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PROGRESS REPORT III
PROCESSING OF SUGAR BEETS

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ABSTRACT

This report describes the work done since the preparation of the Progress Report II. A number of experiments have been performed in which the "explosion" juice as well as the diffusion juice from sugar beets were submitted to the standard purification process including preliming, liming, first and second carbonation. The characteristics of raw and thin juices prepared by these two different methods were compared. To insure good comparison in each run of experiments, one half of each beet of a given sample was used for the explosion process while the other half was used for preparation of the diffusion juice. Preliminary recovery tests have also been conducted using the batch explosion equipment with two different baffle arrangements.

The report is divided into two parts. The first part deals with the technological value of the "explosion" juice as compared with that of the diffusion juice and consists of two sections. In the first section the laboratory methods used for preparation of diffusion and thin juices are described in detail. In the second section the experimental data are presented and discussed. The second part of the report discusses the preliminary recovery tests recently performed on the batch explosion equipment.

PROGRESS REPORT III
PROCESSING OF SUGAR BEETS

PART I. QUALITY OF THE "EXPLOSION" JUICE AS
COMPARED WITH THAT OF THE DIFFUSION JUICE

Section I. Experimental Technique

In the previous experiments reported in Progress Report II the purity of the "explosion" juice was compared with the purity of the juice prepared by hot digestion of cossettes at conditions of time and temperature similar to those used in the diffusion process. This method, although simple and reliable, gave a juice too dilute to be used for further processing to thin juice. Therefore, in the present experiments it was decided to use diffusion juice prepared on a small scale diffusion battery.

Diffusion Battery

Small scale diffusion batteries have proved most successful not only in investigations of the diffusional process itself but also in the study of technological value of different varieties of beets. P. M. Silin in his studies on this subject (1,2) found that a small scale diffusion battery of a capacity 200 gr of cossettes per cell gave good reproducible results.

In this work a diffusion battery was used which consisted of 10 cells (see Fig. 1, 2, 3). The capacity of each cell was 200-250 gr of beet depending on the size of cossettes. The cells were made of copper pipe, 2 inches in diameter, open at the upper end. The center of the bottom of each cell contained a short $\frac{1}{4}$ " copper tube, connected by a piece of rubber tubing with the upper part of the next cell. In order to secure a good distribution of juice and to facilitate discharging of exhausted cossettes a stainless steel screen shown on the right of Fig. 2 was placed on the bottom of the cell. A ring shaped support welded to the screen provided a distributing space $\frac{3}{8}$ inches high between the screen and the bottom of the cell. A metal rod welded to the center of the screen enabled easy removal of the

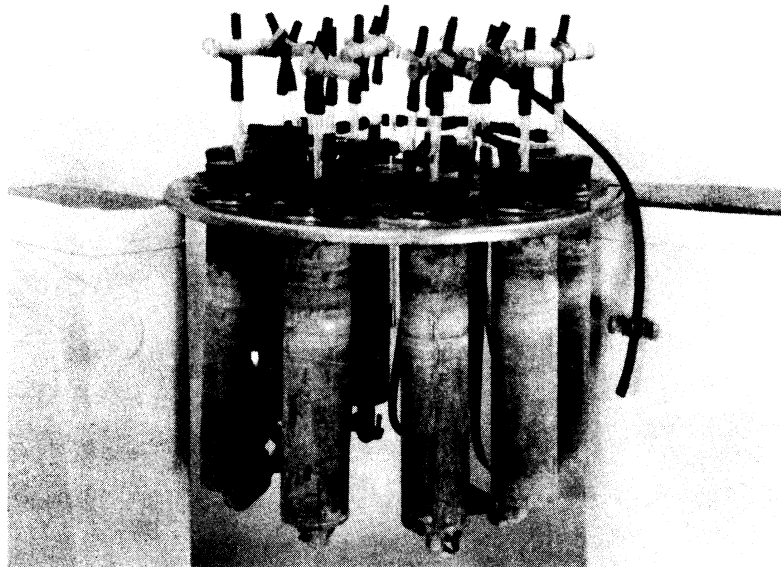


Figure 1. Diffusion Battery

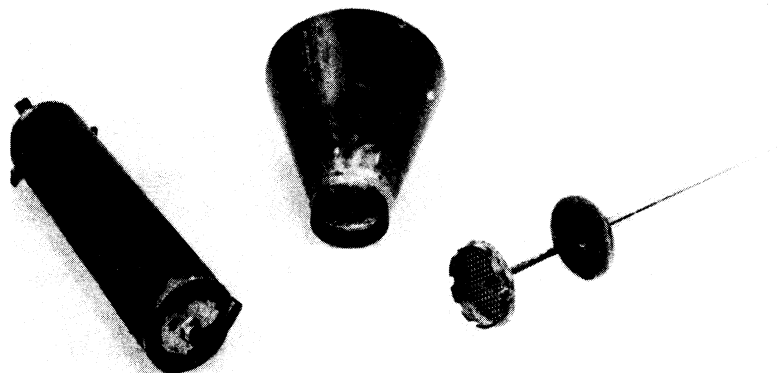
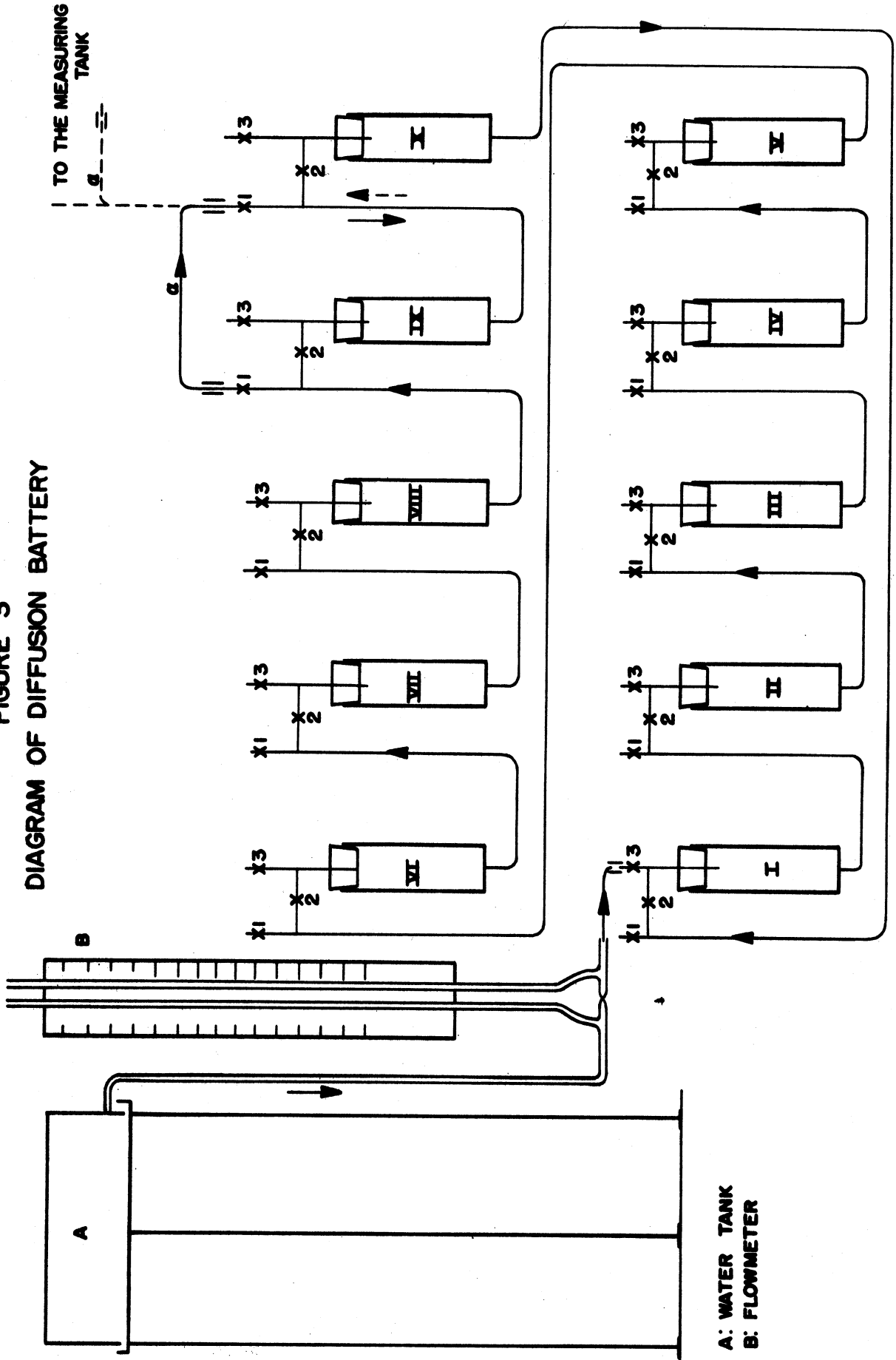


Figure 2. Diffusion Cell and Accessories

FIGURE 3
DIAGRAM OF DIFFUSION BATTERY



A: WATER TANK
B: FLOWMETER

exhausted cassettes. The cells were charged through a copper funnel and lightly pressed with a metal stamp. The hollow handle of the stamp enabled its free movement along the rod of the screen (Fig. 2 right). To discharge the exhausted cassettes, first the funnel was put on the top of the cell (Fig. 2) and then the cassettes transferred to it by lifting the screen. This arrangement worked without difficulty and enabled smooth and quick operation. The upper part of each cell was closed with a rubber stopper provided with a glass tube connected with the bottom of the preceding cell and with the measuring "tank".

The enclosed diagram (Fig. 3) gives a flow sheet of the arrangement and operation of the battery.

Water at temperature 65°C was introduced into the diffusion battery from the water tank A through the flowmeter "B", the flow being regulated so as to keep the time of diffusion equal to 60 minutes. The diffusion cells were connected together in the form of a circle (Fig. 1) and placed in a water bath adjusted to 85°C . The above arrangement of cells gave a very compact diffusion unit. The outside diameter of the bath was 16 inches.

The usual working procedure was as follows (Fig. 3): e.g. Water enters cell I, clamp 3 is open, clamps 1 and 2 on the cell I are closed. Clamps 2 on cells II, III, IV, V, VI, VII and VIII are open and the juice flows from cell I to VIII in the direction from top to bottom in each cell. Cell number IX has been filled with cassettes and is now to be filled with juice, clamps 1 and 3 on the cell IX are left open but the clamp 2 is closed. On the cell X clamp 1 is open and clamp 2 is closed. The juice which emerges from the bottom of the cell VIII flows therefore through the open clamp 1 on the cell IX and through the detachable branch "a" enters the cell IX from the bottom. As soon as the cell IX has been filled, clamps 1 and 3 on the cell IX are closed, clamp 2 opened, and the left side of the branch "a" disconnected and introduced into the measuring "tank" (the dashed line). The juice from the diffuser VIII passes then through the open clamp 2 and enters the diffuser IX from the top. The juice displaced from the cell IX flows from the bottom of the cell and through the open valve 1 (on the cell X) to the measuring "tank". In the meantime the cell X is charged with cassettes. As soon as the required amount of juice has been withdrawn from the cell IX the filling of the cell X with juice begins, the water pressure is switched from cell I to II and the cell I emptied, rinsed with water and charged with fresh cassettes.

As the charging and discharging of cells did not take more than 5 minutes, 9 cells were always in operation. In all experiments presented in this work the charge was always 200 gr of cassettes, per cell, the draft was kept at 110% by vol; the diffusion time 60 minutes, the temperature of water 65°C and the temperature of the bath 85°C .

Usually 1500 ml. of juice were withdrawn from the battery,

1000 ml. were used for processing to the thin juice and the rest left for determination of Brix, Pol, invert sugar and pectins.

Preliming

One liter of the juice withdrawn from the battery was quickly heated to 90°C and submitted to hot progressive preliming (method of Dědek and Vašatko) (3 and 4). Forty ml. of milk of lime containing 10 g. of CaO per 100 ml. was added dropwise to the juice at constant stirring. The time of preliming was approximately 4-5 minutes. It has been noted (3) that for a given juice there is a maximum precipitation of protein at a certain optimum alkalinity or pH. If milk of lime is added at once in preliming, the maximum elimination of protein is reached only if the exact amount of lime is used. Therefore, with the continuously changing composition of the juice in a factory it would be practically impossible to obtain the maximum effect by adding lime at one time.

On the other hand, when lime is added slowly a certain stabilization of the precipitate takes place which even in case when lime is added in excess a very efficient coagulation can be obtained.

Main Liming

The juice cooled to about 80°C during preliming was brought up again to 90°C and 80 ml. of lime of the same concentration as previously used, added in one lot. The limed juice was kept at constant stirring for 5 minutes.

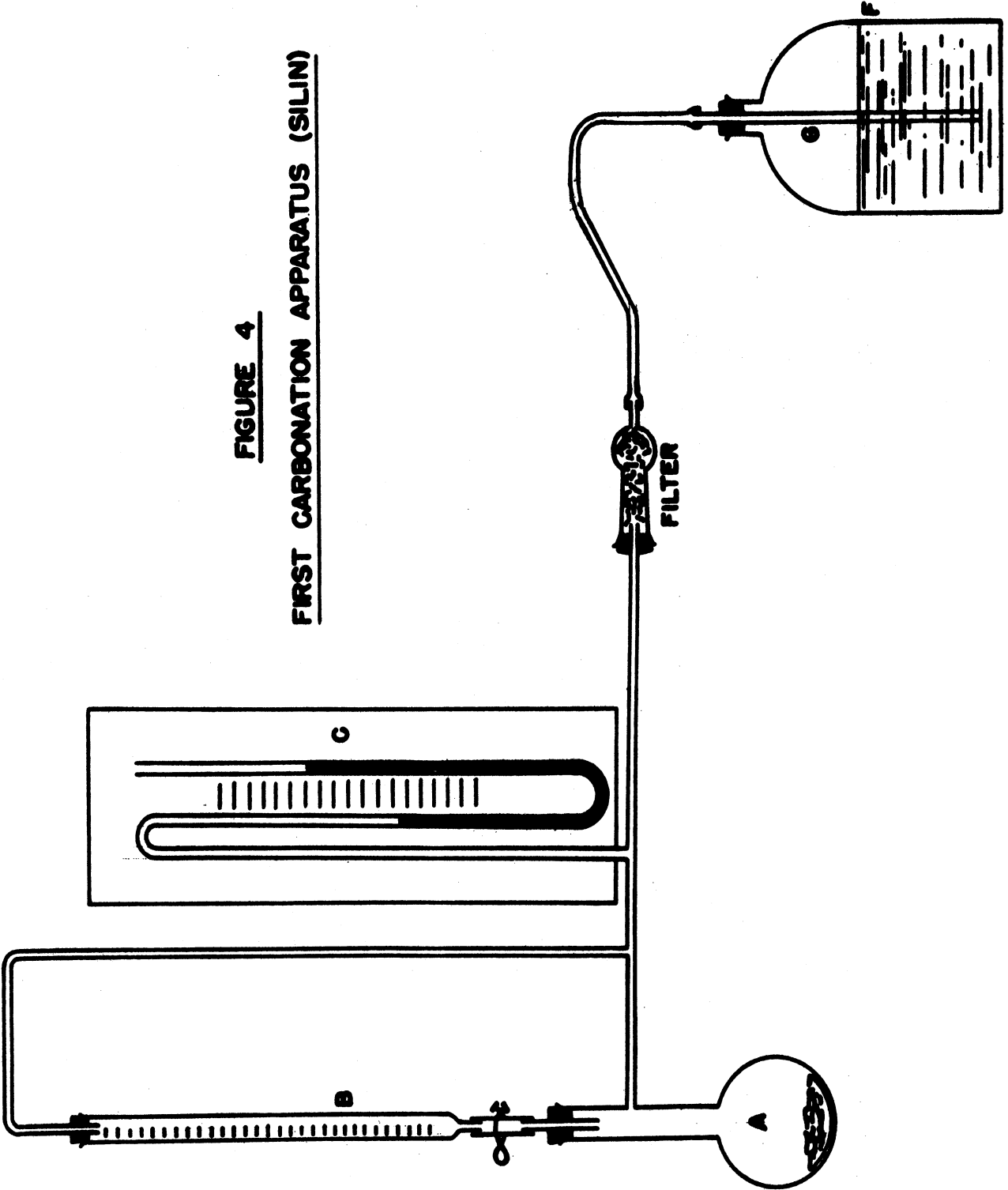
First Carbonation

This is the most delicate stage of the purification process and requires great care and attention. Not only the optimum end point must be reached but the time of carbonation should be kept approximately constant to enable a good comparison of some of the properties of juices. Over and undersaturation as well as too long a time of carbonation influence the velocity of filtration of the first carbonation juice and the color of the thin juice (5). In our laboratory carbonations the method recently published by P. M. Silin (1,2) was used. This method has proved to be very reliable especially when small samples of juice are to be carbonated. To the best of our knowledge this method has not been published in English, therefore, a brief description is given below (see also Fig. 4).

Carbon dioxide for the carbonation is generated by the action of HCl (1 volume of concentrated hydrochloric acid plus 1.5 volume of water) on NaHCO₃. The hydrochloric acid is introduced from a 50 ml. burette "B" into a 500 ml. distilling flask "A" containing 25 gr of NaHCO₃.

The approximate amount of hydrochloric acid necessary for

FIGURE 4
FIRST CARBONATION APPARATUS (SILIN)



carbonation is first determined by titration of 100 ml. of milk of lime (of the concentration used for liming) with the same hydrochloric acid against phenolphthalein. The amount of hydrochloric acid used divided by two gives the approximate amount of acid necessary for carbonation.

Before carbonation, 6 ml. of acid are introduced from the burette to expell the air from the whole assembly by carbon dioxide. The above 6 ml. of acid are not included in the total amount of acid to be added. When the air has been displaced by CO₂ the glass tube "G" is introduced into the carbonation kettle "F" but above the level of the juice. Then three more milliliters of acid are added to expell air from above the level of the juice. However, this amount of acid should be included in the total amount previously calculated. Then the stopper with the glass tube is tightly fixed in the neck of the carbonation kettle and the remaining amount of acid is slowly added. When the mercury gauge "C" shows increase in pressure the kettle is shaken vigorously to keep the pressure of CO₂ below 12 cm. of Hg. When the total calculated amount has been added and the pressure decreased to 1 cm. the juice is tested with thymolphthalein paper. If the reaction is deep blue (Ph>11) more acid is added in lots of 1 ml. In this case the glass tube in the kettle "F" is kept above the level of juice and after addition of 1 ml. of acid the glass tube "G" is removed, the kettle tightly stoppered with another rubber stopper and shaken vigorously for about 30 seconds. When the coloration of the thymolphthalein paper is still deep blue, the same operation is repeated until a light blue coloration (Ph=11) of the test paper is obtained. After a certain experience the time of carbonation can be well regulated. The carbonated juice is adjusted to 75°C, and 300 ml. used for the determination of the rate of filtration. The remaining portion is filtered through a BUCHNER funnel. The resulting filtrates are mixed together, heated to 90°C and submitted to second carbonation.

Determination of the Rate of Filtration of the First Carbonation Juice

It was decided that it would be desirable to determine the rate of filtration of the juice after first carbonation. The importance of this characteristic of the juice cannot be over emphasized. Juices even of low purities can be processed successfully, but bad filtration slows down the production rate and may even stop the production altogether. In our work this property of the juice was of special importance. The cassettes were subjected to direct action of saturated steam, which might have resulted in dissolution of some of the solid constituents of the beet, especially of pectins. On liming, the pectins form voluminous colloidal precipitates and even viscous jelly-like solutions which filter with great difficulty. This is one reason why the rate of filtration of the first carbonation juice was investigated in addition to the determination of pectins in the explosion juice. The measurements of the rate of filtration were carried out on the filtration apparatus designed by SPENGLER, TÖDT, AND BÖTTGER (6) and the equipment is shown in

Figure 5. The principle of this apparatus is the use of a freely suspended paper filter which is held fast at its upper edge between two concentric covers. In our experiments the suspended filter was inserted in a glass funnel to decrease heat losses. The glass funnel was sufficiently large to avoid any contact with the suspended

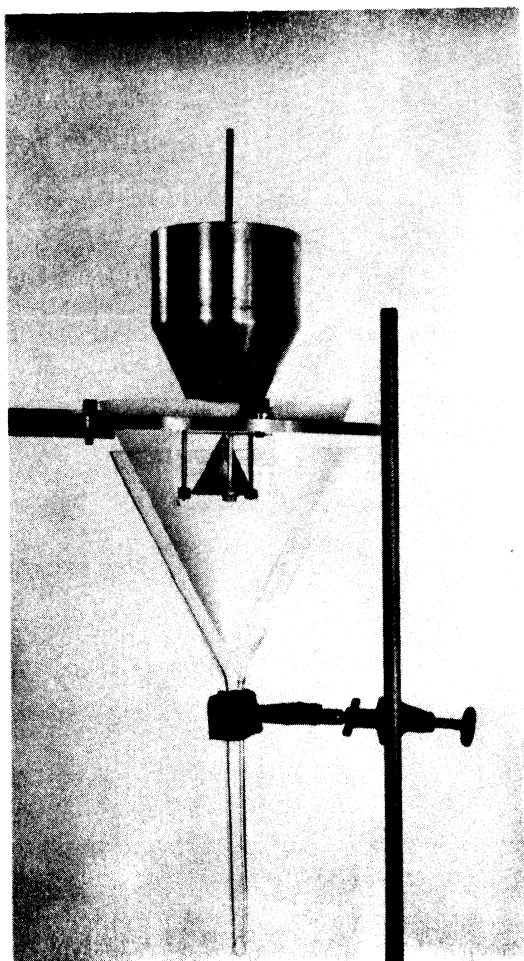


Figure 5. Apparatus for Measuring of the Rate of Filtration

filter. Three hundred milliliters of a well mixed juice at 75°C was placed in the upper container which was closed at the lower end by a conical metal stopper. The stopper was then slowly lifted and the juice allowed to flow on the paper filter, the metal cone mounted inside the filter* insures a uniform distribution of the juice.

* A hole has been cut in the paper filter to show the distributing cone.

The filtrate was collected in a measuring cylinder, and the time was noted during which 50, 100 and 150 ml. of filtrate were obtained. According to the investigation of the German Sugar Research Institute in Berlin (6) this apparatus gives good reproducible results and allows conclusions to be drawn on the filtration properties of the juice in the large scale production.

Second Carbonation:

The filtered first carbonation juice was first heated to 90°C and then saturated with CO₂ to a reaction neutral to phenolphthalein. The carbonated juice was then boiled for 10 minutes in order to decompose calcium bicarbonate and filtered through a BUCHNER filter.

Analytical Determination of Raw Juice

Sugar: determined by the usual method described in Progress Report I.

Brix: determined by BAUSCH and LOMB precision refractometer and by the densimetric method (Pycnometer).

Pectin: determined by SILIN method (7,8). This method is based on the formation of furfural from pentosan and galacturonic acid upon distillation with acid.

The furfural obtained is calculated to pectin by means of an empirical factor. Five milliliters of raw juice (either diffusion or explosion juice) was made up to the volume of 40 ml. by adding 2.5 ml. of concentrated hydrochloric acid plus 32.5 ml. of hydrochloric acid of sp. gr. 1.06. The solution was distilled uniformly and slowly to obtain 30 ml. of distillate in not less than three hours. The use of paraffin bath (8) gave more uniform distillation than gas heating used in the original method. The distillate was then transferred to a 100 ml. volumetric flask, neutralized with NaOH solution and made up to the mark. To 5 ml. of the solution 4 ml. of acetic acid and 0.5 ml. of anilin were added and the sample left in a dark room for 10 minutes. The intensity of the red coloration developed is a function of the concentration of furfural. This coloration is not very stable and it is therefore important to measure it precisely after 10 minutes. The concentration of the furfural in the sample was determined in the Cambridge Spectrophotometer. A correction must be made to this determination to allow for the amount of furfural formed from sucrose. According to Silin 1 g of sucrose forms under the conditions of the method 0.00125 gr of furfural. The corrected furfural was then multiplied by 3.8 to convert it into pectin. It is important to note that in determining this factor Silin assumed as "pectin" ("PEKTINSTOFFE") the dry substance of an extract obtained from completely sugar-free cosettes by boiling with water (7). This extract contained ash for which no correction is made.

Thin Juice

Sugar: determined by the usual method.

Brix: determined by BAUSCH & LOMB Precision Refractometer and by the densimetric method. (Pycnometer). This enables a comparison of pycnometer as well as refractometer purities.

Color: thin juice adjusted to 10 Bx was analysed in the Cambridge DG. Spectrophotometer within a wave length range of 425 to 625 millimicrons. One inch tubes were used in all analyses. The values of $-\log T^*$ are plotted against the corresponding wave lengths.

Specific Conductance

Determined by SALOMETER (Magic-eye model, Sugar Manufacturers Supply Co., London) at a concentration of 5 Bx (9).

Lime Salts

Determined by filtration with soap solution according to SPENGLER and BRENDEL (10). As the concentration of thin juices in each run of experiments was kept approximately constant the lime salts content was expressed in milligrams of CaO in 100 ml. of juice.

Alkalinity

The optimum alkalinity of the second carbonation was determined by the method of SPENGLER and BOTTGER (11). In this method it is assumed that the minimum of lime salts (optimum alkalinity) is reached when neither free hydroxides nor bicarbonates are present. A sample of juice is titrated with hydrochloric acid first with phenolphthalein then with methyl red as indicator. If both titrations give the same result, the optimum alkalinity is reached. If the amount of acid used in titration with phenolphthalein is greater the juice is undersaturated. If, on the other hand, the titration with phenolphthalein required smaller amounts of acid, the juice is oversaturated.

Reproducibility Tests

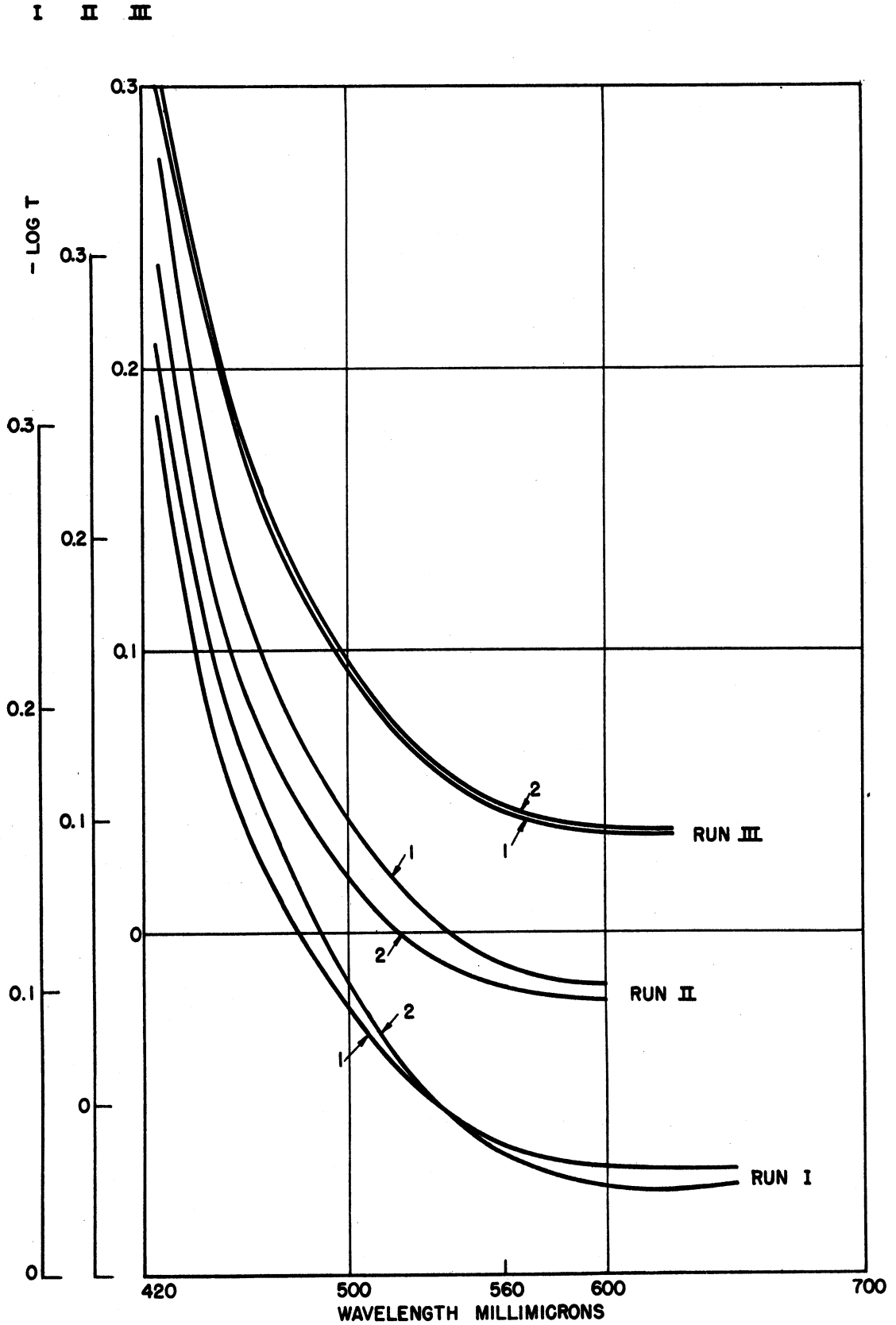
Three separate runs of experiments have been performed to determine the reproducibility of the preparation of the diffusion juice and of its purification by the previously described laboratory methods. In these experiments sugar beets from Michigan area were used. Each beet from a sample selected for a given run was cut lengthwise into two halves and in this way the sample divided into two parts of the same properties and composition. Each was then separately processed under the same standard condition of diffusion, liming and carbonation. The juices obtained were analyzed and their properties compared. The halves were sliced into 2 inch long cassettes by the method described in Progress Report II (page 1).

*T- Transmittancy of a solution (transmission after correcting for reflection at the cell surfaces and absorption by the solvent).

TABLE I
Reproducibility Tests

Run Duplicate	1		2		3		4		5		6		7			8			9			10			11			12			13			14			15			16		
	Diffusion Juice																		1-st Carbonation Juice						Thin Juice																	
	Pol %	Bx (pycn)	Bx (Refr)	Q (pycn)	Q (Refr)	Pectin g/100 ml	Alkalinity % CaO	50 ml	100 ml	150 ml	Phenol-phthalein	Alkalinity % CaO Methyl Red	Pol %	Bx (pycn)	Bx (Refr)	Q (pycn)	Q (Refr)	Lime Salts mg/100 ml																								
a	12.49	13.80		90.5		0.10	0.069	3"8"	3"7"	7"23"	0.025	0.022	13.10	14.08		93.0		1.6																								
b	12.56	13.89		90.4			0.079	3"6"	3"6"	7"26"	0.024	0.022	13.32	14.32		93.0		1.1																								
a	13.48	14.88		90.6		0.10	0.087	1"7"	3"30"	8"30"	0.029	0.025	14.06	15.02		93.6		1.0																								
b	13.67	15.04		90.9		0.09	0.092	55"	2"58"	7"30"	0.031	0.027	14.32	15.34		93.4		1.1																								
a	14.58	15.92	15.84	91.6	92.0	0.09	0.080	1"8"	3"40"	8"40"	0.030	0.027	15.15	16.09	15.91	94.2	95.2	2.0																								
b	14.28	15.55	15.45	91.8	92.4	0.085	0.086	1"4"	3"26"	8"10"	0.033	0.026	14.55	15.36	15.26	94.7	95.3	1.7																								

FIGURE 6
REPRODUCIBILITY TESTS
SPECTROPHOTOMETRIC ANALYSIS
OF THIN JUICES



The sample obtained was well mixed, divided into lots of 200 g weight and each lot tightly wrapped in aluminum foil. One packet of cossettes was then used for each diffusion cell. The draft was maintained at 110 per cent by volume throughout all experiments. The results are presented in Table I. It can be seen from the table that the diffusion battery gave good reproducible results, the maximum difference in purity (column 4) was 0.3, that is almost within the experimental error of the determination. Good agreement was also obtained as far as pectin (column 6) and the concentration of the juice (column 2) are concerned. The alkalinity of the juice after first carbonation shows good reproducibility (column 7). There is also a fair agreement in rates of filtration especially in Runs I and III. A greater difference in rates of filtration in Run II might have been due to the fact that in one duplicate of this run the filter paper broke and the filtration was repeated. Columns 9 and 10 show that the optimum alkalinity of the second carbonation was closely reached. This explains also the comparatively low lime salts content of the juice (column 16). The purities of the thin juice (columns 14 and 15) show also a good agreement. The results of the spectrophotometric analyses of thin juice are given in the form of graphs in Figure 6. The values of $-\text{Log } T$ are plotted against wave lengths on three different ordinates each corresponding to one run. This was done to avoid superposing of curves and to obtain a clearer picture. The reproducibility is good especially at wave length 560 m μ where $-\text{Log } T$ is directly proportional to the concentration of coloring matter.

Generally speaking it can be said that the above described procedure of preparation of the diffusion juice and its subsequent purification can be carried out with good precision and used as a standard of comparison for other methods. A time of about two and a half hours is sufficient to prepare one liter of the diffusion juice and to purify it to the thin juice.

SECOND SECTION

Experimental Data and Discussion

In Progress Report II, Table VI, it was shown possible to obtain juices of comparatively good purity only 0.3 lower than that of the centrifuge juice and 0.7 lower than that of the hot digestion juice by selecting the right conditions of steam treatment. It was suggested (on page 22 of Progress Report II) that this small drop of purity might have been due to the fact that it took about 3 minutes to open the receiving vessel and collect the mash. During this time the mash was in contact with the hot walls of the vessel and thus the time of heat treatment substantially increased. The experiments performed just after publication of Progress Report II (13) and presented in Table II show that when this prolonged action of heat was avoided by flashing after explosion, no drop of purity occurred. (Compare column 17 with columns 5 and 12.)

Obviously the allowable conditions of steam treatment, time and temperature must not be regarded as constant and of general value. They hold for a given lot of beet only and even in this case may change with the duration and condition of storage. This point will be discussed later in detail. The experimental data presented in the following part of this section were obtained with beets from Michigan area. About 300 pounds of undamaged and well washed beets were stored for this purpose in crates at a temperature of +1 to +3 degrees C. The experiments were carried out in the month of February, 1953. Three preliminary explosion tests were made with this material to determine the maximum allowable steaming time. The experimental technique was the same as that described in Progress Report II, pages 22-25. After each explosion test the material was cooled down by flashing. The results are given in Table III. In Run I the cossettes were preheated for three minutes with steam at atmospheric pressure, then the pressure rapidly increased to 55 psig and the cossettes blown out against a baffle in the receiving vessel. Run II was carried out in exactly the same way except that the time of steaming was shortened to 1 minute. In Run III the time of steaming was 3 minutes but the temperature of steam reduced to 90° C. (9 in. Hg. Vac.) It is seen from this table that with this beet material a 3 minute steaming at atmospheric pressure causes a drop in purity of more than one percent. On the other hand one-minute steaming at atmospheric pressure or three minutes steaming at 90° C can be regarded as safe as far as the purity of the explosion juice from this lot of beet is concerned. The influence of high pressure steam was neglected owing to the short time of its action (1 sec. or less).

Table IV gives the results of three complete runs, in which the properties of "explosion" and diffusion juices are compared (16). The sampling procedure was similar to that used in the reproducibility tests. Each beet from a sample selected for one run of experiments was cut lengthwise into two halves and thus the sample divided into two parts of the same composition and properties. One part was immediately sliced by the method previously described, mixed and used either for explosion or diffusion.

The other part was stored for 3-4 hours in a refrigerator, each half covered with a thin layer of paraffin (see Progress Report II, page 12) and wrapped in aluminum foil. After processing the first part to the thin juice, the second part was sliced and processed by the other method. Therefore, the properties of explosion and diffusion juices can be compared in each run, but no comparison is allowed between the runs as each run was carried out with a different sample of beets. In the explosion process the juice was separated from the mash in an 8 inch basket centrifuge, the cake washed with the condensate from the steaming period and the effluent mixed with the juice. In Run I the cossettes were steamed for 3 minutes at atmospheric pressure. Then the pressure suddenly (1 sec. or less) increased to 55 psig. and the cossettes exploded to atmospheric pressure. Run II was carried out in the same way except that the steaming time was shortened to 1 minute. In Run III

TABLE III

Influence of Temperature and Time of Steaming
on the Purity of the Juice

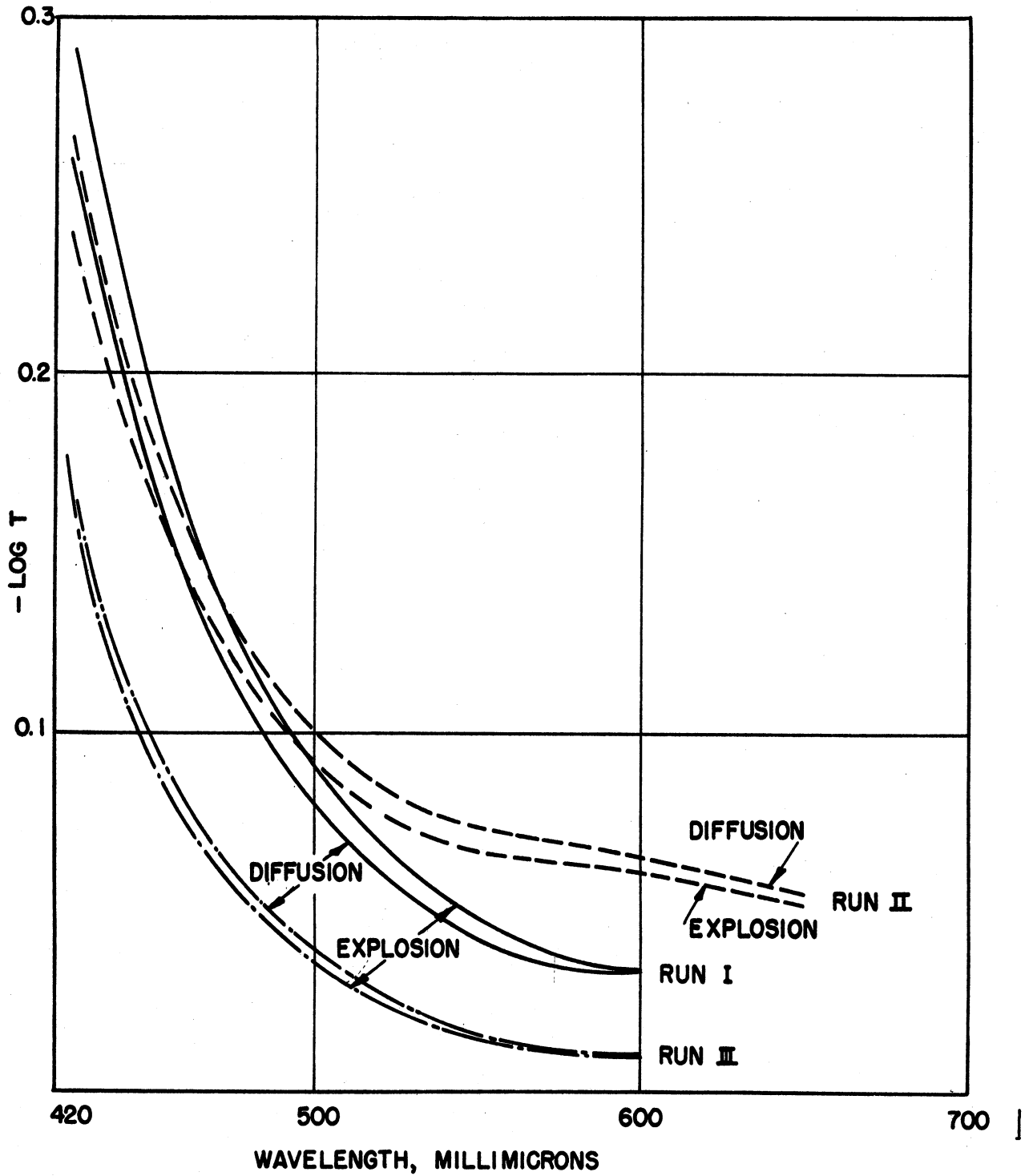
Run	Temp. & Time of Steaming	Digestion Juice			"Explosion Juice"		
		Pol.	Bx	Purity (Refr)	Pol.	Bx	Purity (Refr)
I	100° C. x 3 min.	8.73	9.59	91.0	12.43	13.86	89.7
II	100° C. x 1 min.	" "	" "	" "	13.91	15.28	91.0
III	90° C. x 3 min.	" "	" "	" "	14.28	15.65	91.2

T A B L E I V

Explosion vs. Diffusion Juice

1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	
																		Raw Juice
Run	Process	Pol %	Bx (pycn)	Purity (pycn) (Refr)	Pectin (Silin) g/100 ml	Invert (Ofner) g/100 ml	Alkalinity % CaO	Rate of Filtration			Pol %	Bx (pycn)	Purity (pycn) (Refr)	Lime Salts mg/100 ml	Alkalinity % CaO	Sp Cond at 18°C and 5 Bx mhos/cm	R%	
								50 ml	100 ml	150 ml				Phenol-phthalein	Methyl Red			
I	Explosion 5 minutes steaming at 0 psig	11.58	12.93	89.6	0.16	0.022	0.070	1'20"	4'52"	13'15"	11.53	12.35	93.4	7.0	0.017	0.020	0.001356	39
	Diffusion	11.80	12.91	91.4	0.07	0.033	0.072	58"	3'5"	7'24"	12.48	13.21	94.5	2.3	0.027	0.020	0.001356	38
II	Explosion 1 minute steaming at 0 psig	11.25	12.31	91.4	0.08	0.038	0.065	54"	3'3"	7'30"	11.82	12.35	95.7	2.8	0.018	0.015	0.001246	52
	Diffusion	11.48	12.50	91.8	0.076	0.037	0.067	40"	2'21"	5'50"	11.79	12.42	94.9	1.6	0.018	0.018	0.001308	40
III	Explosion as Run II but cold P.L.	10.99	12.08	91.0	0.06	0.024	0.066	39"	1'58"	4'37"	10.85	11.42	95.0	1.7	0.015	0.018	0.001214	47
	Diffusion	11.55	12.60	91.7	0.06	0.026	0.068	46"	2'19"	5'27"	11.38	12.07	94.3	1.2	0.020	0.026	0.001304	33

FIGURE 7
EXPLOSION vs. DIFFUSION PROCESS
SPECTROPHOTOMETRIC ANALYSIS
OF THIN JUICES.



the condition of explosion were exactly the same as in Run II, but the raw juice prepared by the "explosion" process was prelied at 40 degrees C instead of 90 degrees C which was the temperature of preliing in all the other experiments. In all runs the Brix of the diffusion juice was adjusted to that of the "explosion" juice in order to have the same conditions of concentration, a rather important point when the rate of filtration is to be measured.

As might have been expected the three minutes steaming in Run I resulted in a comparatively high drop of purity of the raw explosion juice (columns 5 and 6). The difference between the purities of "explosion" and diffusion juices was 1.8. There was also a considerable increase in pectin (column 7), but the invert sugar content in the "explosion" juice was small (column 8). The latter was in agreement with our previous experiments (see Progress Report II, Table IV) where even much longer heating increased the amount of invert sugar but little. The rate of filtration (column 10) was decidedly worse in the case of the explosion juice, probably due to the much higher amount of pectin. The purities of thin juices (columns 13 and 14) showed a much smaller difference than those of the corresponding raw juices (columns 5 and 6). Lime salts content was higher in explosion process which might have been partially due to the greater amount of pectin (1). The measurement of spec. conductance showed that the content of mineral constituents was the same in both juices. The last column (18) gives the percentage of nonsugars removed in the process of purification. This value (R) was calculated from Pol. and Brix (Pycn) of Raw and thin juices. In Run I in spite of rather serious decrease of the quality of the "explosion" juice the value of R (effect of purification) as shown in column 18 was practically the same as that of the diffusion juice. In both runs II and III the purity of the raw explosion juice is slightly lower (column 5) but the situation changes after purification and in both runs the juice obtained by the explosion process had a higher purity (column 13). The values of R (effect of purification) in column 18 are considerably greater for the "explosion" juice. This increase of purity, if confirmed by future experiments, would be a great asset of the explosion method. An approximate calculation of the increase of the yield of sugar due to the increased purity of the juice can be made by HULLA-SUCHOMEL-PAAR Formula (14), substituting for Sq the purity

$$X = 10^4 \frac{Fq - Sq}{Fq (100 - Sq)}$$

of molasses, e.g. 60 and for Fq the purity of the juice we obtain for the "explosion" juice in Run II $X=93.26$ percent of sugar recovered from that present in the juice. The same calculation for the diffusion juice of Run II gives $X=91.94$ percent. The increase of sugar recovered from the "explosion" juice equals $93.26-91.94$ which equals 1.32 percent of sugar in the juice or assuming the sugar content of the beets to be 17.5 percent and sugar losses as 1.0 % of beet, the increase of recovery will be 0.22 % of sugar per beet followed by decrease in molosses of about 0.44 % per beet. A similar calculation based on refractometer purity (column 14) gives in case

of Run II the increase of sugar recovered equal to 0.24 percent per beet or 0.48 percent less of molasses per beet. The decrease in formation of molasses can be also calculated by the empirical formula recently published by K. SANDERA, (15).

$$M = 3.7 \frac{100-Q}{Q} \quad (D-1.1)$$

Where M=amount of molasses formed in % of beets in white sugar production

Q = purity of the juice

D = sugar content of the beets.

This formula has been found to show good agreement with various analytical data taken over fifteen years. Substituting values of column 13 (Run II) for "explosion" and diffusion juices and taking D equal 17.5 % it can be seen that the molasses formed in the case of "explosion" juice is about 0.53 % per beet lower than in the case of the diffusion juice. These calculations, although approximate, show the importance of even this small increase of purity. It can be seen from Runs II and III that using a shorter steam treatment (1 minute) the amount of pectin remained unchanged (column 7). The lime salts content (column 15) was small in both cases. In both runs the specific conductance of the "explosion" juice was lower than that of the diffusion juice (column 17) showing that the juice prepared by the explosion method contained smaller amounts of mineral constituents. As can be seen from column 10 the rate of filtration of the first carbonation juice was lower for the explosion process. It was interesting to know whether a change from hot to cold preliming would improve the situation. Therefore, in Run III the raw "explosion" juice was prelimed at 40 degrees C. and the corresponding diffusion juice was prelimed at 90 degrees, that is, at the temperature used in all previous experiments. It can be seen that with cold preliming the explosion method gave better filtration rates than the diffusion process. However, on the basis of these experiments no clear answer can be given as to which of the investigated methods gives better filtration rates. Therefore, in future experiments this problem will be given special consideration.

The Figure 7 gives values of $-\lg T$ for thin juices prepared by the explosion and diffusion methods. On the basis of these three runs it seems that neither of these methods offers an advantage as far as the color of the juice is concerned. It can be seen that the difference between the three Runs carried out with three different samples of beet is greater than that due to the use of a different method although each of the three samples was taken from the same lot of beet.

PART II

Preliminary Recovery Tests

a. Experimental Technique

The first determinations of the yield of sugar from the exploded mash were made in the batch explosion equipment described in the Progress Report II. However, instead of using the short explosion nozzle, the bottom of the lining E (see Figure 8) was connected through a 1 inch brass pipe, 22 feet long, with the receiving vessel "B". A known amount (400 g or 200 g) of freshly cut cossettes* was placed in the lining E, and the brass pipe disconnected and removed from the vessel A. The cossettes were then steamed at atmospheric pressure for the desired time and the sugar-containing condensate removed either as usually through the side tube H or directly from the bottom of the lining E. After the cossettes had been steamed the quick opening valve I was closed and the cossettes cooled down by flashing at 20" Hg vacuum. This flashing was done for the purpose of terminating the steaming period at the required moment. After flashing, the lining was removed and the weight of the cossettes determined. The sugar-containing condensate collected during the steaming period was weighed and preserved for the washing of the cake. The lining with cossettes was then introduced again into the vessel A, the brass pipe connected and the cover fixed. In the preliminary recovery tests, compressed air of 110 psig. pressure was used. The explosion was carried out by a sudden opening of the 1 inch quick-opening valve J connecting the air tank D with the explosion vessel A. The cossettes were blown out against baffle F placed in the receiver B. The screen trap, G fixed on the exhaust opening of the receiver, prevented some of the mash particles being carried away. The time necessary to blow the cossettes out into the receiver "B" was less than one second. Even if, instead of air, saturated steam of the same pressure were used, no harmful effect should be expected during such a short time of contact.

After explosion the mash was carefully collected from the receiver and the juice separated in a centrifuge in the manner previously described in Progress Report II (pages 10 and 11). After 15 minutes spinning, the centrifuge was stopped, hot condensate (90 degrees C.) poured on the cake and the centrifuge set in motion again. After approximately 1 minute of spinning at 1800 rpm the operation was repeated with hot water (90 degrees C.). In some experiments the condensate as well as water was applied in several separate portions. After the last portion of water was poured on the cake the centrifuge was allowed to run for 15 minutes at 1800 rpm. After centrifuging, the juice and cake were weighed separately and the purity (Refr.) of the juice and sugar content of the cake

*The average length of cossettes per 100 g was 30-31 mtr. and the average length of cossette was 4 cm.

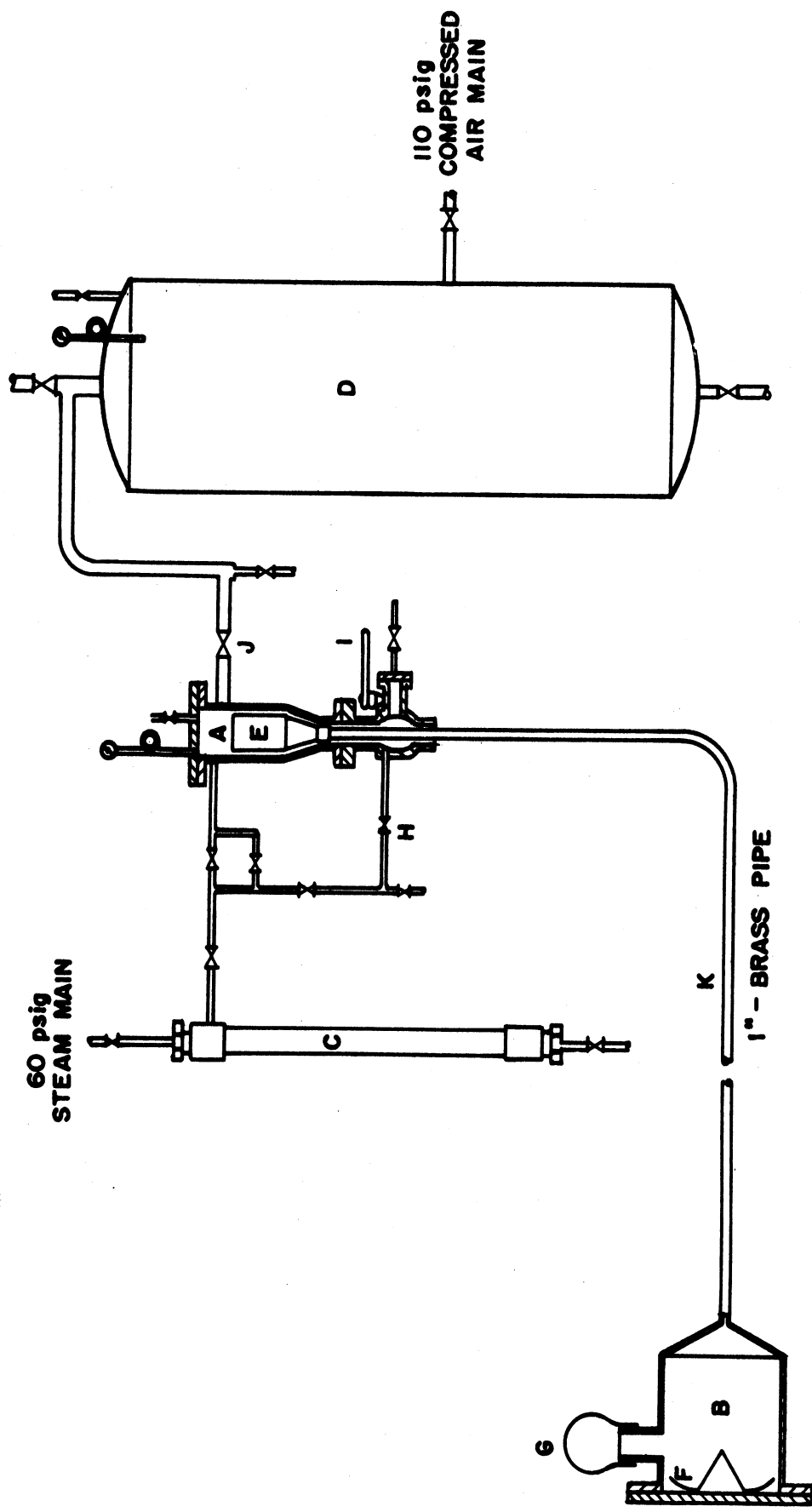


FIGURE 8
EQUIPMENT USED IN THE PRELIMINARY RECOVERY TESTS

determined. In each run of recovery tests a part of a well mixed sample of cossettes was used for explosion and the other for preparation of the hot digestion juice which served as a reference juice in this series experiments.

b. Determination of the Allowable Time of Steaming

It has already been mentioned that the allowable time of steaming depends on the quality of the beet and on the time and conditions of storage. In the explosion experiments carried out in December, 1952, (see Progress Report II, Table V, Run I) a three-minute steaming at atmospheric pressure did not cause any drop of purity of the juice. On the other hand, in experiments carried out two months later with the same beet material a three-minute steaming had proved too long and resulted in a drop of purity of 1.3 (see Table III, Run I of this report). The recovery tests presented in this part of the report were made with beets from California, stored for 6 weeks at +2 to +4 degrees C. and their resistance toward steam treatment had to be determined. Three separate runs were made* in which the cossettes were steamed at atmospheric pressure for one, three and six minutes. In each run the steaming was terminated instantaneously by flashing and the steam treated cossettes passed through an "Enterprise" food chopper. The purity of the juice recovered from the pulp was then compared with that of the hot digestion juice. The results are presented in Table V.

TABLE V

Influence of Time of Steaming on the Purity of the Juice (California Beets)

Time of Steaming at 0 psig.	Hot Digestion Juice			Juice from Steam Treated Cossettes		
	Bx	Pol	Purity (Refr)	Bx	Pol	Purity(Refr)
1 min.	7.22	6.73	93.2	15.70	14.82	94.4
3 min.	"	"	"	12.15 ^{**}	11.41 ^{**}	93.9
6 min.	"	"	"	14.41	13.48	93.6

* The procedure is described in detail in the Progress Report II, page 19.

** The comparatively low value of Bx. and Pol. must have been due to the faulty dehydration of steam.

It can be seen from the table that in this case even a 6 minute steaming did not decrease the purity of the juice as compared with that of hot digestion juice. Therefore, in the following explosion tests six-minute steaming was used in all runs.

c. Results of the Preliminary Recovery Tests

Before the results obtained are presented and discussed some introductory explanations must be given. The main purpose of these experiments was to obtain preliminary data regarding the amount of sugar left in cake, the purity of the juice after washing of cake, and the influence of the shape of the baffle on the yield of sugar. The influence of the amount of wash water used and of the method of washing on the recovery of sugar has also been investigated. As has already been mentioned the experiments were carried out on batch explosion equipment. This equipment although very useful in the previous stage of our work had some disadvantages as recovery tests are concerned. It was batch equipment and therefore its weight and volume was great as compared with the amount of beet used for one test. This resulted in greater heat losses and excessive condensation of the heating steam. Therefore, a corresponding correction must be introduced in the determination of draft. The evaporation due to flashing of cossettes and the evaporation in the centrifuge were also taken into account and corresponding corrections made. The values of draft given in Table VI are therefore the values corrected for excessive condensate, flashing of cossettes and evaporation during spinning. Due to these corrections a certain error in the calculation of the draft was inevitable. The values for the draft are considered correct within the limits of + 3 percent.

The results of six recovery tests are presented in Table VI. In runs I to III the total condensate obtained during the steaming period was used for washing. On the other hand, in runs IV to VI only the theoretical condensate, that is, the total minus the excessive was used for washing of cake. This is why the values for condensate used for washing (column 7) are higher in the first three runs. It can be seen that the purities of the explosion juice after washing (column 5) compare favorably with the purities of digestion juices (column 4) except in run V where the digestion juice has a higher purity. It is possible that this difference, if not due to an error in determination, could have been eliminated by the subsequent purification. It has already been noted (Table IV) that the raw "explosion" juices have a tendency to yield thin juices of higher purity than the diffusion juices.

On the basis of these preliminary experiments it appears that even after washing with condensate and hot water, juices of good purity can be obtained. Obviously this statement applies strictly only to the beets used in the experiments. A certain influence of the quality of the beet may of course be expected. It is interesting to note that in Runs I and II the sugar remaining in cake and the recovery (columns 12 and 13) are approximately

TABLE VI

Preliminary Recovery Tests

1	2	3	4	5	6	7	8	9	10	11	12	13
Run	Experiment	% Sugar in Cossettes	Purity Digestion Juice	Purity Explosion Juice	Draft % by Weight	Washing Condensate g/100 g Beet	Condensate Divided in Portions	Washing Water g/100 g Beet	Water Divided in Portions	Cake % of Beet	Sugar Remaining in Cake % of Beet	Sugar Re-covered %
I	Flat Baffle	16.2	90.1	91.4	102	50.8	1	23.4	1	33.6	1.69	89.6
II	Flat Baffle	16.6	92.1	92.1	122	48.4	3	47	3	37.7	1.88	88.7
III	Concave Baffle with Cone	15.6	92.2	91.6	117	54.5	3	33.4	3	29.8	0.89	94.3
IV	Concave Baffle with Cone	14.33	86.8	86.5	135	21	3	49.3	6	28.2	1.06	92.6
V	Flat Baffle	15.7	91.9	90.6	126	17.8	3	42.6	6	30.9	1.86	88.1
VI	Concave Baffle with Cone	15.4	89.5	89.9	129	19.2	3	45.7	6	30.8	1.2	92.2

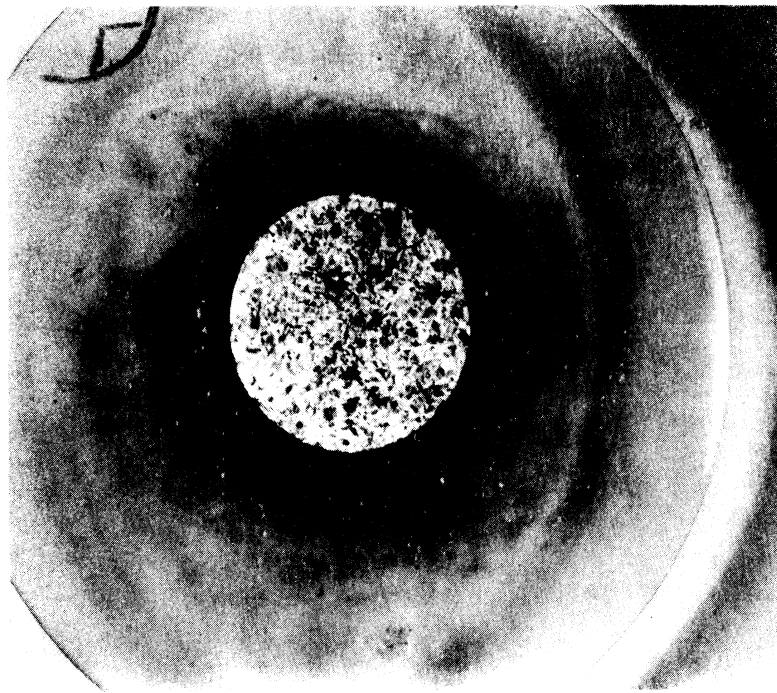


Figure 9. Photograph of Mash (Concave Baffle)

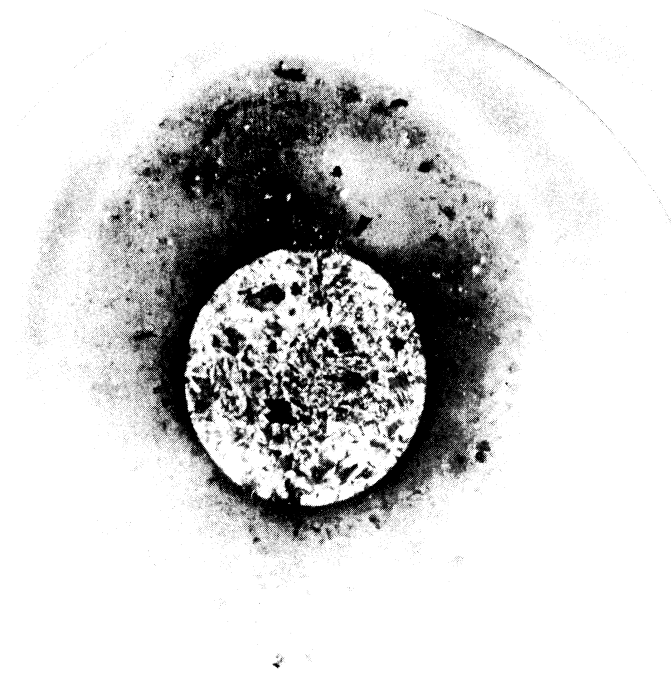


Figure 10. Photograph of Mash (Flat Baffle)

the same although the amount of water used (column 9) was twice as great in Run II and applied in three separate lots. In Run III a concave baffle "F" made of a thick brass screen was used and a distributing cone was placed in the center of the baffle (see "F" Figure 8). With this arrangement a much better disintegration was obtained. The mash was more uniform and contained only a small amount of larger particles (see Figure 9) as compared with the mash obtained with a flat baffle (see Figure 10). This fact was reflected in a smaller amount of sugar left in cake and in a better recovery in Run 3 (columns 12 and 13) where the concave baffle was used. Similarly a better recovery was obtained in Runs IV and VI (concave baffle) than in Run V (flat baffle). On the basis of these experiments it seems that the most important condition of good recovery is a high degree of disintegration of the mash. The amount of water used for washing the cake is probably of secondary importance (see Runs I and II). It is doubtful whether a great advantage can be expected from using the condensate for preliminary washing of the cake. The concentration of the condensate in our experiments approached 10 Bx. It should be expected that in a large scale installation the concentration would be even higher owing to smaller heat losses resulting in a smaller amount of condensing steam. It might be preferable to mix the condensate with the exploded mash prior to centrifuging. This would facilitate the charging of the centrifuge with this almost solid mass. It must be mentioned that some difficulty had been experienced in bringing in line the explosion tube K (Figure 8) with the center of the cone on the baffle. Even a slightly off-center position resulted in a poor distribution of cossettes leaving a part of the baffle ineffective. Obviously this must have had a certain influence on the degree of disintegration, the yield and the reproducibility of results.

The best recovery obtained in the preliminary experiments was 94.3 percent. It is a very encouraging figure considering that only two types of baffles have been investigated and that the optimum conditions of the disintegration have not yet been determined,

As the recovery of sugar depends mostly on the degree of disintegration of the beet an attempt has been made below to list and discuss the factors which according to our observations are of importance in this problem. The order in which the different factors are listed has no connection with their relative importance in the process of disintegration.

1. Quality of the Beet: It has been noticed in many cases that the non-disintegrated particles of the mash contain small pieces of fiber which reinforces the structure of the particle. It should be expected that the more fibrous the beet the more difficult the disintegration.

2. Velocity of Cossettes: The velocity at which the cossettes strike the baffle is obviously a very important factor. It determines the

amount of energy which can be converted into the useful work of disintegration. The velocity of cossettes will depend on the other hand mostly on the pressure gradient of the steam used to accelerate them, on the shape and size of the particles and on dimensions of the nozzle or explosion pipe.

3. Steam Pretreatment: Steam pretreatment of cossettes should be regarded as useful for several reasons. First the structure of the cossettes changes, the cossettes becoming softer and easier to disintegrate. After steaming, the plasmolysis of the cells caused by the action of higher temperature will facilitate the recovery of sugar from the particles not completely disintegrated during the washing of the cake. Preheating of cossettes will prevent the growth of harmful microorganisms. In spite of the above mentioned advantages of steam treatment it must not be regarded as absolutely necessary provided a good disintegration could be obtained by improvement of other process conditions.

4. High Pressure Steam: The action of high pressure steam is complex. The greater the pressure used prior to explosion the greater will be the possibility of rupturing the cells by inflation on "explosion" through sudden evaporation of water in the cells and the greater will be the energy available to accelerate the cossettes. However, excessive treatment with high pressure steam can be very detrimental to the quality of the juice obtained from "exploded" cossettes as shown in Progress Report II. Therefore, some compromise must be made in the pressure selected. It is expected that the optimum pressure will be influenced by the design of the explosion equipment and may vary with different designs.

5. The Baffle: It is already known from the preliminary recovery tests that the type of the baffle plays an important role in disintegration of the cossettes. On the basis of our observations a good baffle should fulfill the following requirements:

- a. It should give every particle a fair chance to strike its bare surface.
- b. Its shape or construction should decrease the deflection of the incoming particles caused by those rebounding from the baffle.
- c. The pulp should be removed from the surface of the baffle as soon as it is formed to decrease the harmful cushion effect.

In our experiments with the flat baffle the comparatively narrow stream of cossettes struck the baffle at a right angle with result that only a small part of the surface area of the baffle was exposed to the stream of material. It is believed that due to the high cushion effect only a small part of cossettes came in direct contact with the bare surface of the baffle and that the majority struck the pulp already formed and not yet removed from the surface of the

baffle. It is also believed that the rebounding particles of the pulp deflected the incoming particles and decreased their velocity. In the above case the efficiency of the process must have been very poor. The use of the concave baffle with distributing cone substantially improved the situation, mainly by increasing the active surface of the baffle and by decreasing the cushion effect.

6. Mass Velocity of Cosettes: It must be expected that the degree of disintegration for a given set of conditions will decrease with increase of the weight of cosettes which strike the baffle in a unit of time. There should be, therefore, a certain optimum mass velocity of cosettes expressed in lb./hr. sq. ft. of the surface area of the baffle. Higher mass velocities would decrease the disintegration and sugar recovery whereas lower mass velocities would decrease the processing capacity without materially improving the recovery.

7. Size of Particles: Obviously the smaller the particle, the easier will be the further disintegration.

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