

THE EFFECT OF SODIUM ALUMINATE
ON BEET JUICE EVAPORATION

by

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INTRODUCTION

The primary objective of this investigation was to ascertain the effect of small quantities of commercial sodium aluminate on quantity of evaporator scale, purity rise, and colloidal decrease of beet juice.

A survey of the literature revealed that very little quantitative work had been done in which sodium aluminate was used in a direct attempt to accomplish the objectives of this investigation. The work of Brown (8) was concerned primarily with the decrease of the amount of colloids in beet juices; in his experiments he used chromium oxide, iron oxide hydrosols, and alumina hydrosol. Jeanprost (15) increased purity, and decreased colloids and coloring materials in beet juices by treating with a glauconite previously charged with aluminum hydroxide which he designated as aluminate. These experiments are, however, only slightly similar to this investigation.

The work of Wayne (31) resembles in some respects that carried on in this study. In his experiments, in which he used sodium aluminate, results were obtained on cane juice during actual campaign; he studied primarily clarity, properties of color, settling rate, purity, and colloids. His

methods of procedure were quite different from those followed in this work.

From a review of the literature it was concluded that studies of the effect of sodium aluminate addition on beet juice might prove both interesting and profitable.

Scale on boiler tubes, evaporator tubes, in pipe lines, valves, etc., has always been an important chemical engineering problem. Much progress has been made in recent years in the study of scale formation. However, the complete elimination of scale on evaporator tubes in beet sugar mills has never been accomplished.

The presence of scale on sugar evaporator tubes has several important economic effects in sugar mill operation. It is the object of every responsible person in a sugar house to keep scale formation at a minimum. Scale of any thickness lowers the heat transfer coefficient, which results in the use of more steam in accomplishing the work necessary for the evaporation of a definite volume of thin juice to the required density. Evaporators are the key or governing point in a sugar house; thus with any increase in scale there will be a proportionate slowing down of plant operations both before and after evaporation. Dirty tubes decrease the number of tons of beets sliced per day, as well as the amount of final product. A decrease in tonnage sliced due

to incrustated tubes obviously increases the actual campaign time. This increase in campaign time results in a corresponding increase in cost of production per pound of sugar.

However, in some beet sugar plants scale is not such a problem as in others. If the composition of the complex organic and inorganic incrustation is such that dilute acids will effectively remove or reduce the amount of scale, it is necessary to "cut out" the fouled evaporator and "boil out." This practice if effective is, however, only partially satisfactory, since even then plant operations must be curtailed so that the bodies remaining in service can effectively handle the increased volume of thin juice.

If "boiling out," using first a base, usually Na_2CO_3 or NaOH , followed by dilute HCl , (6, 23, 3, 27, 22, 5) does not take the formation into solution due to its insoluble composition, mechanical means may be employed. "Rattling" can only be resorted to after the campaign has been completed. This common mechanical method necessitates removing the tubes from each body, and grinding the incrustation from them by the use of sand and a motor-operated steel cylinder. This practice, however, is lengthy, tedious, and costly. There is also the possibility of a too long contact with the sand, and the danger of bending or breaking the tubes during "rattling."

The most important factors governing the amount of scale formation are: the nature of the substances composing the sugar beets (13, 3, 6); the accuracy of chemical and mechanical control of clarification, filtration, and sulfitation (20, 4); the chemical percentage composition of the limestone used, and its calcining temperature (12, 20, 19, 7); and the hardness of the water used throughout the plant processes. (29)

The soil in which the beet was grown influences to a great extent its inorganic and organic constituents. There can be no doubt that these various constituents tremendously affect the quantity of colloidal material present in the diffusion juices.

Inefficient chemical and mechanical control of the early stations in sugar processing also has a direct relationship not only to scale formation but also to purity increase, and quantity of colloids in the beet juice. Lack of close control in the clarification, resulting in an excess of lime and poor carbonation, is often the cause of heavy incrustations upon evaporator tubes (4).

The chemical composition of the limestone should be as high in calcium carbonate as possible, since not only will the amount of lime obtained after burning be decreased by a decrease in calcium carbonate, but foreign materials condu-

cive to scale will be introduced in the process. Calcium sucrate, containing other substances than calcium, added at the carbonation for clarification will introduce these materials in amounts proportional to their percentage in the original limestone. The calcining temperature of the limestone also influences amounts of non-desirable, foreign, scale-forming substances (20).

The hardness of the water used influences the scale formation and purity of beet sugar solutions. It is a well known fact that with increase in hardness of water there will be an increased scale formation on heating surfaces as well as a decrease in purity of juice.

Purity (coefficient of purity, per cent purity) of a beet juice, massecuite, molasses, etc., is the percentage of pure sucrose contained in the total solids (26). It is calculated by multiplying the percentage of sugar by one hundred and dividing by the percentage of solids, or by multiplying the Brix by the polariscope reading.

Since the purity is the percentage of pure sucrose in the total solids, the presence of any foreign material already present or introduced during processing will obviously decrease the percentage of sucrose and increase the amount of non-sugars present. It is therefore imperative that the non-sugars be kept at a minimum. Even a small purity rise is

desirable, since this factor influences the length of campaign, the amount of substances required in eliminating non-sugars, the volume of molasses produced for subsequent Steffen's run, the quality of the final product, scale formation, and probably several other factors. Therefore purity and purity rise are important factors to be considered in calculating costs of production.

Considerable progress has recently been made in the study of colloids in relation to sugar solutions. At least two reasons for this rapid progress have been: first, the technical difficulties experienced in sugar manufacture and the recognition of their connection with colloidal phenomena; and second, the increased knowledge of colloid-chemical principles.(4)

Colloidal substances found in beet juice may be considered in two groups: (a) those which normally occur in the beet, and (b) those which are derived from outside sources or result from factors which are preventable to a certain extent (4).

Beet juices are comprised essentially of a solution of sugars and of salts of inorganic and organic acids molecularly dispersed, together with small quantities of material of colloidal size. These colloidal materials consist mainly of substances such as fats, waxes, gums, proteins, pectins,

coloring matter, and inorganic constituents such as lime and silica. In the second group, colloids from outside sources may be derived from adhering soil or produced by the action of lime and heat upon fine particles of fiber which were not filtered out after diffusion. Colloids may also be produced by fermentation, as well as by injury to the beet from abnormal climatic conditions such as freezing. (4)

Colloidal proteins often cause serious difficulties in sugar mill operation. They are considered molasses formers, and hinder pan boiling operations. Contamination of sugar by nitrogenous substances increases greatly the susceptibility of the sugar to deterioration by microorganisms (4). It is also frequently responsible for objectionable colors.

Fats and waxes in colloidal form are usually removed almost entirely in the clarifying processes, but a small quantity remains in the emulsified state and is thus present in small amounts in all subsequent products.

Gum is generally considered to consist of various complex polysaccharides which are soluble in water but are readily flocculated by acidulated alcohol. Pectins, which are similar to gums in chemical and physical properties, are included in this group. Gums are objectionable in that they increase viscosity and hinder boiling of sugar solutions. (4)

Coloring substances of beet juices are usually regarded

as colloids. For a long time they were assumed to be essentially polymerized and dehydrated products of sugars and were called caramel, which is known to diffuse and dialyze slowly. However, other colloidal coloring substances found in beet sugar juices are far more important. Coloring materials are in some cases polyphenol compounds resembling anthocyanins. Perhaps the most important compounds which cause color and are in the colloidal state are the tannins. These give a green color with ferric salts and oxygen, and are considered to be the cause of the dark color which beet juice assumes immediately after diffusion. So far, it has not been definitely ascertained which exactly are the various coloring matters colloiddally dispersed, but it seems certain that polyphenols and their iron compounds, as well as the polymerization products of sugars, belong in this category. These coloring materials not only are undesirable in beet juices, but are considered responsible for the very dark color of molasses. (4)

Inorganic constituents are not definitely known as to their proportion in colloidal dispersion, but are believed to be molecularly dispersed. Some of the mixed compounds such as silicon compounds, calcium salts of organic acids, and organic phosphorus are probably colloiddally dispersed, since they are always found with colloids separated from sugar

liquors by dialysis or ultrafiltration. (4)

From the foregoing discussion of colloids one can see that it is of great importance to decrease to a minimum the colloidal material in beet juice. Colloids in sugar mill operation may be summarized as being objectionable since they are the cause of molasses, high viscosity, color, slow boiling, foaming or frothing in evaporators, and possibly scale formation on heating surfaces. It is not known definitely whether scaling is a direct function of colloidal material, although it is theoretically possible. Colloids which are dehydrated and flocculated most easily may become attached to the heating surfaces and eventually form a scale as a result of electrical neutralization, the colloid particles and the heating surface bearing opposite electrical charges. This theory may account for the tenacious attachment of scale to heating surfaces in a manner somewhat similar to electro-deposition of metals. Sugar factory heating surfaces (evaporator tubes) are more commonly incrustated with a scale containing a fairly high proportion of silica probably derived from organic silicon compounds of colloidal nature. (4)

As has been suggested, scale decrease or removal may be effected by one or both of two methods: (a) the addition of various chemicals, usually bases followed by acid in dilute

solutions, or (b) mechanical removal, known as "rattling." These two methods are only employed after the incrustation has formed. A third method is by chemically precipitating out constituents organic and inorganic, non-sugars and colloids present in thin juice before evaporation which affect scale formation and purity of the juice. The third means was employed in this preliminary investigation.

Very small quantities of sodium aluminate are used in the treatment of industrial waters to coagulate and adsorb the suspended or colloidal impurities, and to aid in the removal of complex silicon and magnesium compounds. Sodium aluminate removes colloidal silica from water very effectively through adsorption, and reacts chemically with soluble silicates to form the insoluble compound, sodium aluminum silicate. Sodium aluminate also has the property of accelerating the usual water softening reactions, and in many instances has been known to soften waters which, owing to the presence of complex magnesium and silicon compounds, could not be successfully treated even with large excesses of lime and soda ash. In view of these properties of sodium aluminate it was considered possible that small quantities of a juice-soluble aluminate added and allowed to react immediately before evaporation would react with the silica and magnesium of the juice to form an inorganic precipitate of high

adsorptive and clarifying power. The precipitate thus formed would help produce a clarified juice of higher purity, containing fewer lime salts and fewer of the mineral constituents responsible for scaling of evaporator tubes and other heating surfaces. (31) It was also considered possible that sodium aluminate would react with the negatively charged colloidal particles by adsorption and neutralization to decrease the number present in the resulting clarified juice.

In this investigation varying small amounts of sodium aluminate were added to the thin juice immediately before evaporation since: (a) All constituents which cause scale upon evaporator tubes are present, and any decrease in these substances at this point would obviously decrease the resulting amount of scale; (b) A decrease in colloidal material before evaporation would decrease viscosity, foaming, color, etc., of the resulting thick juice, which would result in smoother boiling in the vacuum pans as well as in a better quality white sugar product and a subsequent decrease in molasses; and (c) Any decrease of scale constituents or colloidal material would be indicated by a purity rise immediately after evaporation. The evaporation was carried out using only one vacuum evaporator, and thus all scale-forming constituents would precipitate out as scale on the tubes of

the one effect, whereas scale in actual factory operation is found on the tubes of all the effects. This simplified the calculation of amounts of scale formed per definite volume of untreated and treated thin juice.

Perhaps the greatest difficulty in arriving at any definite conclusion as to the effect of any substance added to beet sugar juice during actual campaign is the constant and uncontrollable changing of juices throughout the process which affects the reliability of results. Table 6 gives results of actual factory tests with and without sodium aluminate addition. These cannot be accepted as conclusive since uncertainty arises as to when a treated juice arrives at the station from which samples are taken for purity and lime salt determinations. In other words, the samples may not have been the treated juice, since only an estimate can be made on rate of flow from station to station. Therefore a more reliable method must be devised if conclusive results are to be obtained from juices during campaign.

However, by the method used in this research the major difficulties mentioned above were eliminated.

APPARATUS AND PROCEDURE

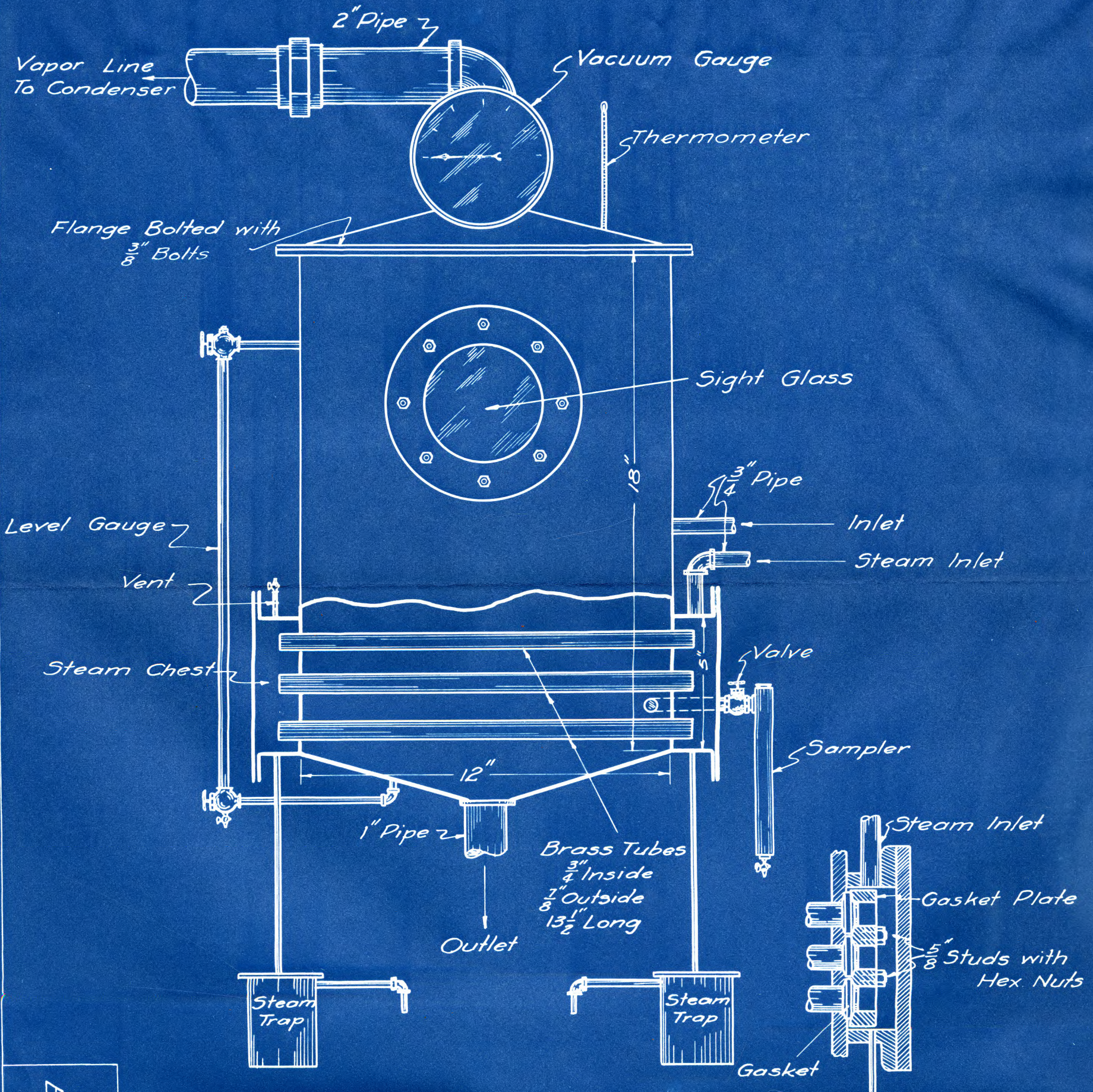
Apparatus Used

Experimental Evaporator. The evaporator used is shown on Diagram 1 and by the photograph on Plate I.

This piece of equipment consists essentially of two main parts: (a) the vacuum evaporator from which the vapors of the boiling liquid pass into (b) the low-level jet type condenser.

The evaporator consists primarily of a rectangular steel box of eighteen inches inside height and twelve inches side dimensions. The top and bottom sections are pyramidal in shape. The top section is constructed with flanges and bolts so that in case of necessity the entire top section may be easily removed by removing the bolts and breaking the union of the vapor line. The vapor line is welded into the center of the top section and leads to the cold water condenser. The top also holds a vacuum gauge and a thermometer well which extends down inside but above the liquid level. The bottom section, also pyramidal in shape, has no flange, but is welded completely around to the side edges.

Into the center of the bottom is welded the thick juice outlet line which is connected to a small centrifugal pump



CROSS-SECTION OF STEAM CHEST

DIAGRAM 1
EXPERIMENTAL VACUUM
EVAPORATOR

used in discharging concentrated liquor. Also welded into the bottom is one "arm" of the liquid level gauge, the other "arm" extending into the side approximately six inches from the top of the evaporator. The level gauge consists of a Pyrex glass tube connecting the two "arms" mentioned above. Valves are arranged on both ends of the glass so that it may be removed in case of accident during evaporation.

On the side opposite the level gauge are the "sampler" and thin juice inlet valve and line. The "sampler" is constructed so that intermittent samples may be obtained for Brix determinations during actual evaporation. In the front and also in the back are two heavy glass sight openings for observation of boiling liquid.

The heating surface consists of horizontal brass tubes, arranged in three rows of eight tubes each. These tubes have the following dimensions: inside diameter three-fourths inch, outside diameter seven-eighths inch, and length thirteen and one-half inches. Steam introduced into the front steam chest passes through all the tubes, and the condensate escapes to the sewer through steam traps connected into the bottom of both the front and back chests. The liquid within the evaporator is prevented from leaking into the steam chests by means of especially manufactured soft-rubber gaskets. Each gasket fits snugly over the outside end of each

tube, and is held into the bevel of the tube plate by heavy steel gasket plates. The plates are also beveled to correspond with the bevel angle of the gaskets.

The condenser, which is of cylindrical shape, has an outside diameter of fifteen inches and an overall height of fifty-two inches. Originally the cylinder was used as a fire extinguisher, and although larger than necessary, it served as the condenser for this apparatus very satisfactorily.

The vapor line from the evaporator is welded into the condenser side approximately 17 inches above the condenser bottom.

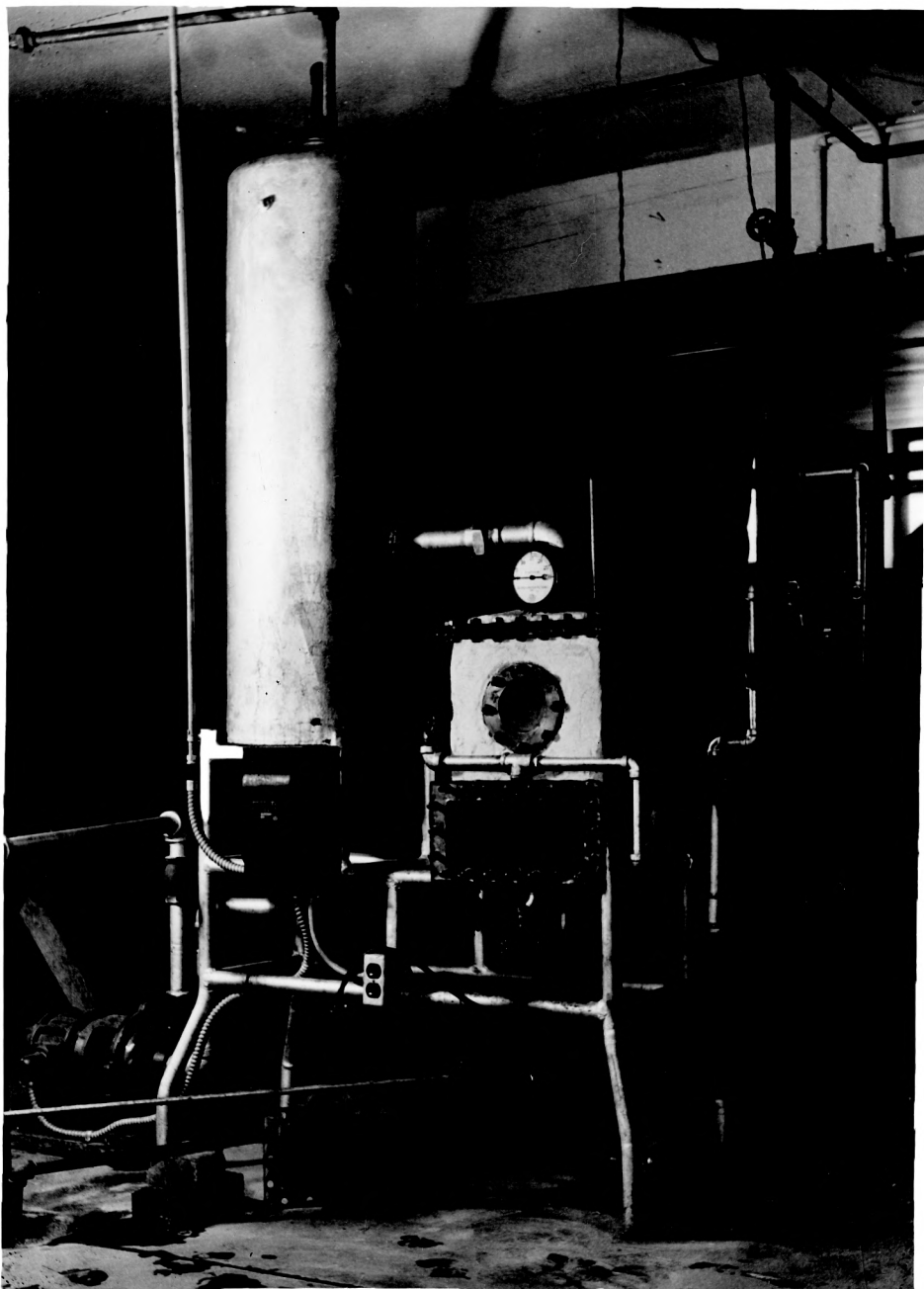
Into the condenser top are welded two pipe lines. One line is connected to the vacuum pump and the other to a cold water line. The cold water introduced at the top of the condenser must first pass through a small-holed shower-bath spray before coming into contact with the incoming hot vapor from the evaporator. This allows more uniform distribution of cold water, and better contact between vapor and liquid. Into the condenser bottom is welded a pipe which connects to a one-and-one-half-inch centrifugal pump, used for discharging vapor condensate and excess water.

Ultramicroscope and Cataphoresis Cell. Exact instructions as given by Badollet and Paine (1) were followed in

Plate I

Experimental Vacuum Evaporator with
Front Steam Chest Plate Removed

Plate I



the construction of the ultramicroscope and cataphoresis cell. Reference to their article is suggested for complete details of construction and operation for dye value determinations.

Saccharimeter. All purity determinations were made by means of a new Bausch and Lomb, water-jacketed saccharimeter.

Discussion of Method

Determinations of Brix, purity, pH, and dye value were made on all thin, untreated beet juice and thick, treated and untreated juice with the exception of the first and second samples. The procedure for each sample is described later in the text.

Brix. Brix on thin juice was determined by placing each sample in a 250 cc. graduated cylinder filled to overflowing. Filling each cylinder to overflowing reduces to a minimum the error introduced by surface films. The Brix sugar spindle, calibrated at 20° C., was inserted and the apparent Brix was taken. If the solution was not exactly 20° C., corrections for temperature were made by the use of a correction factor chart.

Brix for thick juice was determined in a similar manner. However, all thick juices were first diluted with hot water to a Brix while hot of about ten degrees. The solution was

then cooled to a temperature of approximately 20° C. If the solution was not exactly 20° C., corrections for temperature were made.

Purity. Purity was determined by using a method common in sugar house control work.

After the Brix of each sample had been recorded, a portion of the sample was poured into a convenient-sized bottle, and enough dry subacetate of lead added to decolorize the solution. The mixture was shaken well, allowed to stand for a short time, and then filtered. The clear, filtered liquid was then transferred to a 200 mm. water-jacketed tube, and the temperature of the solution adjusted to 20° C. by the circulation of cold water. At 20° C. the solution was polarized by means of a Bausch and Lomb saccharimeter. Purity was then calculated from the saccharimeter reading and the Brix, using a table by means of which the purity is derived directly from these readings.

pH Determinations. All pH determinations were made by comparison with a quinhydrone electrode.

Dye Value. Dye value or amount of colloidal material present in a sample of beet juice was determined by a modification of the original dye value method of Paine and Badollet, as used by Dawson, Keane, and Paine (9).

The principle of dye value determinations is that flocculation of colloids is accomplished by the addition of free ions with an electrical charge opposite to that of the colloidal particles, or by adding the quantity of an oppositely charged colloid required to produce mutual electrical neutralization. When a negatively charged colloid is mixed with a positively charged colloid in a certain proportion, a point of electrical neutralization is reached at which the electrical charges exactly neutralize each other, causing mutual colloid flocculation and producing a precipitate which settles out of solution. If at this isoelectric point the liquid containing the suspended flocs is examined with an ultramicroscope and cataphoresis cell, no movement of colloidal particles or flocs toward either electrode is observed when a direct current is applied. In applying the theory of electrical neutralization of colloidal particles to sugar house liquors it has been found that all colloidal particles are electro-negatively charged. Therefore, neutralization of negative colloids can be effected by the addition of varying amounts of a solution containing one gram night blue dye dissolved in freshly distilled water and made up to one liter. (1)

The modification of the original dye value method is as follows:

A 10-cc. quantity of the diluted sample at 10° Brix was further diluted with 50 cc. of distilled water, and the pH adjusted to approximately 6.5 by the addition of dilute hydrochloric acid.

pH was adjusted for four reasons:

- (a) With pH constant for all samples, comparison of dye value results are more accurate.
- (b) A high pH changes the basicity of the night blue dye.
- (c) pH of between 6 and 7 is considered the most satisfactory flocculation range.
- (d) Adjustment of pH to approximately 6.5 greatly decreases the electrolysis which takes place without adjustment. Electrolysis causes uncertainty as to when neutralization of particles has been accomplished.

This solution, contained in a large flask (250 cc.) to allow violent agitation without loss of solution, was then titrated with a night blue dye solution, containing one gram per liter, to the first indication of neutralization or a slowing down of migration of particles when examined in the ultra-microscope cataphoresis apparatus. When this point was reached, the titrated solutions were left to stand overnight to allow more complete equilibrium to become established.

Complete neutralization was then effected by the addition of a small amount of dye. The end point is recognized when there is no movement of particles in the cataphoresis cell upon application of a 250 volt direct current.

Dye value is expressed in grams of dye required to neutralize the colloids in 100,000 grams of solids in sugar solutions, and may be calculated by one of two methods: (18)

First, where a definite volume of sample is taken:

$$\text{Dye value} = \frac{\text{wt. dye required} \times 100,000}{\text{vol. of sample} \times \text{S.G.} \times \text{Brix}}$$

Second, where a definite weight of sample is taken:

$$\text{Dye value} = \frac{\text{wt. dye required} \times 100,000}{\text{wt. of sample} \times \text{Brix}}$$

Procedure on Samples

Each three-drum sample of thin juice was taken at the Garden City Sugar Co. factory from the Danek filters immediately before evaporation. The juice was allowed to flow by means of gravity into 55-gallon steel drums which had previously been cleaned by means of hot soft water, sodium carbonate, and sodium hydroxide solutions. The composite samples were preserved by the addition of one gram of toluene per gallon of thin juice. The drums, tightly sealed, were then shipped to the author for research investigation. The

samples, consisting of three 55-gallon drums, were labeled, upon arrival, Drum No. 1, No. 2, and No. 3. By this method data on each drum of each sample was more conveniently recorded.

First Sample. The first sample (three drums) consisted of thin juice with Steffen's house run. The pH, purity, and Brix on each drum were determined before evaporation. Since these determinations were very close, an average was taken and recorded. No sodium aluminate was added to this sample. On the thick, untreated juice, pH, purity, and Brix were recorded as an average of several samples.

The primary objective with this sample was to ascertain the amount of scale formed per definite volume of thin, untreated juice.

Before evaporation began, the tubes were removed and carefully weighed on a torsion balance. The tubes were then replaced and the apparatus adjusted for the "run." The entire first sample, or 150.2 gallons of thin juice, was evaporated to an average Brix of 59.9. After completion of the run the tubes were removed and carefully washed to remove adhering thick juice, dried at 105° C., and weighed. The weights (given in Table 1-a) indicate that no scale had formed. Reasons for the absence of a weighable amount of

scale formation are given under discussion of results.

An example of control data as kept on all samples during evaporation is given in Table 1-b. As will be noted from this table, one-third the content of a drum, or approximately 16 gallons, was evaporated per run. This procedure proved very satisfactory, since it required between 15 and 16 gallons of thin juice to give 2.5 gallons of thick juice, or evaporator capacity. When this volume was obtained the entire batch of thick juice was discarded and the next run started on the next one-third drum of thin juice. This procedure was followed on all samples (of three drums each) received.

Second Sample. Since no weighable scale was formed using all of the first sample, which was with Steffen's run and no sodium aluminate addition, the scale phase was disregarded in the second and third samples. Brix, purity, and pH were determined on untreated thin juice and treated thick juice of the second sample. The thin juice was treated before evaporation with increasing amounts of sodium aluminate.

The second sample (three drums) consisted of thin juice with Steffen's run. On each drum Brix, purity, and pH were determined before evaporation. These determinations were very close; therefore an average was taken and recorded. To each one-third drum (approximately) sodium aluminate was add-

ed in increasing amounts, and thoroughly mixed before evaporation.

At the completion of each batch a sample of the thick juice was taken for Brix, purity, and pH measurements. Each thick juice batch was then discarded, and the next one-third drum evaporated. Results of all determinations of the second sample are given in Table 2.

Third Sample. The third sample also consisted of thin juice with Steffen's house run. The method of procedure was exactly similar to the method used for the second sample. However, dye value determinations were also made on the untreated thin juice before evaporation, and on treated thick juice.

Results of all calculations are given in Table 3.

Fourth Sample. The fourth sample consisted of thin juice of only Steffen's house run. The objective using this sample was to combine the objectives of the first, second, and third samples. As has been stated above, the objective of the first sample was scale determination, and the objectives of the second and third samples were the determination of the effect of addition of increasing small amounts of sodium aluminate on purity and dye value.

The Brix, purity, pH, and dye value were calculated on

each of the three drums comprising the fourth sample. Since there was a variation in these determinations, not encountered in the preceding drums, the values for each drum are given in Table 4-a.

The tubes of the evaporator were removed and weighed before evaporation of the sample was begun. The tubes were replaced, and one-half of the total volume, or 75.6 gallons of the thin untreated juice, was evaporated. A sample of each thick juice batch was taken for analysis. After 75.6 gallons of untreated thin juice had been evaporated, the tubes were again removed and weighed.

The difference in weight before and after evaporation represents the amount of scale formed from untreated Steffen's house juice.

The tubes were replaced and the last half (or 76.1 gallons) of the total volume, adding increasing amounts of sodium aluminate, was evaporated. A sample of each thick juice batch was taken for Brix, purity, pH, and dye value calculations. The difference in weight of the tubes before and after evaporation of the last half of the fourth sample represents the weight of scale formed from sodium aluminate treated juice. The calculations on scale for the fourth sample are given in Table 4-b.

RESULTS

In accordance with the purposes of this investigation a series of tests were made in correlating scale, purity, and dye value data in relation to the effect of sodium aluminate added in varying amounts to thin beet juice. The results of these tests are given in the first four tables which follow.

An incomplete analysis of water used in the actual factory processes at the Garden City Sugar Company plant is given in Table 5.

Data determined during the 1935 campaign of the Garden City Sugar Company, Garden City, Kansas, are given in Table 6. These results were obtained from beet juice similar to samples used in this investigation. However, sodium aluminate was added in the troughs of the second or third hot presses. The results given in Table 6 were determined by the Garden City Sugar Company laboratory staff on actual plant tests made during the campaign.

To aid in understanding results given in Table 6, a brief outline of the beet end processes in beet sugar manufacture might be helpful. It follows:

Cosettes or chips from the beet slicers are exhausted of sucrose in the diffusion battery. The solution drawn from the battery is measured for volume in the measuring tanks.

From the measuring tanks the thin juice solution is heated by passing through the raw juice heaters. From the heaters the juice is pumped into the first carbonation tanks for defecation. The resulting carbonated juice is then pumped through the first and the second hot presses. After filtration, the partially clarified juice is pumped into the second carbonation tanks for the second defecation. The juice then is filtered through the third hot presses and sulfured at the thin juice sulfur station. After sulfitation the juice is again heated and filtered through the Daneks. The filtered juice is then concentrated to the required Brix in the evaporators and sent to the thick juice sulfur station for final sulfitation. After filtration at the thick juice presses the juice is sent to the pan floor.

Table 1-a. First Sample Analysis. Thin Juice with Steffen's House Run.

	<u>Untreated Juice</u>	
	THIN JUICE	THICK JUICE (AVERAGE)
pH	8.3	7.5
Purity	87.5	87.6
Brix	14.7	59.9

Scale Data:

Total volume untreated thin juice evaporated.....	150.2 gal.
Weight of tubes before evaporation	6,815.0 grams
Weight of tubes after evaporation.	6,814.6 "
Weight of scale formed.....	No scale

Table 1-b. First Sample Analysis. Example of Control Data During Evaporation.

Drum #1 Untreated Juice
Volume 51.8 gallons

HOUR	VACUUM (In. Hg)	TEMPERATURE (° C.)	BRIX
3.35	24	62	14.7
4.05	24	62	35.8
4.20	23	64	39.4
4.30	24	61	58.1
10.05	25	58	14.7
10.15	23	63	19.0
10.35	23	61	34.
10.45	22	65	39.
10.55	24	60	42.
11.25	24	60	60.
1.00	25	58	14.7
1.30	25	56	33.
1.45	23	62	37.
1.50	24	60	46.
2.00	23	58	51.
2.15	24	58	60.

Drum #2 Untreated Juice
Volume 50.1 gallons

2.25	24	60	14.7
2.40	24	60	25.
2.55	25	58	32.
3.05	25	61	43.
3.20	25	62	49.
3.40	25	60	61.9
3.45			14.7
4.10	25	60	31.0
4.20	25	58	42.
4.30	25	60	50.
4.35	25	60	52.4
4.45	25	61	60.0
8.40	25	60	14.7
9.15	25	60	37.
9.20	25	58	42.
9.30	25	58	50.
9.45	25	60	55.

Table 1-b (continued).

HOUR	VACUUM (In. Hg)	TEMPERATURE (°C.)	BRIX
	Drum #3	Untreated Juice	
	<u>Volume</u>	<u>49.3 gallons</u>	
10.30	25	55	14.7
10.45	25	58	22.
11.00	25	60	42.
11.20	25	60	51.
11.35	24	60	60.
2.10	24	58	14.7
2.20	24	59	22.
2.30	25	58	25.
2.45	24	60	45.
3.00	24	60	47.
3.10	25	59	55.
3.15	25	60	60.
3.30	24	58	14.7
3.45	25	61	25.
4.15	24	60	50.
4.35	25	60	60.

Total volume of untreated thin juice with Steffen's run, 150.2 gallons.

Table 2. Second Sample Analysis. Thin Juice with Steffen's House Run.

<u>Thin Juice before Evaporation,</u>	<u>"Untreated"</u>
BRIX	PURITY
13.8	87.7
	pH
	8.16

Table 2 (continued).

DRUM NO.	<u>Treated Juice</u>			THICK JUICE		
	Lbs. $\text{Na}_2\text{Al}_2\text{O}_4$ Added per 1000 gallons	Gallons Evaporated	Brix	Purity	Purity Rise	pH
1	.0	16.55	58.2	87.7	0	7.50
1	.2	15.7	62	87.6	-.1	7.67
1	.3	14.8	60	87.8	.1	7.84
2	.4	14.8	60	88.0	.3	7.84
2	.5	15.7	62	87.9	.2	7.84
2	.6	15.6	60	87.9	.2	7.84
3	.7	15.6	62	88.0	.3	7.67
3	.8	16.5	60	88.0	.3	7.75
3	.9	14.0	63	88.1	.4	7.75

Table 3. Third Sample Analysis. Thin Juice with Steffen's House Run.

<u>Thin Juice before Evaporation,</u>			<u>"Untreated"</u>
BRIX	PURITY	pH	DYE VALUE
12.3	87.6	8.38	645

DRUM NO.	<u>Treated Juice</u>			THICK JUICE			
	Lbs. $\text{Na}_2\text{Al}_2\text{O}_4$ Added per 1000 gallons	Gallons Evaporated	Brix	Purity	Purity Rise	pH	Dye Value
1	0.	15.6	62	88	.4	8.11	625
1	.2	16.6	64	88.4	.8	8.25	675
1	.4	15.4	64	87.5	-.1	8.33	577
2	.6	16.4	64	88.6	1.	8.19	673
2	.8	15.6	64	89.0	1.4	8.25	577
2	1.	16.5	55	88.7	1.1	8.19	585
3	.4 *	16.4	64	88.6	1.	8.19	585
3	.4 *	14.9	58	88.5	.9	8.19	590

No run on last one-third of drum No. 3 because of leakage.

* Drum No. 3 used as a check on Drum No. 1.

Table 4-a. Fourth Sample Analysis. Thin Juice with Only Steffen's Run.

Thin Juice before Evaporation, "Untreated"

DRUM NUMBER	BRIX	PURITY	pH	DYE VALUE
1	11.5	90.4	8.75	817
2	11.5	91.5	8.80	669
3	11.5	89.9	8.70	605

First Half of Total Volume, "Untreated"

DRUM NO.	THIN JUICE			THICK JUICE			
	Lbs. $\text{Na}_2\text{Al}_2\text{O}_4$ Added per 1000 gallons	Gallons Evaporated	Brix	Purity	Purity Rise	pH	Dye Value
1	0	18.	54.3	91.2	.8	8.25	770
1	0	16.4	56.2	91.1	.7	8.15	770
1	0	16.5	54.3	90.3	-.1	8.15	669
2	0	16.5	60.0	91.8	.3	7.90	621
2	0	8.2	60.0	No sample taken			

Second Half of Total Volume, "Treated"

DRUM NO.	THIN JUICE			THICK JUICE			
	Lbs. $\text{Na}_2\text{Al}_2\text{O}_4$ Added per 1000 gallons	Gallons Evaporated	Brix	Purity	Purity Rise	pH	Dye Value
2	.4	14.8	60.0	92.7	1.2	7.85	528
2	.4	12.0	59.0	No sample taken			
3	.6	16.4	54.3	90.0	.1	7.95	577
3	.8	17.3	58.2	90.3	.4	8.0	480
3	1.	15.6	56.2	90.5	.6	8.12	480

Table 4-b. Fourth Sample Analysis. Scale Data on Thin Juice with Only Steffen's House Run.

<u>Untreated Juice</u>	
Total volume of thin juice evaporated	1½ drums or 75.6 gal.
Weight of tubes before evaporation	6,812.8 grams
Weight of tubes after 1½ drums evaporated	6,814.9 "
Weight of scale formed	2.1 "
<u>Treated Juice</u>	
Total volume of thin juice evaporated	1½ drums or 76.1 gal.
Weight of tubes before evaporation of second half volume	6,814.9 grams
Weight of tubes after evaporation	6,815.1 "
Weight of scale formed	.2 "
Weight of scale formed by evaporation of untreated juice	2.1 grams
Weight of scale formed by evaporation of treated juice	.2 "
Difference	1.9 "

Table 5. Analysis of Samples of Garden City Sugar Co. Process Water in Milligrams Calcium Carbonate per Liter.

	TEMPORARY HARDNESS	RESIDUAL ALKALINITY	TOTAL ALKALINITY
Hard Water	394	83	477
Water before Softener	198	88	286
Water after Softener	3	55	58

Table 6. Actual Factory Data of the Garden City Sugar Co. during the 1935 Campaign.

UNTREATED THIN JUICE				TREATED THIN JUICE $\frac{1}{2}\# \text{Na}_2\text{Al}_2\text{O}_4 / 1000 \text{ gal. Juice}$			
Test No.	Sample No.	Purity	Purity Rise	Test No.	Sample No.	Purity	Purity Rise
1	I	87.5		4	I	86.6	
	II	88.0			II	88.2	
	III	87.9	.4		III	88.4	1.8
2	I	88.6		5	I	89.0	
	II	88.8			II	88.4	
	III	89.1	.5		III	89.3	.3
3	I	88.4		6	I	87.0	
	II	88.6			II	87.4	
	III	88.6	.2		III	87.8	.8

Untreated Juice:

Sample I taken at the second presses, II at the third presses, and III at the Daneks before evaporation.

Treated Juice:

Sample I taken at the second presses before $\text{Na}_2\text{Al}_2\text{O}_4$ added in troughs, II at the third presses, and III at the Daneks before evaporation.

UNTREATED THIN JUICE					TREATED THIN JUICE $\frac{1}{2}\# \text{Na}_2\text{Al}_2\text{O}_4 / 1000 \text{ gals. Juice}$				
Test No.	Sample No.	Purity	Lime Salts	Purity Rise	Test No.	Sample No.	Purity	Lime Salts	Purity Rise
1	I	88.4	.034		2	I	88.3	.027	
	II	89.0	.017			II	88.9	.013	
	III	89.3	.010	.9		III	88.9	.011	.6
					3	I	90.0	.026	
				II		90.0	.018		
				III		90.5	.012	.5	

Untreated Juice:

Sample I taken at the first presses, II at the third presses, and III at the Daneks before evaporation.

Treated Juice:

Sample I taken at the third presses before $\text{Na}_2\text{Al}_2\text{O}_4$ added in troughs, II at the thin juice sulfur station, and III at the Daneks before evaporation.

DISCUSSION OF RESULTS

A thorough study of any one of the three major points outlined previously, namely, the effect of sodium aluminate on scale, purity, and colloidal material, would necessitate the making of far more runs on samples than were possible in this preliminary investigation. However, from the data obtained a correlation of the effect of sodium aluminate addition upon scale, purity, and colloidal material of beet juice may be made.

The Effect on Scale

First Sample. The first sample, consisting of thin juice with Steffen's house run, would be expected to form at least a small percentage of total scale, as is actually the case during the campaign of beet sugar mills. However, since there is only a comparatively small volume of Steffen's house juice introduced during the slicing campaign, in comparison with the volume of juice drawn from the diffusion cells, it is to be expected that the scale formed during this rather long period will not nearly equal the amount formed during the much shorter Steffen's campaign. In other words, it is known that the greater percentage of scale does form during Steffen's campaign.

Therefore, with the use of the entire first sample it was thought that a small but weighable amount of scale would be detected. However, no weighable amount of scale was obtained, for the following reasons:

- (a) Corrosion of brass tubes by ammonia liberated from compounds in the juice.
- (b) Lack of a large enough volume of thin juice available for evaporation.
- (c) Lack of sensitivity of the torsion balance in detecting very small differences on large weights (within limits of error).

Fourth Sample. For the above reasons no attempt was made to obtain scale data on the second and third samples, consisting of thin juice with Steffen's run.

The fourth sample, consisting of thin juice of only Steffen's house run, was expected to form a weighable amount of scale, since the greater portion of scale is known to come after slicing and during Steffen's campaign. Comparatively little difficulty was experienced during the 1935 slicing campaign at the Garden City Sugar Company plant, but after Steffen's campaign a very heavy incrustation was found on the tubes of the final evaporator.

To determine the effect of additions of sodium aluminate on scale, the first half volume of the fourth sample was

concentrated without treatment, and the second half volume with treated thin juice.

Results shown in Table 4-b indicate that sodium aluminate reacts with scale-forming constituents, either organic or inorganic, to precipitate them before evaporation, or reacts to form soluble compounds which are not precipitated as scale during concentration. Little is known as to the exact mechanism of reaction; therefore further study on this phase is desirable.

The Effect on Purity

Second and Third Samples. From Tables 2 and 3 it is noted that there is practically a linear increase in purity with increased addition of sodium aluminate. Since purity depends on quantity of sucrose in relation to non-sugars, it is logical to conclude that sodium aluminate reacts with these non-sugars to decrease the amount present in the resulting thick juice after evaporation. Since no analysis of the precipitate was made, no definite reaction products are known. The data obtained, however, indicate that sodium aluminate has a desirable effect on the purity of thin beet juice. As has been stated, sodium aluminate probably reacts with at least the silica and magnesium of the juice, and thereby produces a juice containing fewer of the lime salts

and other mineral constituents which if present would tend to form scale and decrease purity.

There are probably several important factors which influence the reaction of sodium aluminate and account for variations in purity rise, such as:

- (a) Time necessary for reaction.
- (b) Thoroughness of mixing of sodium aluminate with thin juice.
- (c) Temperature influence on the speed of the reaction.
- (d) Concentrations of the reactants.
- (e) Varying concentrations of ammonia in thin juice.
- (f) Varying volumes of CO_2 and SO_2 introduced during actual processing.
- (g) Effect of boiling on aluminate addition.

Since the mechanism of the reaction, and the resulting reaction products are not definitely known, it seems desirable, but beyond the scope of this work, to investigate further the factors mentioned above.

Fourth Sample. From Table 4-a it is noted that the purity of the three drums comprising the fourth sample varies. This may be accounted for by assuming that varying amounts of impurities remained in the drums before filling, because of insufficient cleaning.

The effect of sodium aluminate on Steffen's house juice

is not so marked as was expected; nevertheless, a purity rise is indicated as aluminate is increased. Steffen's house juice is very complex, and its composition during actual processing changes very rapidly.

It is not understood why the purity of this sample is higher than the purity of the thin juice with Steffen's obtained during the slicing campaign. It may, however, be due to the presence of unknown complex substances in the solution. These substances might influence the polarization of the solution, thereby increasing the apparent purity, although the juice actually may have contained less sucrose. This seems to be the only explanation, since it is definitely known that Steffen's juice does cause the greater per cent of scale formed. Deposition of scale and decrease in purity would be expected, since hard water was used in the Steffen's campaign. (See Table 5 for analysis.) The discussion under Second and Third Samples may well be applied to the Fourth Sample or Steffen's house run.

The Effect on Colloids

Since several substances are known to be in colloidal form in beet juice, and since these substances are negatively charged, it is reasonable to expect a decrease in colloidal content by the addition of an oppositely charged aluminate

solution. However, a decrease in colloidal content may be due to either adsorption or electrical neutralization, or both.

Inorganic constituents may react with the added aluminate to form insoluble reaction products which precipitate from solution, adsorbing colloidal organic substances as they settle out.

Organic and inorganic colloidal particles may be electrically neutralized by the positive action of sodium aluminate in solution, and thus precipitate out. The Hardy-Schulze rule for precipitation by electrical neutralization states:

(a) Precipitation occurs only by the adsorption of an exact amount of oppositely charged ions, and

(b) The precipitating value of an ion having a charge opposite to that of the suspended particles depends upon its valence, and the precipitating efficiency increases with valence.

The rate of addition and amount of stirring also greatly influence the resulting flocculation.

Regardless of the mechanism of the reaction, it is evident that sodium aluminate affects the amount of colloids in beet solutions. Although this investigation did not establish that there is a constant linear decrease in colloidal

content with increasing amounts of aluminate, Tables 3 and 4-a indicate that this may be expected in subsequent studies.

SUMMARY AND CONCLUSIONS

A small vacuum evaporator and ultramicroscope-cataphoresis cell were constructed for semi-plant determinations of the effect of sodium aluminate on scale, purity, and colloids.

It was found that very small, unweighable quantities of scale were formed from slicing campaign beet juice. With the use of only Steffen's house juice, scale was decreased by sodium aluminate treatment.

Although the effect is more pronounced when original purities are comparatively low, results indicate that sodium aluminate increases purity and decreases colloids.

To determine definitely the exact effect of sodium aluminate treatment much more research is necessary, since several factors mentioned in this investigation are entirely or almost entirely unknown.

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