5.19 2.11 1.60 1.54 0.03 2.14
		DISTRICT	NO. 2			
SiO2 B2O3 CaO MgO K2O Na2O Cl SO3 P2O5 CO2 (Calc <sup>t</sup> d)	0.09 0.20 0.28 1.04 5.10 2.03 1.20 0.46 0.77 2.66	0.02 0.08 0.04 0.84 5.09 2.01 1.34 0.50 0.31 2.57	0.05 0.11 0.93 5.56 2.20 1.18 0.75 0.64 2.82	0,03 0.11 0.04 0.67 5.55 2.25 1.31 0.69 0.34 2.85	0.02 0.00 0.07 0.02 5.49 2.40 1.19 0.58 0.40 3.25	0.02 0.02 0.08 0.02 5.55 2.40 1.28 1.73 0.03 2.57



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43.7 Pressed Juice Colloid Free P.J. 12.5 32.2 9.8 à.a 14.1 Diffusion Juice 12.1 39.1 Colloid Free D.J. 34.8 9.5 10.2 13.5 Thin Juice 26.8 12.8 3.5 Molasses 28.8 5.8

Chart No. 1 Pounds Non-Sugars per Ton of Beets