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Final Report

CONTINUOUS DIFFUSION OF SUGAR BEETS

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TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

ABSTRACT

A continuous counter-current diffuser was constructed in order to study the extraction of beet sugar. A supply of California and Michigan beets was acquired for experimental use.

A series of experiments was made to determine the optimum operating temperature and diffusion water rate of the continuous diffuser. On the basis of apparent purity of the diffusion juice and recovery of beet sugar, the optimum conditions were chosen to be 75°C and 1.0 pound water per pound beet.

The continuous process employing fresh cossettes was compared to one employing chemically pretreated cossettes in a series of comparative experiments. In a series of six tests, cossettes were treated with both calcium oxide and aluminum sulfate in dosages of 0.05, 0.10, and 0.15% by weight of beet. The normal continuous process compared favorably to the calcium oxide pretreatment process in regard to the apparent purity, color, and content of impurities of diffusion juice, draft, and diffuser throughput. The aluminum sulfate pretreatment process was considered to be of value in view of the better color and less impurities in the diffusion juice.

The batch-wise diffusion battery process was compared with the continuous process at the same operating conditions, namely, 75°C and 1.0 pound water per pound beet. The experimental results were not conclusive enough without an economic study.

OBJECTIVE

A study was made of the extraction of beet sugar by means of a continuous counter-current diffuser. It was also desired to make a preliminary investigation on the effects of pretreatment of beet cossettes with reagents to determine the acidity or alkalinity on either side of neutrality.

I. INTRODUCTION

The diffusion of a solute through a porous solid has in the past been given considerable attention due to its important application in industries, such as the extractions of vegetable oils, beet sugar, and tannin. As a basic research in this connection, Boucher and Brier* studied the extraction of soybean oil from uniformly porous clay by use of perchloro ethylene. With the data obtained by a semi-batch method which they developed, the extraction process was found to be one of pure molecular diffusion. The diffusion coefficients were substantially constant for any one temperature and system, independent of liquid concentration. Further work was done by King, Katz, and Brier** on the solvent extraction of soybean flakes. Although the application of the simple diffusion theory to the extraction of oil from a uniform porous inorganic solid was confirmed, it does not apply to the extraction data for the soybean flakes. The structure of the soybean flakes was considered to be the cause of divergence from the theory. The semi-batch extraction method, however, proved to be a very economic but efficient way to establish the instantaneous diffusion rate data under prescribed conditions.

In the extraction of beet sugar, accordingly, divergence from the simple molecular diffusion theory could also be expected in view of the non-uniform structure of beet cells and the exposure of beet juice to solvent by mechanical rupture of the slicing process. A basic study of any beet sugar process should undoubtedly be made by the semi-batch method employed in previous investigations described above. Thus, the process can be evaluated on the basis of instantaneous diffusion rate. In the continuous diffusion process, the validity of continuous data can also be examined by the semi-batch data.

To comply with Contract No. A-1s-33464 of the United States Department of Agriculture, however, it was necessary to study the effects of temperature and diffusion water rate on the apparent purity of diffusion juice,

*D. F. Boucher, J. C. Brier, and J. C. Osburn, Trans. A.I.Ch.E., 38, 5, 967-993 (1942).

**C. O. King, D. L. Katz, and J. C. Brier, Trans. A.I.Ch.E., 40, 5, 533-556 (1944).

draft, and recovery of sugar in a continuous counter-current diffuser. Amount of impurities in the diffusion juice was to be observed in the comparative study between methods: the continuous, the continuous pretreatment, and the batch methods. Calcium oxide and aluminum sulfate were to be employed in the continuous chemical-pretreatment process. Experimental results in this respect are therefore included in this report.

In the meantime, the primary interest of the authors—to study the diffusion process of beet sugar with semi-batch technique—was made possible by financial support of the Farmers and Manufacturers Beet Sugar Association. Experimental work in this report was extended to establish the instantaneous diffusion rate of beet sugar by the semi-batch method, and the correlation between the continuous and differential rate data. Results of this work will be reported at a later date.

II. CONTINUOUS COUNTER-CURRENT DIFFUSER

A. DESIGN AND LAYOUT OF EQUIPMENT

The continuous counter-current diffuser was designed on the principles that it would be similar in general features to commercial units and reasonably simple in construction. Furthermore, it would permit sampling along its length in order that point-to-point data could be obtained. A survey on the various types of continuous diffusers showed that a majority of these designs employ the principle of screw conveyor to handle the transport of solid through the solvent. These designs usually provide for the possibility of lengthened time of detention for a small part of the solid in transport. In the case of sugar-beet diffusion, this causes the bacterial attack on the sugar juice as well as the equipment. To provide a positive movement for the cossettes through the diffuser, therefore, the flight conveyor design was adopted. It was also decided to use brass as the construction material to minimize possible occurrence of corrosion.

The diffusion tube, as shown in Figures 1 and 2, is in the shape of a U-tube. Since it is rather difficult to fabricate a bend of uniform circular cross section, the diffusion tube was made square in its cross section, 1-1/2 x 1-1/2 inches. It was fabricated from 1/32-inch brass sheet in two straight and one U-bend sections. These were flanged and bolted together. A hopper was mounted on the right-hand end of the tube, for charging purposes. A discharge plate was attached to the left end.

The diffusion tube is surrounded by sections of hot water jacket. These sections were made so as to allow six openings of sampling lines along

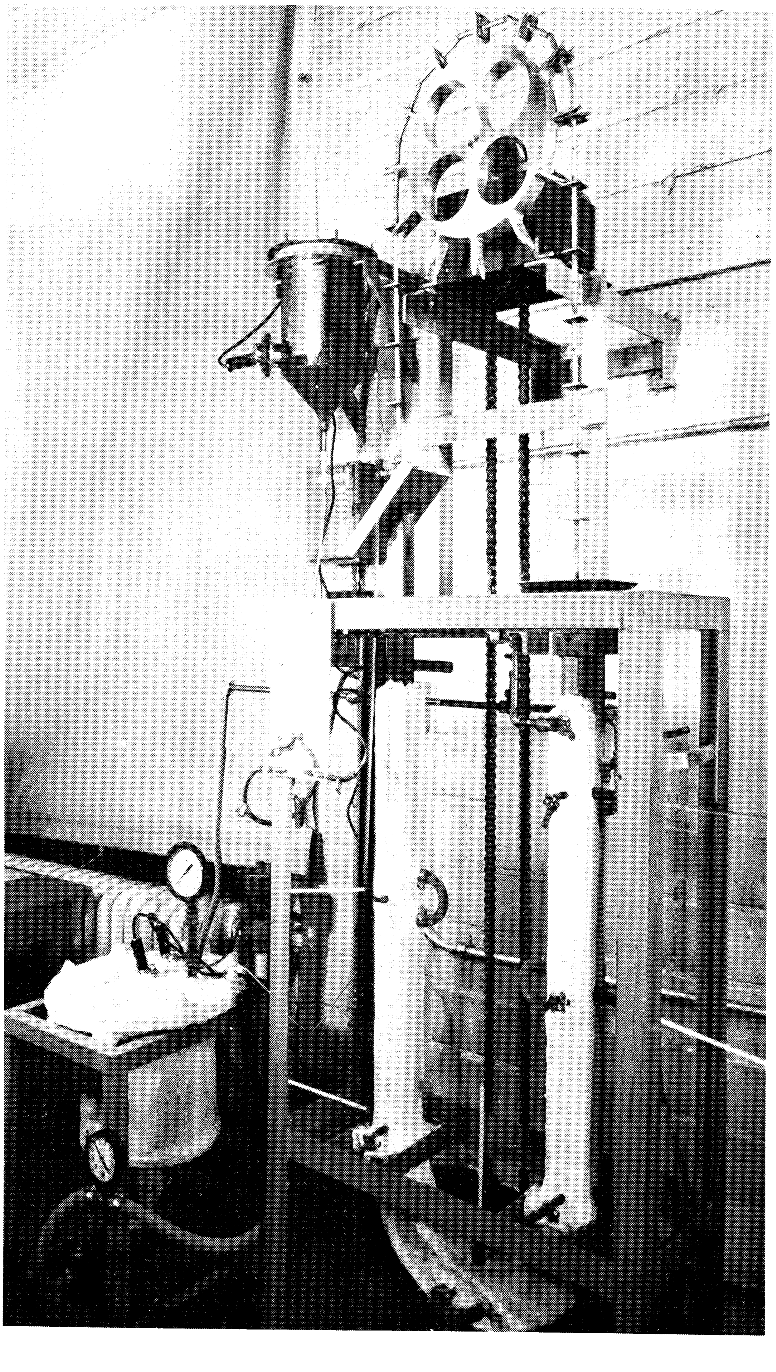


Figure 1. Assembly of continuous-diffusion equipment

the diffusion tube. One-half-inch heavy-duty rubber tubes connect these sections from one to another to complete the jacket water circuit.

The diffusion water inlet is located on the upper left section of the diffusion tube, while the diffusion juice outlet is on the upper right section. Seven 1/4-inch connections were made to the diffusion tube for insertion of thermometers. All sampling lines, except the one at the bottom of the U-tube, are made of 1/8-inch brass tubing, about 1-3/4-inch in length. They are connected by rubber tubes and closed up with clamps. The bottom sampling line is a 1/2-inch connection which serves also as a drain.

The whole diffuser assembly was insulated to reduce heat loss.

The design of the drag chain conveyor is shown in Figure 3. It was fabricated from brass with a screen plate attached to a 9/32-inch bar. The effective length of each unit is 4 inches. The screen plate is 1-13/32-inches square in its size, providing a total clearance of not more than 1/32-inch to the diffusion tube wall. On each screen plate there are 32 evenly spaced 1/16-inch openings to permit the flow of diffusion water. The whole conveyor consists of 45 chain units. For the purpose of operational convenience, the chain units were numbered in the order of entering the diffusion tube. The number was engraved on the brass bar and can be easily identified.

A gear motor, an endless roller bushed chain, a reducing-gear set, and a chain wheel were installed to drive the drag chain conveyor. The gear motor is hidden by the diffusion-tube bend in Figure 1 and cannot be seen. It has a 3/4-HP motor and a 36 to 1 gear set. The gear speed is 48 rpm. The reducing gear is a worm-gear set made by the Boston Gear Works. It was placed above the extraction apparatus together with the chain wheel. The chain wheel sprocket was fabricated from aluminum, and its design is given in Figure 4. It carries the drag chain conveyor in clockwise direction during operation.

Other accessory equipment are the diffusion water tank and the jacket water tank. As can be seen in Figure 1, the diffusion water tank was placed to the left above the diffuser, and the latter to the lower left. Both tanks are equipped with electric heaters.

An inverted U-tube manometer was made to regulate the diffusion water. The manometer was placed to the upper left of the bench structure, as can be seen in Figure 1.

A motor-driven centrifugal pump is available to circulate water from the jacket water tank to the diffuser jackets.

A flow diagram of the continuous counter-current extraction is given in Figure 5. Cossettes of known quantities are charged between the screen

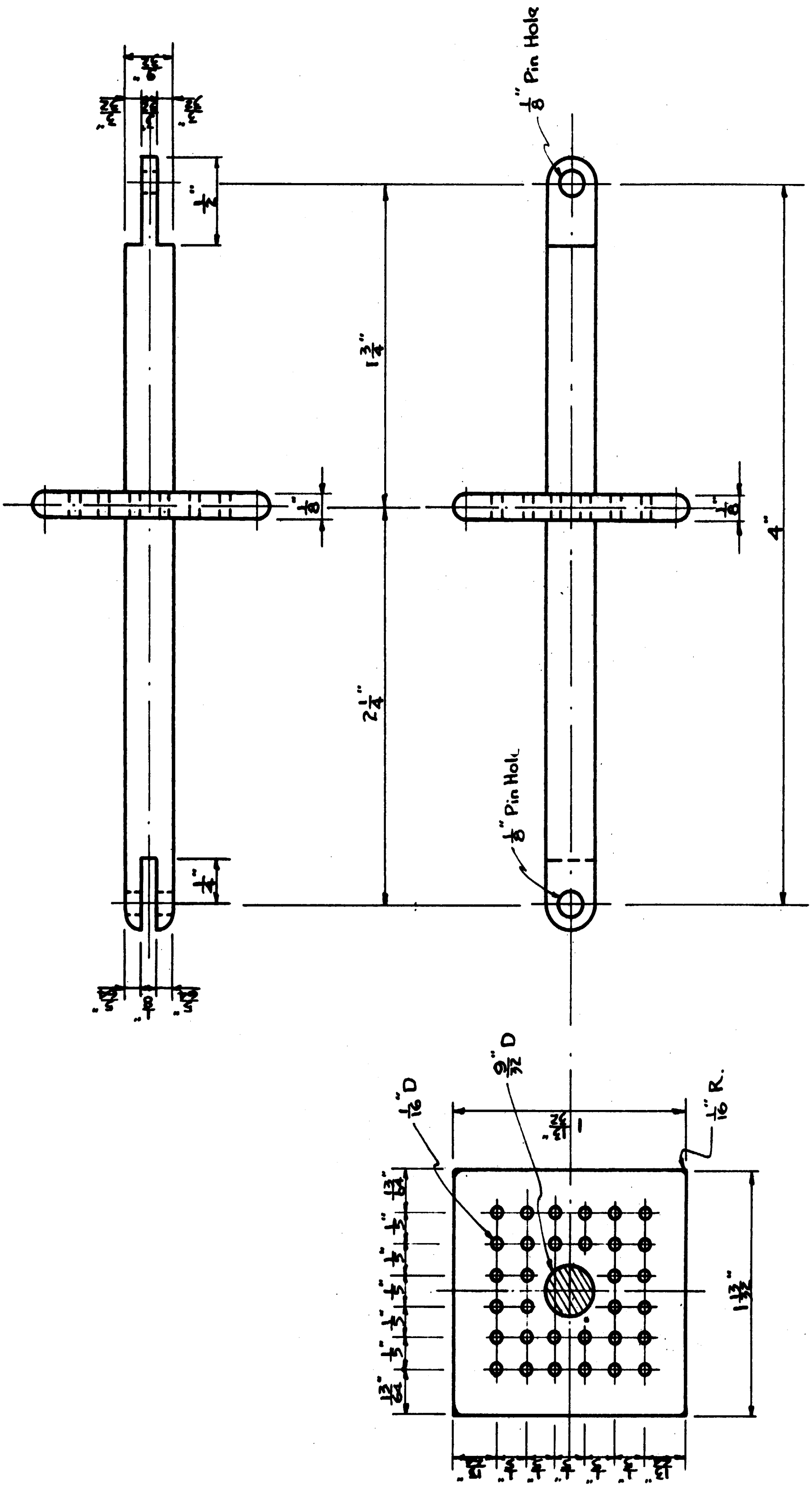


Figure 3. Conveyor unit

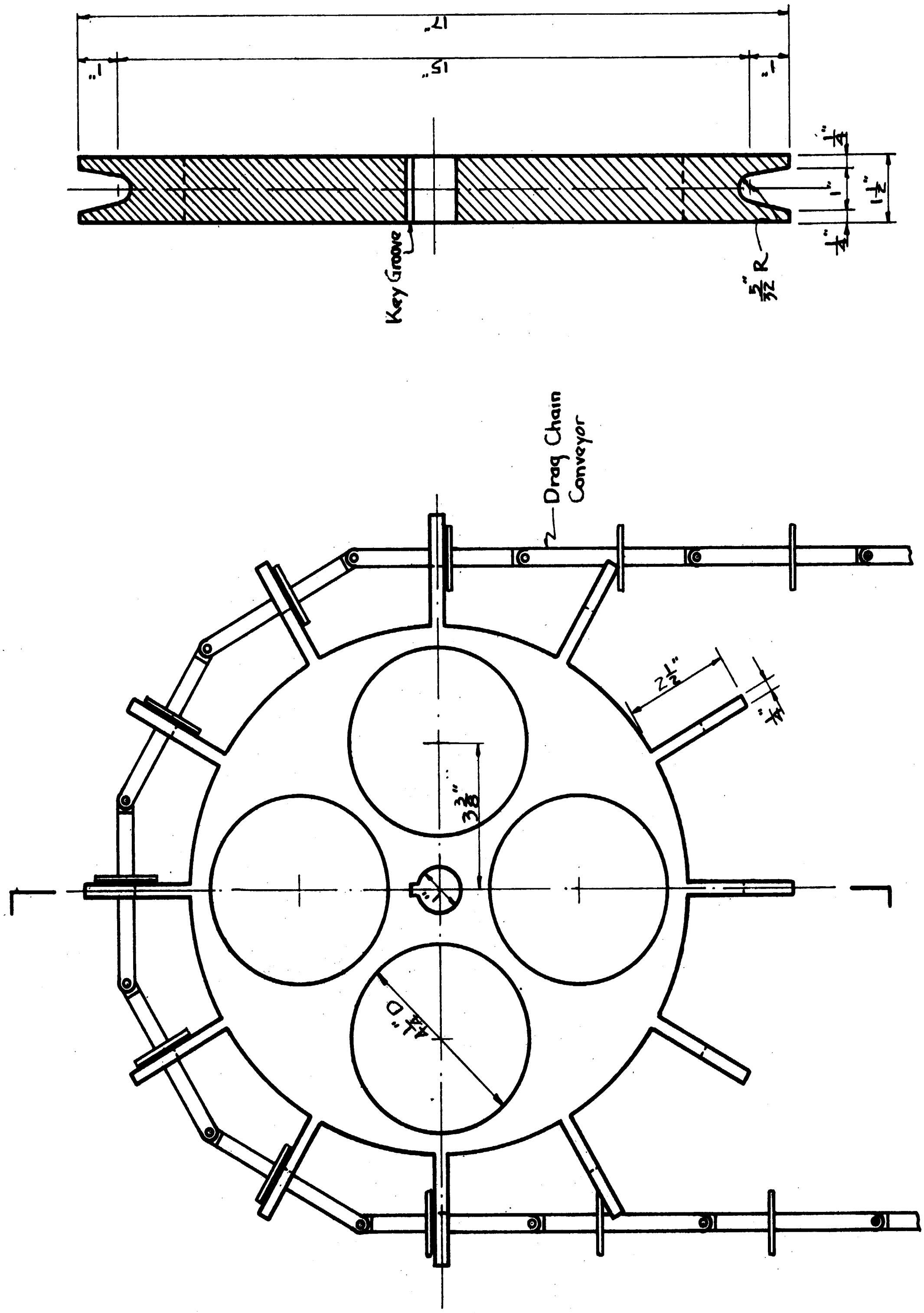


Figure 4. Chain wheel

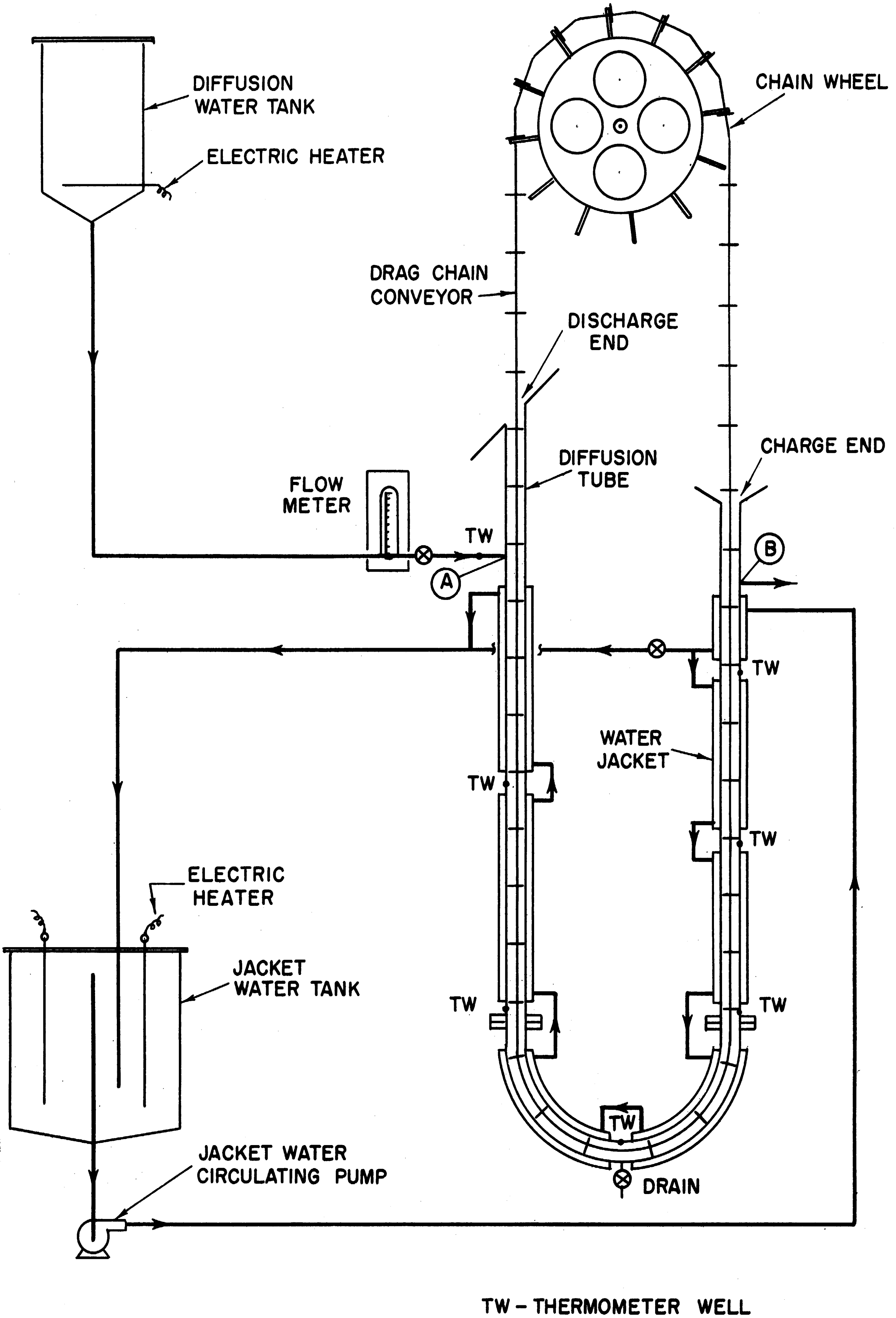


Figure 5. Flow diagram

plates of the drag chain conveyor through the charge hopper. The cossettes, carried by the conveyor, move along toward the discharge end, where the exhausted pulp is discharged. The diffusion water enters the diffuser at A. The flow meter allows adjustment of the required rate of diffusion water. The diffusion juice is collected from the diffuser at B. The jacket water pump withdraws water from the bottom of the jacket water tank and delivers it to the upper right jacket of the diffuser. A part of the water leaving this jacket is allowed to pass through the remaining 6 jackets. The rest of the water is returned directly to the jacket water tank. This is done to reduce the discharge pressure of the circulating pump and, in the meantime, bring the entering cossettes and the surrounding juice to the required diffusion temperature in a very short time.

A summary of the design data of the diffuser is given below.

1. Effective length of diffusion tube expected = 89.26 inches

2. Cross-sectional area of tube = 2.068 square inches

3. Volume of each conveyor "cell" unit = 0.616 cubic inches

4. Effective volume of diffusion tube = 166.46 cubic inches
   = 2.73 liters

5. Speed of conveyor chain = 1.494 in./min

6. Time of diffusion = 59.7 min

7. Assumed filling = 55%

8. Capacity of diffuser = 1.614 kg/hr = 3.56 lb/hr

B. PREPARATION OF COSSETTES

In order to work out the operating details of the equipment and establish satisfactory technique, The Spreckels Sugar Company, San Francisco, California, supplied five hundred pounds of sugar beets. The California beets, packed in moist saw dust, arrived in excellent condition.

For the major portion of the work, Michigan beets were used. About a ton and a half were supplied for the project by the Michigan Sugar Company through the efforts and cooperation of the Farmers and Manufacturers Beet Sugar Association. The beets were stored in a cold storage room at the University of Michigan Food Service.

To complete one experiment on continuous diffusion, from 20 to 25 beets were usually required. These were assorted from one large lot. The abnormal beets or those damaged or unhealthy because of prolonged storage were discarded. The sample of beets was washed with water at about 40°C to remove adherent mud and sand and rinsed with cold water. The washed beets were then kept in a refrigerator at 0°C. As a general rule, washing was usually done 10 hours before the experiment.

On the day of experiment, the beets were first cut to the same length of 6 inches by cutting off the crowns and tails. Each beet was placed in the beet cutter (Progress Report 4, page 4) and a number of incisions were made in the direction perpendicular to the long axis of the beet and 1 inch apart. A small portion at the bottom of the beet was left uncut so that after removal from the cutter, the slices would be held together by the uncut portion. The sliced beets were then fed into the slicer (Progress Report 4) and cut into V-shaped cossettes.

When the required amount was prepared, the cossettes were transferred to the cossettes mixer (Progress Report 4, page 5) and mixed for 5 minutes to obtain a uniform average sample. About 1 pound of cossettes was usually taken for the determination of sugar in cossettes. This sample was kept in a jar, tightly stoppered, and left in the refrigerator. The remaining cossettes were used for the diffusion experiment.

In the meantime, the slicing equipment was thoroughly cleaned with water, steam, and compressed air.

The slicer knife used had 46 divisions in the 165-mm length. The knife setting was the same in all experiments. The cossettes had a total length of 22.402 meters 100 grams on two determinations.

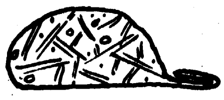
C. PREPARATION OF CHARGE STOCK

As shown in the summary of diffuser design data, a filling of 55% was assumed for the experimental purpose. Consequently, a charge stock of 61 grams of cossettes was required for each conveyor "cell" unit. The lots of cossettes were weighed out immediately after slicing and mixing.

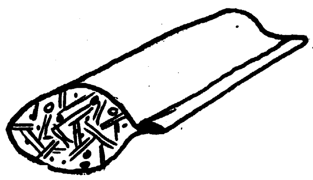
In order to minimize the evaporation and oxidation of cossettes, each lot of charge stock was wrapped with a sheet of aluminum foil. The aluminum foil was 0.0065 in gauge and 8 x 12 inches in size. The wrapping was done in the manner shown in Figure 6.

The wrapped lots of cossettes were put in the refrigerator for later use.

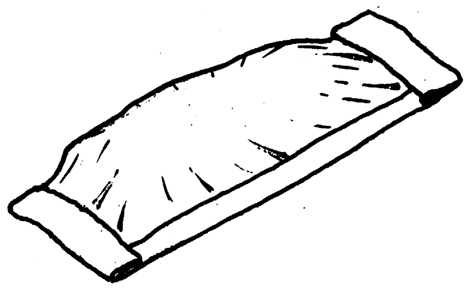
(a) Weigh cossetts on a sheet of aluminum foil



(b) Fold the long edge



(c) Fold again



(d) Double-fold both ends

Figure 6. Preparation of diffuser charge stock

D. PREPARATION OF CHARGE STOCK FOR COMPARATIVE EXPERIMENTS

Since a complete experiment took on the average 20 man-hours to accomplish, the comparative experiment and its accompanying control run were always made on two consecutive days. A slightly different procedure for the preparation of charge stock for these experiments was followed. This was to assure that charge stocks of approximately equal properties were employed in two experiments for comparison.

Referring to Figure 7, each clean beet was first cut into two halves along its long axis. The two parts were approximately symmetrical and of about the same weight. The exposed surfaces were even and smooth.

One half of each beet was then wrapped up in aluminum foil in such a way that the exposed surface was sealed as completely as possible. The wrapping was done to minimize the evaporation and oxidation of the juice present in the open cells. These halves were stored at 0°C for use on the next day.

The remaining halves were cut to the length of 6 inches by cutting off the crowns and tails. They were again sliced in the direction perpendicular to the long axes of the beets. The bottom parts of the beets were left uncut to keep the slices together.

After the incisions had been made, the beets were transferred to the slicer flat side down and sliced into cossettes.

The steps of mixing, weighing, and wrapping of charge stocks were the same as those previously described in Section C.

E. OPERATING PROCEDURES

1. The Continuous Diffusion Test.—The operation of the continuous diffuser requires the services of two men, one being responsible for the charging of cossettes and discharging of pulp, and the other on the control of diffusion water rate and temperatures, collecting samples of juice and pulp, and making Brix determinations during the progress of the experiment.

Three hours before the experiment was begun, the diffusion water and jacket water tanks were filled with water and the electric heaters were put into use. The diffusion water was introduced into the diffuser at predetermined rate. In the meantime, the jacket water pump was started to circulate the jacket water. By the time the cossettes were prepared, the temperature of water in the diffuser should have almost reached the desired level.

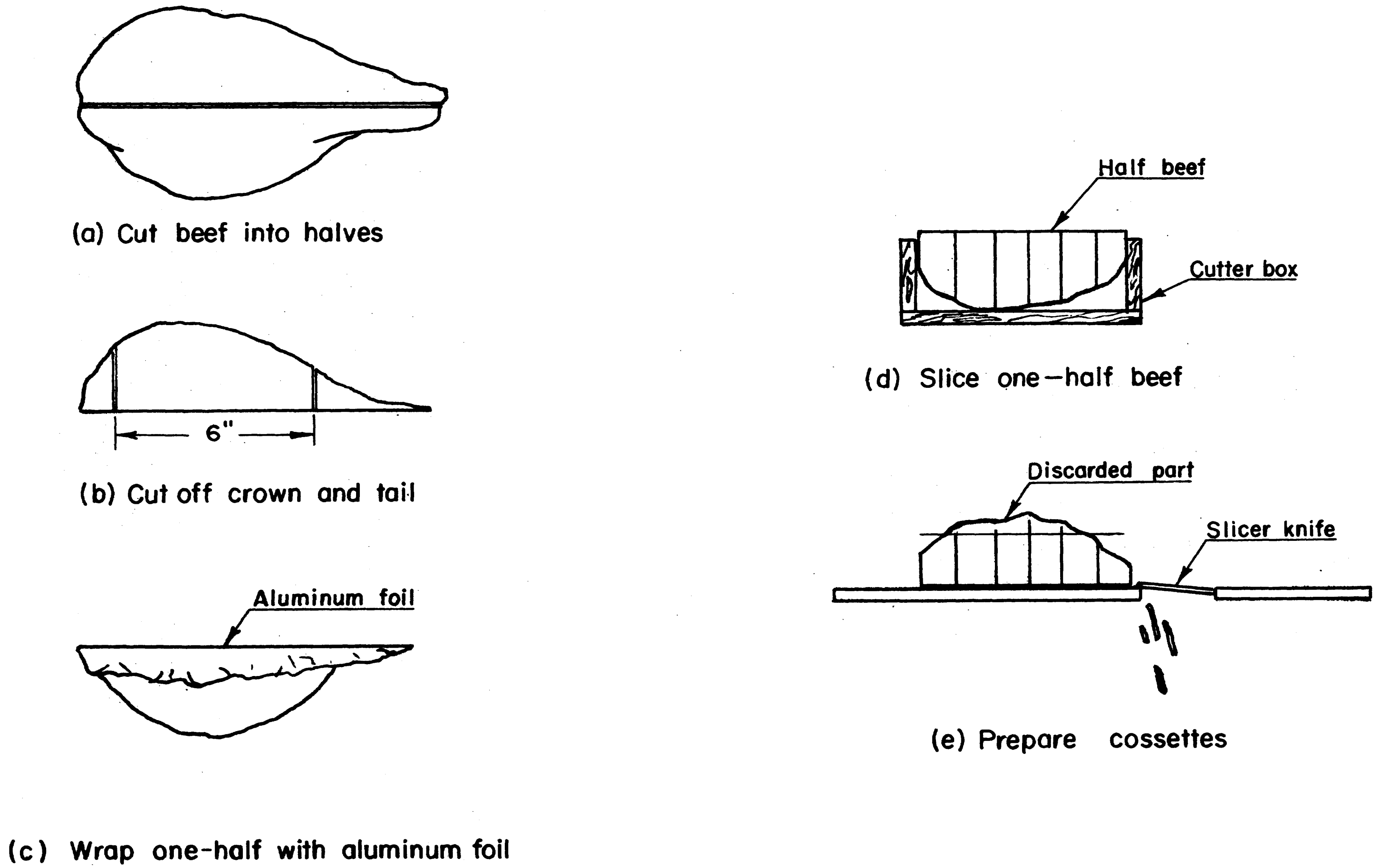


Figure 7. Preparation of cossettes for comparative experiments

The experiment was started when this temperature appeared to be constant for a period of 15 minutes.

The drag chain was started and charging of cossettes begun. The aluminum-foil-wrapped charges of 61 grams were opened and fed to the conveyor section traveling at a rate of 1.494 inches per minute. This allowed almost 2.7 minutes for charging of fresh cossettes and removal of spent ones. Thus, ample time was provided and exposure of cossettes to atmospheric conditions for prolonged periods was avoided.

Whenever possible, the experiment was always begun with conveyor unit No. 1. This facilitated the counting of the total number of lots of cossettes charged.

The diffusion juice generally had a temperature of 55-67°C. The average diffusion temperature, however, was controlled according to that indicated by the thermometer 5.75 inches below the juice outlet. It was found out that, in most cases, the temperature drop from this point to the last thermometer on the left leg was usually 1.5-2°C. Therefore, the temperature at this point was always controlled to be 1-1.5°C above the required average diffusion temperature.

The diffusion tube was 106.63 inches in length, approximately equivalent to that of 26.6 conveyor units. That is, the first lot of exhausted pulp would come out of the discharge end of the diffusion tube while the 27th unit was charging.

In order to check the flow rate of diffusion water, the weights of diffusion juice and pulp collected during a period of 10 units were determined as the experiment proceeded. The collection of juice was usually begun with the 35th diffusion unit when it reached the level of overflowing juice. This was continued until the 45th unit reached the same level. Thus a sample juice of 10 units was obtained while 10 units of pulp were also collected in the meantime. These samples were weighed and recorded. The flow rate of diffusion water and draft could then be computed by a simple material balance.

After 25 units of cossettes had been charged, refractometric Brix readings were taken on the exit juice for every five units. When this reading became reasonably constant over a period of 26-30 units, it was assumed that steady state had been reached. At this time, a sample juice of 10-25 units and one lot of pulp were taken for analysis. For experiments on the diffusion data, juice samples were also withdrawn from the sampling tubes as quickly as possible. The juice samples were taken in 4-inch screw-capped test tubes, 6 in all.

The conveyor was continued until all the pulp had come out. The diffusion tube was drained and thoroughly flushed with water for several hours. Formaldehyde was used to prevent bacterial action.

2. The Chemical Pretreatment Test.—The operating procedures of a chemical-pretreatment experiment differed from its control run only in the method of charging cossettes. That is, before it was charged into the diffuser, each lot of cossettes was mixed in a beaker with 5 ml of the solution of aluminum sulfate, or suspension of calcium oxide. The cossettes were mixed for about 2 minutes and then introduced into the diffuser. The amount of the solution was sufficient to distribute uniformly on the surface of cossettes, leaving no spare liquid in the mixer.

The solution (or suspension) was prepared in such a way that 5 ml of it would contain the amount of chemical corresponding to 0.05, 0.10, or 0.15% by weight of one lot of cossettes.

3. The Diffusion Battery Test.—The diffusion battery, as described in Progress Report 3, was also employed for comparative studies. The same operating procedures were followed in this work.

F. ANALYSIS

The analytical methods employed in this work may be summarized as follows:

1. Sugar in Cossettes.—The Sachs-Le Docte method was followed. Hot digestion at 75-85°C was usually done before polarization. A correction for the higher marc content of the beet was believed to be unnecessary because the correction was smaller in value than the error introduced by the sensitivity of the saccharimeter.

2. Sugar in Exhausted Cossettes.—The hot Sachs-Le Docte method was employed.

3. Brix of the Diffusion Juice.—The refractometric Brix of raw juice was obtained with a Baush and Lomb precision sugar refractometer.

4. Sugar in Diffusion Juice.—The sugar content of raw juice was determined by the method of polarization. The method was described in Progress Report 1.

5. Nitrogen Results on Nitrogen Determination.—These include ammoniacal and organic nitrogen, but do not include nitrate nitrogen. The determinations were made by the standard Kjeldahl method. For a better com-

parison of results, the percentage of nitrogen was divided by the Brix number of the juice to give percent nitrogen per 100 Brix.

6. Pectin.—Silin's method as described in Progress Report 3 was used to determine the pectin content of the raw juice.

7. Color.—The spectrophotometric transmittancy was employed as the color scale of raw juice. The juice was diluted to 10 Brix and analyzed with a Cambridge spectrophotometer for its transmittancy. A wave length of 600 milimicrons and one inch tube were used in all analyses. Distilled water was employed as reference solvent.

8. Sugar in Waste Water.—For tests on the diffusion battery process, the sugar loss in the waste water was determined in the following procedure. One hundred ml of diffusion waste water was taken. This was mixed with some drops of lead subacetate solution, filtered, and polarized in a 200-mm tube. The reading was multiplied by 0.26 to obtain the percentage of sugar in the waste water.

9. Acidity.—The German official method was followed in the determination of acidity of beet juice.

G. DISCUSSION OF DATA

The results of all experiments carried out in this work are presented in Table I. In the major part of the table, data of every two runs are grouped together in order to distinguish that one of them was a comparative run and the other a control run.

Referring to Table I, Column 2 gives the average diffusion temperature in each run. Column 3 denotes the flow rate of diffusion water, as expressed in pounds per pound of beet. Values of draft in percentage are given in Column 4. As is the usual practice, the draft was defined as the percentage of the weight of diffusion juice divided by the weight of sugar beet charged in the same period of time.

Columns 5, 6, 7, and 8 give the results of various sugar analyses.

The apparent purity of diffusion juice as given in Column 9 was defined as

$$\frac{\text{percent sugar in juice x 100}}{\text{refractometric Brix of juice}}$$

TABLE I

SUMMARY OF EXPERIMENTAL RESULTS

1 Run No.	2 Diffusion Temp, °C	3 Diffusion Water, lb/lb Beet	4 Draft, %	5 Sugar in Juice, %	6 Solid in Juice, %	7 Sugar in Fresh cossettes, %	8 Sugar in Pulp, %	9 Apparent Purity, %	10 Percent Recovery	11 Sugar in Waste Water, %	12 Remarks
3	75	1.00	109.5	12.32	13.61	13.77	0.27	90.5	98.3		Reproducibility test
4	75	0.983	109.2	12.13	13.38	13.66	0.29	90.5	98.16		Reproducibility test
7	75	1.123	120.3	11.40	12.72	13.96	0.23	89.7	98.58		
8	70	1.075	113.1	10.74	11.99	12.32	0.17	89.5	98.79		Control run
9	65	1.097	116.3	10.22	11.37	12.21	0.31	89.8	97.77		
10	70	1.128	116.3	10.52	11.69	12.48	0.23	90.0	98.38		Control run
11	75	0.995	111.8	11.63	13.06	13.39	0.38	89.0	97.53		
12	70										Unsuccessful run
13	80	1.105	109.5	13.18	14.54	15.01	0.56	90.65	96.82		
14	70	1.112	113.6	12.77	13.47	15.08	0.58	94.70	96.71		Control run
15	70	1.012	119.0	11.83	13.88	14.51	0.44	85.20	97.40		Control run
16	75	1.005	117.3	12.20	13.62	14.76	0.45	89.60	97.39		
17	70	1.039	111.3	11.95	13.60	14.80	0.50	87.80	97.11		Control run
18	65	1.042	113.8	12.76	14.41	15.04	0.54	88.50	96.93		
19	70	0.992	112.4	12.58	13.99	14.93	0.58	89.9	97.07		Control run
20	80	0.993	109.2	13.0	14.36	14.78	0.58	90.5	96.63		
21	75	0.922	103.0	14.65	16.31	15.74	0.65	89.70	96.58		
22	70	0.889	102.5	14.37	16.15	15.46	0.72	88.75	96.05		Control run
23	65	0.870	102.5	14.63	15.94	15.93	0.92	91.75	95.10		
24	70	0.866	103.3	14.58	15.88	16.02	0.97	91.75	95.30		Control run
25	80	0.875	99.6	15.11	16.45	16.09	1.02	91.75	94.62		
26	70	0.888	103.6	14.67	15.91	16.27	1.07	92.05	94.43		Control run
27	65	1.211	128.0	11.76	12.91	15.53	0.38	91.0	97.98		
28	70	1.187	125.5	12.09	13.26	15.56	0.46	91.15	97.49		Control run
29	75	1.193	125.0	12.60	13.77	15.97	0.22	91.50	98.84		
30	70	1.205	129.6	12.19	13.51	16.07	0.27	90.20	98.58		Control run
31	80	1.184	127.0	12.42	13.77	16.06	0.31	90.15	97.81		
32	70	1.170	124.5	12.81	14.12	16.39	0.44	90.65	97.31		Control run
33	75	1.172	123.5	11.93	13.15	15.72	0.37	90.65	98.02		
34	75	0.983	111.1	13.43	14.69	15.49	0.57	91.27	96.87		Control run
35	75	1.094	120.7	12.85	14.10	15.91	0.46	91.10	97.56		
36	75	1.004	113.2	14.10	15.20	16.48	0.53	92.70	97.30		Control run
37	75	0.916	102.0	14.80	16.27	15.95	0.85	90.90	95.92		
38	75	1.023	114.2	13.79	15.27	16.31	0.57	90.25	97.06		Control run
39	75	0.796	94.25	16.11	17.89	16.38	1.17	90.10	93.94		
40	75	1.012	110.8	14.20	15.81	16.30	0.57	89.75	97.06		Control run
41	75	0.9885	114.7	14.13	15.79	16.87	0.68	89.50	96.63		Control run
42	75	1.00	45.3	14.92	16.39	16.83	4.93	91.0	74.38		0.15% CaO
43	75	1.009	104.5	13.17	14.95	16.08	0.58	88.0	96.9		0.05% CaO
44	75	0.983	107.2	14.44	15.75	15.98	0.51	91.70	97.30		Control run
45	75	1.005	108.6	14.54	15.86	16.38	0.58	91.65	97.04		Control run
46	75	0.995	115.3	13.15	14.54	16.11	0.69	90.40	96.48	0.24	Batch process
47	75	1.001	112.2	14.37	15.59	16.69	0.59	92.15	97.04		Control run
48	75	1.080	98.7	13.87	15.26	16.59	0.59	90.35	97.02		0.1% $Al_2(SO_4)_3$
49	75	0.993	110.7	13.86	14.97	16.08	0.73	92.6	96.16		Control run
50	75	1.004	115.7	13.70	14.82	16.92	0.79	92.4	96.09	0.26	Batch process
51	75	0.980	114.3	14.14	15.50	16.65	0.50	91.20	97.47		Control run
52	75	(1.00)									0.15% $Al_2(SO_4)_3$ Unsuccessful run
53	75	0.9815	98.3	13.25	14.17	16.23	1.18	93.50	93.84		0.05% $Al_2(SO_4)_3$
54	75	0.982	108.9	14.16	15.39	16.11	0.71	92.05	96.28		Control run
55	75	1.023	105.9	13.95	15.40	16.12	0.97	90.55	94.90		0.1% CaO
56	70	0.994	108.5	14.17	15.33	16.13	0.71	92.30	96.24		Diffusion data test
57	70	1.057	112.3	13.94	15.35	16.13	0.46	90.75	97.57		Diffusion data test
58	70	1.221	127.0	13.32	14.54	16.13	0.32	91.5	98.31		Diffusion data test
59	65	1.007	110.8	13.97	15.31	16.14	0.67	91.2	96.49		Diffusion data test
60	65	1.119	114.3	13.66	15.05	16.14	0.51	90.72	97.33		Diffusion data test
61	65	1.192	124.5	12.87	14.24	16.14	0.32	90.25	98.33		Diffusion data test
62	80	0.978	110.6	14.69	16.14	16.81	0.56	91.0	97.21		Diffusion data test
63	80	1.132	117.8	14.05	15.48	16.81	0.43	90.7	97.86		Diffusion data test
68	75	1.00	115.3	13.33	14.48	16.38	0.79	92.0	95.94	0.26	Batch process
69	75	1.017	110.2	14.20	15.54	16.13	0.49	91.3	97.44		Diffusion data test
70	75	1.113	115.6	13.64	15.21	16.13	0.39	89.7	97.90		Diffusion data test
71	75	1.203	124.3	13.59	15.18	16.13	0.34	89.6	98.23		Diffusion data test

Column 10 gives the percentage of recovery of sugar, which was defined by the following approximate equation

C_o = percent sugar in fresh cossettes
C_f = percent sugar in exhausted pulp

$$\text{Percent Recovery} = \frac{\frac{C_o}{1 - C_o} - \frac{C_f}{1 - C_f}}{\frac{C_o}{1 - C_o}} = 1 - \frac{C_f(1 - C_o)}{C_o(1 - C_f)}$$

Column 11 gives the sugar loss in diffusion waste water for tests on diffusion battery.

Two test runs, of which the results are not included in Table I, were made to "tune up" the equipment and to schedule out the operating procedures. It was found that, under full load of the conveyor, the mechanical friction loss could just be overcome by the driving system. A fluid level gradient of 2-3 inches was required for the diffusion water to flow through the bed of cossettes. This was less than the expected magnitude due to fluid friction, calculated on the basis of a Reynolds number of 20.

As far as the raw material is concerned, the California beets were used in Runs 3-11, and Michigan beets in all the other experiments.

The discussion of experimental data may now be made according to the classification of experiments as below.

1. <u>Reproducibility Tests</u>.—In order to determine the reproducibility of the diffusion experiments, Runs 3 and 4 were made on two consecutive days. The procedures of preparing raw material as described in Section D were followed. The diffusion temperature and flow rate of diffusion water in both runs were kept as much the same as possible. Since a close reproducibility was attained, it was decided to proceed on the effects of temperature, diffusion water rate, and chemical pretreatment.

2. <u>Effect of Temperature</u>.—The experimental results on the effect of diffusion temperature were rearranged and tabulated in Table II for clarity. The levels of diffusion temperature were made to be 65, 70, 75, and 80°C. The flow rates of water were taken to be 0.9, 1.0, 1.1, and 1.2 pounds per pound of beet. Since the effect of temperature was to be studied, 70°C was chosen as the control temperature. In each group of experiments, a comparative run was made at 65, 75, or 80°C and a certain flow rate of water. A control run was then conducted at 70°C and the same flow rate. Values of apparent purity of the diffusion juice in both runs were compared as a criterion of the effect of

TABLE II

EFFECT OF TEMPERATURE ON APPARENT PURITY OF DIFFUSION JUICE

Control Temperature: 70°C

Diffusion Water, lb/lb Beet	Diffusion Temp, °C	Run No.	Apparent Purity, %	Relative Apparent Purity	Remarks
0.90	65	23	91.75	1.0	
0.90	70	24	91.75	1.0	Control run
0.90	75	21	89.70	1.0107	
0.90	70	22	88.75	1.0	Control run
0.90	80	25	91.75	0.99674	
0.90	70	26	92.05	1.0	Control run
1.00	65	18	88.50	1.0080	
1.00	70	17	87.80	1.0	Control run
1.00	75	16	89.60	1.0516	
1.00	70	15	85.20	1.0	Control run
1.00	80	20	90.5	1.0067	
1.00	70	19	89.9	1.0	Control run
1.10	65	9	89.8	0.9978	
1.10	70	10	90.0	1.0	Control run
1.10	75	7	89.7	1.0022	
1.10	70	8	89.5	1.0	Control run
1.10	80	13	90.65	0.9572	
1.10	70	14	94.7	1.0	Control run
1.20	65	27	91.0	0.9984	
1.20	70	28	91.15	1.0	Control run
1.20	75	29	91.5	1.0144	
1.20	70	30	90.2	1.0	Control run
1.20	80	31	90.15	0.9945	
1.20	70	32	90.65	1.0	Control run

temperature. For the purpose of correlation, the relative purity of diffusion juice of a comparative run was defined as:

$$\frac{\text{apparent purity of comparative run}}{\text{apparent purity of control run}}$$

A plot of relative purity as a function of diffusion temperature was given in Figure 8.

On the basis of relative purity of juice, a conclusion may be drawn that the highest apparent purity was attained at a diffusion temperature of 75°C. The effect of flow rate of diffusion water, however, cannot be rationally shown in Figure 8, since different raw materials were employed among the groups of experiments. Therefore, a cross correlation was not attempted.

As another effect of the diffusion temperature, the percentage of recovery of sugar increased with increasing temperature.

3. Effect of Diffusion Water Rate.—As a result of the experiments on the effect of temperature, 75°C was taken as the optimum diffusion temperature. Hence, all experiments were carried out at 75°C in this part of the experimental work. A flow rate of 1.0 pound per pound of beet was taken as the control value. In the four groups of experiments given in Table III, the flow rate varied from 0.8 to 1.2 pounds per pound. Applying the criterion of relative purity, it could be seen that the apparent purity of diffusion juice increased with decreasing flow rate. On the other hand, a general plot of percent recovery vs flow rate was made in Figure 9. It showed the increasing trend of percent recovery with the flow rate of diffusion water. As a compromise, a flow rate of 1.0 pound per pound of beet was chosen to be the optimum value.

At this point, the characteristic operating conditions of this continuous diffuser may be concluded to be 75°C and 1.0 pound per pound of water to beet.

4. Effect of Chemical Pretreatment.—All the experiments in this part were made at 75°C and 1.0 pound per pound of water to beet, according to the results obtained in previous experiments. The dosages of calcium oxide and aluminum sulfate, as described in Section E2, were 0.05, 0.10, and 0.15% by weight of cossettes charged. The experimental results are summarized in Table IV.

On the addition of calcium oxide, the cossettes turned dark green in color immediately. Such coloration, however, was not observed in case of pretreatment with aluminum sulfate.

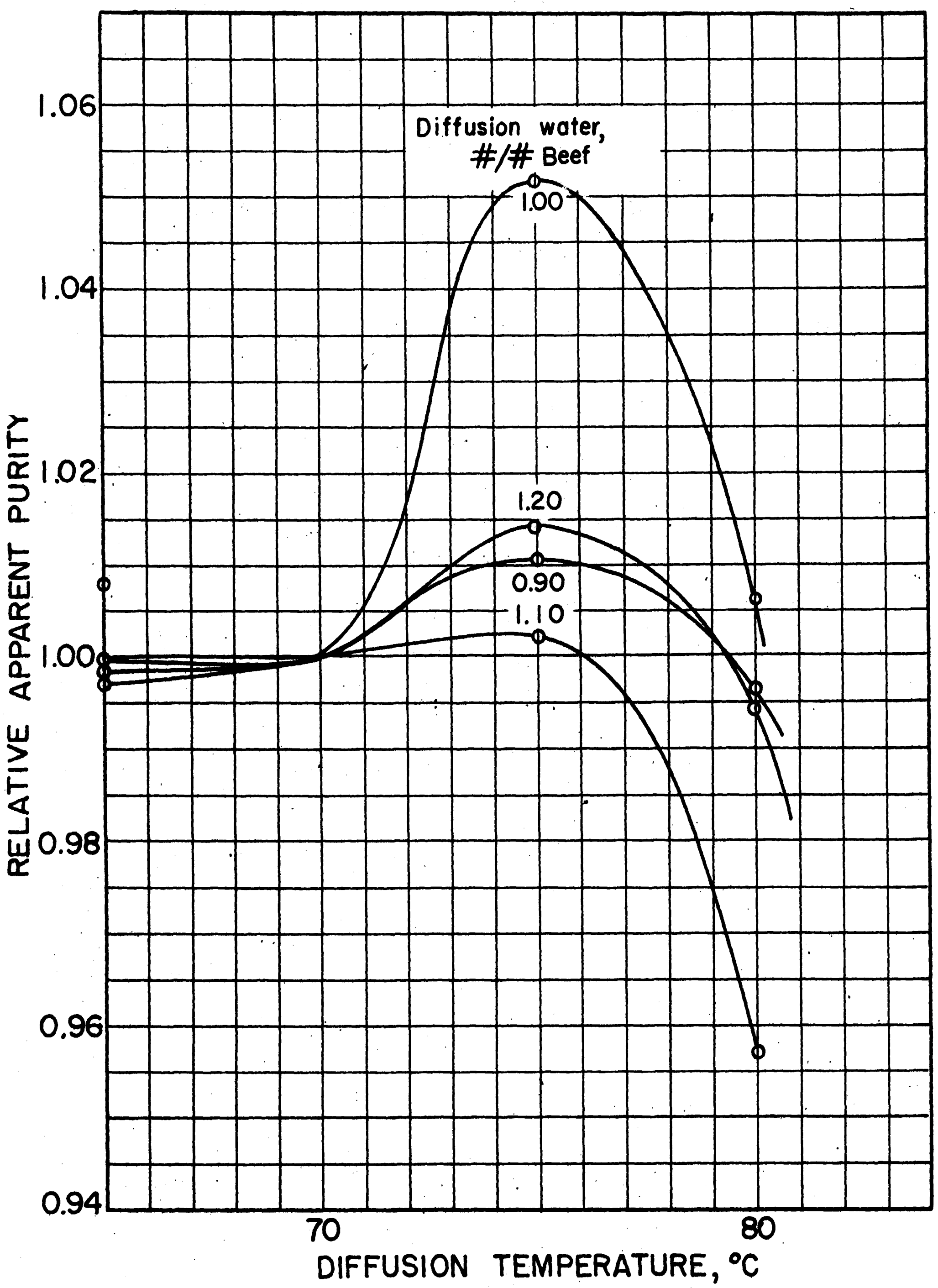


Figure 8. Effect of temperature on apparent purity (control temperature: 70°C)

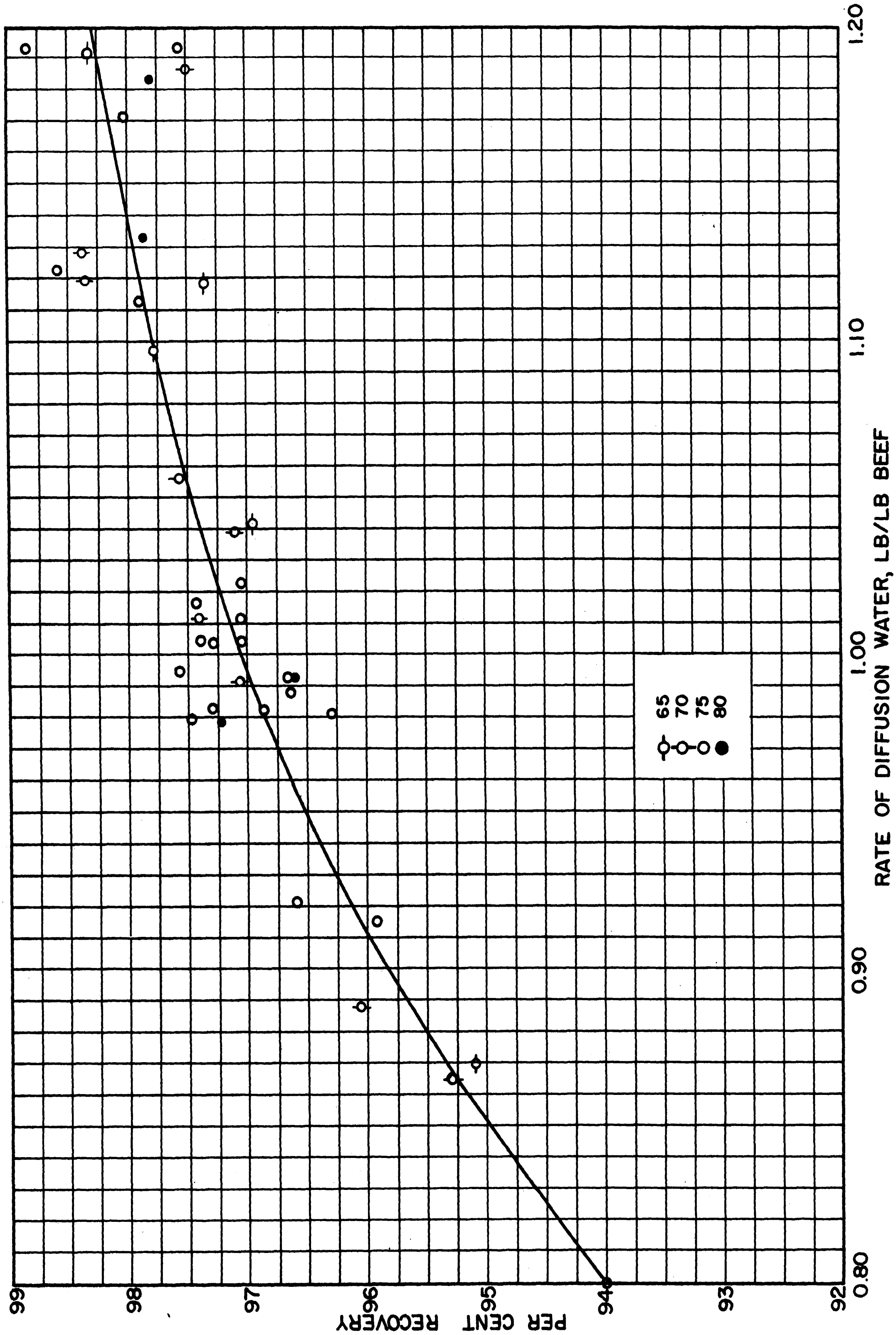


Figure 9. Percent recovery vs diffusion water rate

TABLE III

EFFECT OF WATER RATE ON APPARENT PURITY OF DIFFUSION JUICE

Control Rate of Diffusion Water: 1.0 lb/lb of Beet

Diffusion Water, lb/lb Beet	Diffusion Temp, °C	Run No.	Apparent Purity, %	Relative Apparent Purity	Remarks
0.80	75	39	90.1	1.0039	
1.00	75	40	89.75	1.0	Control run
0.90	75	37	90.9	1.0072	
1.00	75	38	90.25	1.0	Control run
1.10	75	35	91.1	0.9827	
1.00	75	36	92.7	1.0	Control run
1.30	75	33	90.65	0.9932	
1.00	75	34	91.27	1.0	Control run

An apparent swelling of cossettes as a result of chemical pretreatment was also observed. In normal operations of continuous diffusion, the exhausted cossettes in one lot usually occupied an apparent volume of about 65-70% of a conveyor cell unit. Flow resistance to the diffusion water under such condition, as described in previous section, was readily overcome by a fluid level of 2-3 inches. In case of chemical pretreatment with aluminum sulfate and calcium oxide, the apparent volume of exhausted cossettes in one lot was estimated to be 75-78% of each conveyor unit on low dosages. A fluid level of 6-8 inches was required to overcome the increased friction due to the swelling of cossettes at a dosage of 0.10%. Thus, back flow of diffusion water to the discharge end of the diffuser was caused at still higher friction drop. In Run 53, for example, the level gradient required was over 10 inches so the duration of extraction had to be cut down. The diffusion juice was taken from the sampling tap, 5-3/4 inches below the normal outlet. Total diffusion time was about 50.2 minutes in this run as compared to 59.25 minutes in normal runs. The accompanying control run, Run 54, was shortened in the same manner.

In Run 42, in which 0.15% of calcium oxide was added, the back flow of diffusion water resulted in a very low draft and poor recovery of sugar. Also, in Run 52, a pretreatment run with 0.15% of aluminum sulfate, the flow

TABLE IV

DATA ON CHEMICAL PRETREATMENT AND DIFFUSION BATTERY TESTS

Average Diffusion Temperature: 75°C
Average Rate of Diffusion Water: 1.0 lb/lb of Beet

Process	Run No.	Draft, %	Recovery, %	Apparent Purity, %	PH	Acidity, % CaO	N_2 per 100 Brix, %	Pectin, g/100 ml	Color
Normal	44	107.2	97.3	91.7	6.45	0.0384	0.654	0.082	12.0
0.05% CaO	43	104.5	96.9	88.0	6.85	0.0102	0.634	0.089	1.5
Normal	54	108.9	96.08	92.05	6.48	0.052	0.666	0.083	13.9
0.10% CaO	55	105.9	94.90	90.55	7.22	-0.0155	0.644	0.093	4.2
Normal	41	114.7	96.63	89.5	5.92	0.0552	0.673	0.086	16.7
0.15% CaO	42	45.3	74.38	91.0	7.39	-0.0218	0.642	0.076	1.7
Normal	54	108.9	96.28	92.05	6.48	0.052	0.666	0.089	13.9
0.05% $Al_2(SO_4)_3$	53	98.3	93.84	93.5	6.25	0.055	0.632	0.078	23.4
Normal	47	112.2	97.04	92.15	6.04	0.0424	0.654	0.085	9.2
0.10% $Al_2(SO_4)_3$	48	98.7	97.02	90.35	5.65	0.0458	0.653	0.073	18.7
Normal	51	114.3	97.47	91.2	6.35	0.0337	0.649		12.8
0.15% $Al_2(SO_4)_3$	52	-	-	-	-	0.0311	-		20.9
Normal	45	108.6	97.04	91.65		0.0661	0.685	0.093	11.8
Batch	46	115.3	96.48	90.4		0.0257	0.682	0.09	23.2
Normal	49	110.7	96.16	92.6		0.0516	0.659	0.088	13.5
Batch	50	115.7	96.09	92.4		0.0245	0.658	0.089	34.7
Normal	69	110.2	97.44	91.3			0.688		
Batch	68	115.3	95.94	92.0			0.677		

of diffusion water was completely out of control due to high friction drop through the cossettes. No result was obtained on two unsuccessful runs.

The high friction drop and consequent low draft attained in all the pretreatment experiments may of course be attributed to the plasto-elastic nature of the sugar beets. That is, they are elastic at low dosage of chemicals, and deformable at high load.

In order to make a comparison between the normal continuous and chemical-pretreatment operations, the following items are checked between the comparative and control runs: recovery of sugar, apparent purity, nitrogen, pectin, and color of the juice.

In case of pretreatment with calcium oxide, lower draft, lower recovery of sugar, and lower purity of juice resulted. The nitrogen content, for Runs 43 and 45, compares favorably with that of control runs. Increased dissolution of colloids, as shown by the pectin content, was observed. The color of the juice was very poor, possibly due to the coloration of cossettes upon pretreatment.

The pretreatment of beet with aluminum sulfate also gave lower draft and lower recovery of sugar. At a dosage of 0.05%, however, the purity of juice was better than that of the control run. The juice obtained in the pretreatment runs contained less impurities and was in much better color.

Evidently, the swelling effect of cossettes in all cases of chemical pretreatment can be interpreted to cut down the processing capacity of the diffuser.

5. Diffusion Battery Tests.—Results of the comparative experiments on the continuous diffuser and the diffusion battery were included in Table IV. All experiments were set at 75°C and 1.0 pound per pound of water to beet.

The batch process gave higher draft, lower recovery, and lower purity of juice than the continuous process. Content of impurities, as far as nitrogen and pectin are concerned, was in the same order of magnitude in both processes. The batch process yielded juice of better color than the continuous process, however.

6. Diffusion Rate Tests.—The final part of Table I consists of a number of experiments, Runs 56-71 except 68, which were carried out for the correlation of diffusion rate data. The experiments in each group were made in one day by keeping the diffusion temperature constant and merely changing the flow rate of diffusion water. At the time of steady state in each run, seven juice samples were taken along the diffusion tube. The obtained data then enabled the calculation of instantaneous diffusion rate in the continuous

diffuser. The diffusion data, being beyond the scope of this report, are not included. The data presented in Table I, however, could serve to confirm the effects of temperature and flow rate ratio on the purity and percent recovery.

H. BACTERIAL EFFECT

As a check on the bacterial effect in the diffuser, if any, a plot of P_H of diffusion juice vs distance along the diffusion tube was prepared in Figure 10. Data on commercial units, given in "Beet-Sugar Technology" (edited by McGinnis), were transcribed and included in the figure to serve as a basis of comparison. Although there was an abrupt change in P_H near the water inlet due to the high alkalinity of industrial water, it is safe to conclude that no serious bacterial attack in the diffuser occurred. Consequently, the function of the chain conveyor proved to be rather satisfactory and efficient.

III. CONCLUSIONS

1. In view of the apparent purity of raw diffusion juice and the percentage of recovery of sugar as attained by the continuous counter-current diffuser, the optimum diffusion temperature at various water rate was found to be 75°C.

2. The optimum flow rate of diffusion water was taken to be 1.0 percent per pound of beet.

3. The apparent purity of raw diffusion juice from the continuous counter-current diffuser fell within the range of 89-92% which is exceedingly good.

4. The raw juice from the continuous diffuser contained from 10 to 15% sugar, depending on the flow rate of diffusion water.

5. The sugar content of exhausted cossettes ranged from 0.17 to 1.17%.

6. The pretreatment of sugar beet cossettes by calcium oxide and aluminum sulfate resulted in a marked swelling effect of the cossettes.

7. This swelling effect cut down the processing capacity of the continuous diffuser substantially.

8. While the swelling effect of the chemical-pretreatment process increased with the dosage, the maximum amount of aluminum sulfate or calcium oxide added was 0.10% by weight of beet for this continuous diffuser.

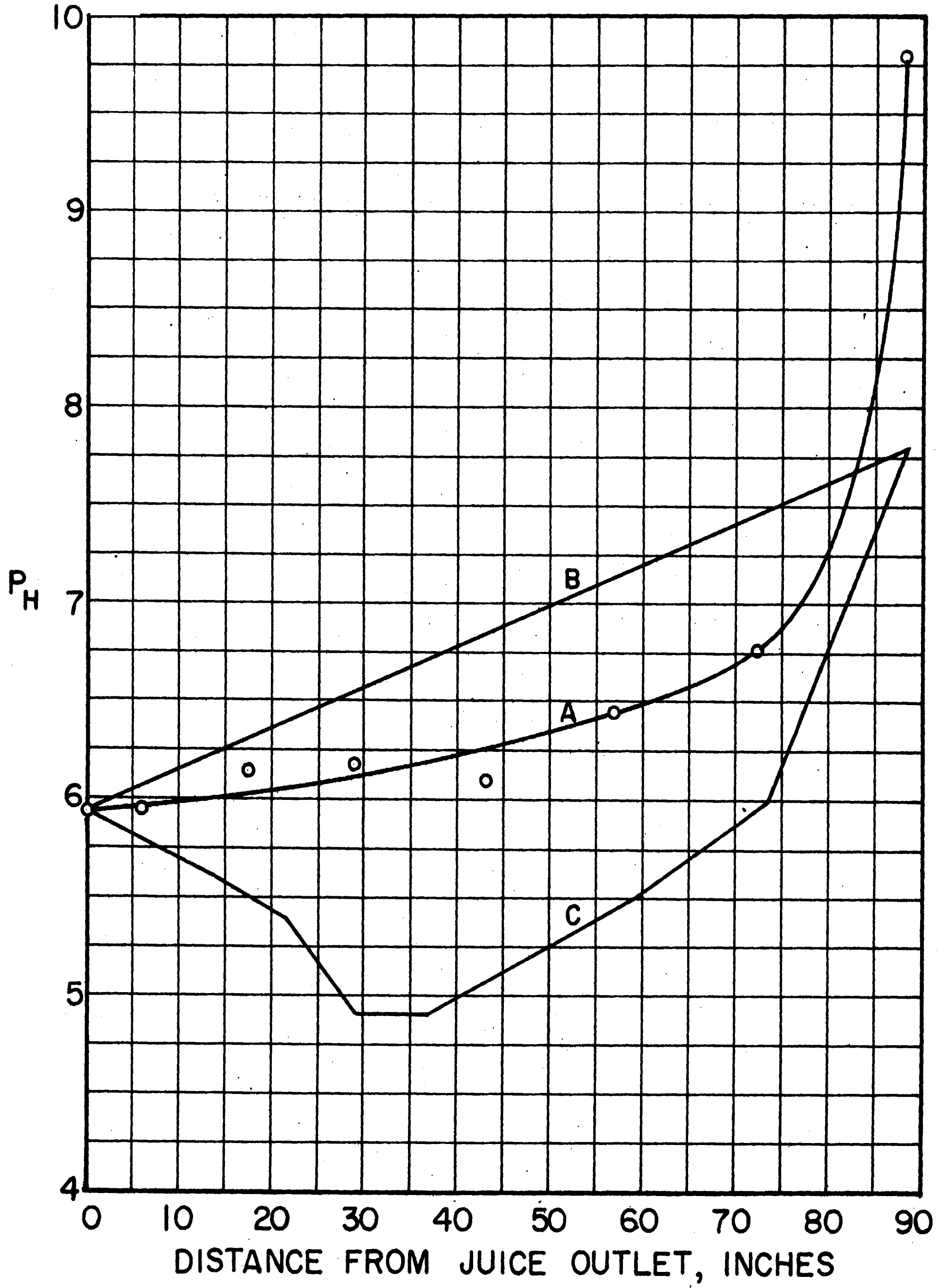


Figure 10. Effect of bacterial attack

Curve A, continuous diffuser data; curve B, transcribed diffusion battery data; curve C, transcribed diffusion battery data, showing effect of bacterial attack

9. Lower draft, lower recovery of sugar, and poorer apparent purity of raw juice were observed in all pretreatment experiments with calcium oxide in comparison to the normal continuous process. The diffusion juice from the pretreatment process was of much poorer color.

10. Lower draft and poorer recovery of sugar were also observed in case of pretreatment with aluminum sulfate. However, the pretreatment process yielded diffusion juice containing less impurities and having better color.

11. The control of P_H below 5.0 in the diffuser, in view of the purity, content of impurities, and color of juice from the pretreatment process with aluminum sulfate, is worth further investigation. The swelling effect on the cossettes may be avoided by operating at low temperature or adding aluminum sulfate directly to the diffuser at some intermediate location.

12. The continuous process compared favorably with the batch process in regard to draft, recovery of sugar, and apparent purity of raw juice. The batch process, however, yielded juice of better color. A conclusive comparison between the continuous and batch processes cannot be made until an economic study as to the initial and operating costs is made.