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REPORT
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COMMITTEE ON THESIS

THE undersigned, acting as a committee of
the Graduate School, have read the accompanying
thesis submitted by Arthur K. Anderson
for the degree of Master of Science.

They approve it as a thesis meeting the require-
ments of the Graduate School of the University of
Minnesota, and recommend that it be accepted in
partial fulfillment of the requirements for the
degree of Master of Science.

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A Study
of the Defecation of Sorghum Juice for Sirup

* * *

A thesis submitted to the Faculty of the
Graduate School of the University of Minnesota

by

Arthur K. Anderson

in partial fulfillment of the requirements
for the degree of Master of Science.

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A Study of the
Defecation of Sorghum Juice for Sirup.

- Introduction -

Historical: The early history (1 & 2) of sorghum in America dates back to Colonial times but it was not until about the year 1860 that it came into importance as an agricultural crop. At that time it was thought that this crop would furnish a large part of the future sugar supply both because of the high sugar content of the plant and because of the varied conditions under which it will thrive. After considerable investigation both by the State and Federal Departments of Agriculture the conclusion was reached that sorghum for sugar was impracticable because of the presence in the juice of relatively large amounts of reducing sugar and gummy material which, it was thought, prevented the crystallization of the sucrose.

With the conclusion that sorghum for sugar was impracticable interest in sorghum decreased. It is only recently that there has been a revival of interest. At

present the interest is from the standpoint of sirup production. Of late there has been a scarcity of maple and cane sirups. There has been much done in the production of corn sirup but this is not a desirable substitute as a table sirup. A good quality of sorghum sirup would undoubtedly become popular as a table sirup.

Table I, showing Production of Sorghum Sirup in Gallons in the Census Years since 1860:

	United States	Minnesota
1860	6 749 123	14 178
1870	16 050 089	38 735
1880	28 444 202	543 369
1890	24 235 219	340 792
1900	16 972 783	157 605
1910	16 532 282	145 934

Sorghum in Minnesota: Table I (2) shows that the growing of sorghum in Minnesota has been on the decline since 1880. With this decrease in production there has been an increase in the value of the product which shows

that the demand is not decreasing. In Minnesota in 1900 the total value of the product was \$56 896.00, and in 1910 it was \$80 718.00 (3). Sirup now retails for seventy-five cents per gallon.

With the change in conditions of farming in Minnesota from small grain raising to diversified farming there is a place for sorghum as a cultivated crop. With these points in view the work on sorghum at this Station has been undertaken.

The production of sorghum sirup in Minnesota is distributed quite generally through the southern half of the state with most of the mills owned by farmers who make enough sirup for themselves and for their neighbors. Methods of Sirup Manufacture: There are three general methods of sirup manufacture used in the state. The simplest method and one used by many farmers who make sirup on a small scale is to press the cane through a small press operated by horse power. The juice is filtered as it comes from the press through straw or hay

and after settling for a short time in barrels or some other form of settling tank is run directly into an open pan evaporator. This consists of a metal bottomed pan placed over a brick fire box. The evaporating pan in some cases is divided into several compartments in such a way that the juice must pass through the series before the desired concentration is reached. This process, of necessity, requires much skimming and long continued evaporation. In some cases lime is used in the settling tank to neutralize some of the acidity and to facilitate settling.

The second method differs from the first in that the juice is heated in a tank, before evaporation, and allowed to settle, the clarified juice being used for evaporation. Lime is used by some. The open pan evaporator is also used in this method.

The third method might be called the factory method. It is confined to half a dozen places in the state, the largest factory having an estimated output

of 30 000 gallons in 1914. At these places steam is used for power, in defecation and in evaporation. The cane is carried by means of a conveyor to the rolls where it is pressed so dry that the crushed cane may be used for fuel without further drying. The juice from the mill is passed through a sieve into a well from which it is pumped to the defecators. These are usually three in number and are elevated to such an extent that they can be drained by gravity to the evaporator. Each defecator holds from two to four hundred gallons of juice. They are square and on the bottom is a series of steam pipes used in heating the juice during defecation. When the defecator is full to about eight inches from the top milk of lime is added, if it has not been added automatically to the well, as the juice comes from the press. The amount of lime added has been enough to bring the juice nearly to neutrality. If the juice is made alkaline it becomes dark brown and makes a dark sirup with an undesirable taste. In some places

litmus has been used to determine when the right amount of lime has been added. After the addition of lime the juice is well stirred and heat is applied until the "cracking point" is reached. This is just below the boiling point and is called the "cracking point" because at this point the skum, which covers the surface, breaks to let steam through. After cracking the juice is allowed to stand for about half an hour to allow the impurities to settle. Floating on the juice is a frame consisting of two boards on edge in such a way that the skum may be removed between them. When this is done the clear juice is drawn off from the surface between these two boards by means of a pipe which goes through the bottom of the defecator and has a loose joint at the base such that by fastening its end to the float it will lower with the float thus always removing the juice from the surface. When all the clear juice has been drawn off the defecator is washed out and made ready for the next lot.

Table II. Summary of Common Methods of Defecation.

Method	Materials Used	When Added	Time Required
Lime Process	$\text{Ca}(\text{OH})_2$ until nearly neutral	Any time before juice is heated to boiling	30 minutes
Lime & Clay Process	A mixture of lime & clay	Added to heated juice	40 minutes
Lime & Tannin Process	$\text{Ca}(\text{OH})_2$ until neutral or slightly alkaline. Tannic substance neutralizes excess of lime.	$\text{Ca}(\text{OH})_2$ to cold juice, let settle draw off clear juice, add tannin, heat, let settle and filter	Several hours
Lime & Phosphoric Acid Process	$\text{Ca}(\text{OH})_2$ until alkaline, $\text{Ca}_2\text{H}_4(\text{PO}_4)_2$ until acid	Same as above. Acid phosphate used instead of tannin.	Several hours
Lime & CO_2	$\text{Ca}(\text{OH})_2$ until alkaline, CO_2 to ppt. lime	Same as above. CO_2 instead of tannin.	Several hours
Heat Alone	Juice simply heated to cracking point and allowed to subside.		30 minutes
Alcohol	90 % alcohol	Juice concentrated to 55% Dry Matter. Equal volume of alcohol added.	Several hours

The juice is passed directly into a steam evaporator of the Porter type where it is evaporated rapidly. It enters at one end of the evaporator and by gravity flows over steam pipes. The incline of the evaporator can be regulated in such a way that the sirup will have the proper consistency by the time it reaches the farther end of the evaporator. The sirup is drained off fast enough to keep the boiling point at the outlet at a temperature ranging from 225° to 230° F. It is passed over cold water pipes to cool quickly. This cooling prevents darkening due to standing in a heated condition in the storage tanks.

Methods of Juice Clarification: From time to time various modifications have been suggested for clarifying the juice preparatory to evaporation. Table II (4) shows a summary of these methods. In these cases the lime is used to neutralize acidity and also aid in the precipitation of foreign substances. Where clay is added its purpose is to hasten and make more complete

the settling due to the lime. Where acids are added their purpose is to remove the lime after it has served its purpose. With tannin, albuminous materials will also be precipitated. With phosphoric acid tri-calcium phosphate is formed which as a precipitate will occlude undesirable substances. In the heat method there is simply a mechanical separation of impurities. Under this head might be classed the method used in many of the small Minnesota mills where open pan evaporation is practiced and the juice without any special treatment is purified by slow boiling and much skimming. From the above descriptions it is seen that methods of manufacture differ in two respects, namely, in the method of defecation and the method of evaporation. The main factor in determining the quality of a sirup is the purity of the juice which is evaporated. The purity of the juice depends upon the efficiency of defecation, so defecation is then the important operation in the manufacture of sorghum sirup. With the hope of learning

more about defecation and of improving the methods at present in use in the state the work in this thesis was undertaken. Much of the field work was done in co-operation with Wessale Brothers of Waconia and Bieder Brothers of Belle Plain. Credit is due them especially for the opportunity which they have given to make this study possible and for the personal assistance which they have so willingly rendered. Credit is also due to all those who have co-operated by furnishing samples of their sirups.

- Object of the Investigation -

The work taken up in this thesis has been the study of the defecation of sorghum juice for sirup with special reference to the methods commonly used in the state. The method of procedure was, first, to find out the changes in chemical composition which the juices undergo during defecation and evaporation; second, to study the composition of sirups made by the different methods; third, to study the quality, (color, taste, clarity, etc.) of sirup made by the different

methods; fourth, to make a special study of the lime method as practiced in the larger mills with reference to finding out what is the best amount of lime to add and a method for determining the acidity of the juice with its application in determining the amount of lime to add; and fifth, to try out a phosphate method.

- Methods of Procedure -

In studying the chemical changes which take place during defecation most of the work was done under actual factory conditions. Various methods were tried out using different amounts of lime. Lots were run with no lime, with lime added to alkalinity, to neutrality, and with the calculated theoretical amount to neutralize the acidity of the juice. A patent phosphate method was tried in which the juice was first boiled for half an hour with acid calcium phosphate and then made neutral with lime. The idea of boiling with the phosphate first being to produce an inversion of sucrose thus preventing crystallization.

In connection with these factory experiments a series of laboratory defecations was run in which juice taken from cane produced on University Farm was used. These experiments were carried on in Erlenmeyer flasks using 500 c.c. of juice for each experiment. Samples were taken of original juices and of the juices after defecation. At the factory samples of sirup made from these juices were obtained. When juices had to be kept for any length of time before being analyzed they were preserved with a one per cent solution of mercuric iodide, using 10 c.c. to 250 c.c. of juice. Where this was used corrections were made in the subsequent calculations.

The study of the sirup was made on forty samples collected from various parts of the state. They were made under various conditions and for study are classified according to the method of defecation. The quality of the sirups was considered from the standpoint of color, clarity, and taste, market value being the

criterion upon which the standards of judging were selected.

In studying the lime method, with special reference to the amount of lime to use, 50 c.c. of the juice from a defecator were titrated with standard tenth normal potassium hydroxide using phenolphthalein as an indicator. The theoretical amount of lime to neutralize this acidity was added. Also other amounts were added to serve as a comparison. Where this was carried out in the factory the opinion of the owner was obtained as to the desirability of the product. A careful study of this was made in the laboratory on a small scale where the conditions could be carefully controlled.

- Analytical Methods -

The following determinations were made on the juices. Acidity was determined with standard tenth normal potassium hydroxide, using phenolphthalein as an indicator. Some difficulty was experienced in determining the end point but with sufficient dilution the

method is accurate to within .5 of a c.c. Results are expressed as the number of c.c. of tenth normal acid in ten grams of dry matter.

Nitrogen was determined by the Kjeldahl Gunning Method. Difficulty was encountered due to foaming during the digestion and it was found that the best method was to heat very slowly at first with 25 c.c. of acid and when the foaming had nearly stopped to add more acid, washing down the neck of the flask if necessary and then boiling rapidly till the digestion was complete.

Dry matter in the juices was determined by drying in a hot water oven for eight hours, the bulk of the water having first been removed on the steam bath. In the sirups a new method for determining dry matter was used in which the amount of moisture present was found by measuring the acetylene generated when a weighed sample of the sirup was treated with calcium carbide in a special apparatus.

Ash was determined on the same sample in which the dry matter was determined in the case of juices. With sirups a special sample was used and in this case the soluble and insoluble ash were determined together with the alkalinity of the soluble ash. In all cases calcium oxide was determined on the ash. The calcium was precipitated as calcium oxalate and determined volumetrically with standard potassium permanganate solution. In a few cases phosphorous pentoxide was determined.

The lead subacetate precipitate was determined. In the case of juices the Official Method (5a) was carried out exactly but where 5 c.c. of sirup was used it was found that the volume of the precipitate was too large to be measured in the tube and also that with the amount of lead subacetate called for there was an incomplete precipitation. It was found that by using $1 \frac{1}{2}$ c.c. of sirup the volume of the precipitate could be read and that there was also an excess of lead. The results

were afterwards calculated to the 5 c.c. basis.

Sugars were determined as sucrose and reducing sugars. The sucrose was determined by the polariscope and the reducing sugars, which are reported as dextrose, by means of the Low's Volumetric Method (5b) modified. The modification being the determination of the unprecipitated copper in a standard Fehling's solution.

- Physical Changes During Defecation -

The physical changes (6) which take place in the juice during defecation are briefly as follows. The juice from the press as it fills the defecator is green, the surface being covered more or less with frothy bubbles. When the heat is turned on these bubbles gradually become larger and break, and in a short time the surface of the juice is covered with a heavy skum. By the end of the process this skum has become from three to four inches thick. As the heating is continued a point is finally reached where the surface of the skum cracks and fine bubbles pour out through the cracks over

onto the surface of the skum. This point is what is called the "cracking point", and it is at this point that the heat should be turned off. If this should be neglected and if the defecator was full it would foam up and run over. If not too full the juice would boil which is undesirable at this stage of the process.

As the juice comes from the press it contains impurities which must be removed. In the first place some sand and clay may be on the cane as it comes from the field. Much of this, of course, gets into the juice. Then there are pieces of leaves and crushed cane which always get into the juice. Besides these mechanical impurities there are substances in the cane itself which are in the juice and should be removed. These substances according to Maxwell (7) are nitrogenous, mucilaginous and carbohydrate bodies. As the juice is heated its specific gravity becomes less. This hastens the settling out of the heavy impurities such as sand and clay. The woody, fibrous particles contain cells

filled with air and as the temperature rises this air expands forming small bubbles which cling to the fiber and float it to the top. If the liquid is boiled this air bubble will detach itself and the fiber will fall back into the main body of the liquid. If well boiled the fiber will settle to the bottom if given enough time. If the impurities in the juice were only dirt and fiber it would be an easy matter to filter the juice and get a clear product but there still remain the "gummy materials". One fortunate thing in a defecator is that the surface liquid has a higher specific gravity and is cooler than the lower liquid. For this reason as the fiber floats it comes to a cooler region and the bubbles of air contract thus holding on to the wood much firmer. The contraction is partly made up for by the increase in specific gravity of the liquid so the fiber continues to rise. By the time the cracking point has been reached all of the fiber has risen to the surface. What happens next is known as the subsiding of the

Table III. Showing Composition of Juice before and after Defecation and of Sirup.

Lab.No.	Description	cc N/10 acid in 10 gm. D. M.	cc lead- subse- tate ppt on D.M. basis	% DM	Results expressed as Dry Matter.									
					Ash	Suc- rose	Dex- trose	Sugar	Solids not Sugars	Solids not Sugar- Ash	CaO	N.	% N. in solids not sugar.	
Phosphate Method														
Series 1.														
3085	Original Juice	17.4	6.9	17.42	5.38						.364	.18		
3086	After adding phosphate	17.0	9.0	18.83	5.14	40.21	53.53	93.74	6.26	1.12	.335	.11	1.78	
3087	After neutralizing		10.3	18.84	4.87	41.72	52.55	94.27	5.73	.86	.525	.06	1.79	
3352	Sirup	8.1	8.5	66.50	4.77	41.92	53.02	94.94	5.06	.29	.436	.16	3.19	
Series 2.														
3174	Original Juice	21.6	9.4	16.03	3.91	58.95	22.12	81.07	18.93	15.02	.449	.29	1.65	
3175	After phosphate def.	7.4	8.5	17.56	3.99	62.39	19.64	82.03	17.97	13.98	.539	.05	.26	
3314	Sirup	21.0	14.4	72.70	4.33	65.81	24.73	90.54	9.46	5.13	.281	.20	2.06	
Theoretical Amount of Lime														
Series 3.														
3174	Original Juice	21.6	9.4	16.03	3.91	58.95	22.12	81.07	18.93	15.02	.449	.29	1.65	
3176	After defecation	11.7	11.8	16.17	3.97	64.23	19.35	83.58	16.42	12.45		.20	1.25	
3315	Sirup	17.0	13.8	72.50	4.48	66.82	23.66	90.48	9.52	5.04	.327	.20	2.06	
Series 4.														
3200	Original Juice	17.4	11.4	18.80	3.24	57.38	34.57	91.95	8.05	4.81	.280	.28	3.65	
3201	Defecated Juice	4.6	6.2	18.46	3.43	59.38	33.31	92.69	7.31	3.88	.414	.06	.79	
3319	Sirup	13.8	12.6	75.60	3.56	57.57	34.55	92.12	7.88	4.32	.403	.23	2.88	
Lime to Neutral (Litmus)														
Series 5.														
3174	Original Juice	21.6	9.4	16.03	3.91	58.95	22.12	81.07	18.93	15.02	.440	.29	1.65	
3177	After Defecation	8.6	10.7	16.32	3.91	63.06	18.63	81.69	18.31	14.40	.530	.20	1.00	
3316	Sirup	15.9	13.9	77.20	4.47	66.40	24.35	90.75	9.25	4.78	.332	.16	1.75	
Lime to Neutral added after heating														
Series 6.														
3174	Original Juice	21.6	9.4	16.03	3.91	58.95	22.12	81.07	18.93	15.02	.449	.29	1.65	
3178	After Defecation	3.0	7.2	15.95	4.11	64.28	18.79	83.07	16.93	12.82	.672	.23	1.42	
3317	Sirup	9.1	17.8	64.70	4.90	68.50	24.68	93.18	6.82	1.92	.592	.23	3.36	

defecator. During the heating, and due to the presence of lime, nitrogenous compounds and mucilaginous materials have been thrown out of solution and for some time after the fiber has risen these bodies slowly rise forming a blanket underneath the fiber which helps to hold it up. It is easy to see that boiling during the process of defecation is undesirable. In the first place boiling would start violent convection currents in the liquid from the top to the bottom of the defecator and in the second place the air bubbles on the fiber would be scattered through the body of the liquid by the convection currents. After subsiding for about half an hour the process is complete. The heavy impurities are at the bottom of the defecator and the lighter ones are in the skum at the surface. A clear juice is left between which should be a light amber in color. This is drawn off and passed into the evaporator.

- Chemical Changes During Defecation -

Factory Experiments: Table III shows the analyses of

six series of defecations showing the composition of the original juice, the juice after defecation and of the sirup resulting from the juice. In the case of one phosphate defecation an analysis is given of the juice after adding the acid phosphate and boiling for thirty minutes. In the phosphate method calcium acid phosphate was added, 137 gms. to 80 gallons of juice. This amount was equivalent to that found in a commercial phosphate advocated for this purpose. The use of five pounds of the commercial phosphate to 1000 gallons of juice was recommended. An analysis showed it to contain 78 per cent calcium acid phosphate. After boiling for thirty minutes lime was added until the juice was neutral to litmus.

In the experiments where the theoretical amount of lime was added 50 c.c. of the juice was titrated with tenth normal potassium hydroxide using phenolphthalein as an indicator. From the amount of potassium hydroxide used the amount of calcium oxide necessary to neutralize

the acidity in the volume of juice in the defecator was obtained and this amount was added.

In the last cases lime was added until the juice was what the manufacturer considered neutral to litmus. It was still slightly acid.

In series #2 and #3 the lime had been calculated as calcium oxide instead of calcium hydroxide due to a misunderstanding of the analysis which came with the lime. So there is really only 75 per cent of the theoretical amount added. This accounts for the high acidity after defecation. In the other sample the true theoretical amount is added.

A closer study of the acidity as related to lime added will be made later. At this point it is of interest to note that the acidity of the sirup is in all cases higher than that of the defecated juice. It is also interesting to note that it is lower than in the original juice.

According to Maxwell (7) the material precipitated

from sorghum juice by alcohol is composed largely of mucilagenous materials. We may consider that in ordinary defecation with heat and lime much of this material remains in solution. According to O'Sullivan (8) the gums are complex compounds which on hydrolysis give rise to sugars and complex acids of high molecular weight. This being the case the increase in acidity could be accounted for on the theory that during the high temperature of evaporation the gums are hydrolyzed liberating the free acid. This theory would also account for some of the increase in dextrose after evaporation.

Lamy (9) gives the following figures for the amounts of calcium oxide dissolved by 1000 gms. of a 10 per cent sugar solution at various temperatures.

At °C	0	15	30	50	70	100
Gms. CaO in solution	25.0	21.5	12.0	5.3	2.3	1.55

If this calcium oxide is not simply in physical solution but partly in chemical combination in the form of saccharates it may be that with the high temperature

of evaporation some of the calcium is split off from the sugar and deposits on the bottom of the evaporator. It is a known fact that at this stage of the process there is actually a deposition of lime. The decrease in lime content of the sirup as compared with the juice also suggests the possibility of this theory. As long as the lime was present in solution it would react alkaline to the indicator, upon its removal the acidity of the sirup would rise.

The darkening of the sirup during evaporation indicates an oxidation. The oxidation of glucose produces acids. This would also contribute to the increase in acidity.

The volume of the lead subacetate precipitate was determined with the hope that it would serve as an indication as to the completeness of defecation. The results, however, are not as consistent as was expected. The high value in the case of sirups is undoubtedly due in part to the high acidity of the sirup.

The ash content increases after lime is added. This would indicate that some of the lime added remains in solution in the juice and sirup. The decrease in ash after defecation in series #1 would be explained by the fact that insoluble phosphates are formed which remove some of the iron and calcium.

At this point it is interesting to notice the change in the percentage of calcium oxide. In all cases the calcium oxide content increases after defecation and decreases again in the sirup. The increase in the defecated juice would seem perfectly natural. The decrease in the sirup would be accounted for by the lower solubility of calcium salts in sugar solution at a high temperature, (9).

Another explanation is that in the juice the calcium is present as acid salts which are soluble and which at the high temperature of evaporation and the higher concentration change over to the normal salts and are precipitated.

In studying the sugar content the surprising thing is that in all cases the sucrose content increases after defecation. According to Maxwell (7) solutions clarified by lead contain nitrogenous compounds which affect polarized light rotating it to the left. This being the case the percentages of sucrose on original juices are too low. The increase in sucrose in defecated juices would be accounted for by the removal of these levogyrous bodies by the heat and lime during defecation.

Maxwell's work shows the following results on a sirup clarified with lead, with phospho-tungstic acid, and with alcohol. The following are the polariscope readings:

Solution clarified by lead	64°.99
After treatment with $H_{11}PW_{10}O_{38}$	67°.37
After treatment with $H_{11}PW_{10}O_{38}$ & alcohol	72°.93

Maxwell accounts for the increase in reading after adding alcohol as due to the reduction of the levorotatory power of levulose in alcoholic solution. He says,

"the body, precipitated by alcohol, is in appearance dextrinoid, and the logical supposition would be that the dextrogyrous property of the solution would be diminished, which in fact is not the case".

From the uniform decrease in reducing sugar after defecation it appears that some reducing substance must be removed. The alcohol precipitate of Maxwell may contain some of the simpler dextrans which reduce Fehling's solution.

The decrease in dextrose after defecation would be explained by the removal of dextrin like bodies which reduce Fehling's solution.

The increase in reducing sugars may be explained as being due to the hydrolysis of sucrose or as pointed out in connection with acidity to the hydrolysis of mucilagenous materials.

The increase of total sugar is of course due to removal of impurities during defecation and evaporation.

In all cases the percentage of nitrogen decreases

after defecation. This is especially noticeable in the phosphate method. This seems natural in that if the nitrogen is present as albuminous bodies it would be coagulated and thus removed after defecation. The marked decrease in the case of the phosphate method would be due to the clearing effect of the phosphate precipitate. Why the sirup should have a higher nitrogen content is harder to explain. In Series #s 2, 3, 5, and 6, the same original juice was used. The cane from which this juice came had been cut for some time before pressing. As will be noted in connection with laboratory defecation it appears that on standing the nitrogen changes its form. It changes to a form which is not removed by defecation except where a phosphate and lime are used. The protein is evidently changed to simpler molecules, likely peptones. In the next column is found the percent of solids not sugar. In each of these samples there is a big decrease in the sirup. In the next column is given the percent of nitrogen in the

Table IV. Showing Results of Laboratory Defecation. Results expressed as % of Dry Matter.

Lab.No.	Description	cc N/10 acid in 10g. DM	cc lead subacet- ate ppt on bas- is DM	% DM	Ash	Sucrose	Dextrose	Sugar	Nitro- gen	CaO
3205	Original Juice	23.0	12.9	15.56	5.80	31.43	51.99	83.42	.44	.587
3206	Phosphate Method. After boiling 30 m. with phos- phate	26.9	14.2	20.05	5.75	33.44	49.55	83.26	.44	.464
3207	Phosphate Method. After adding lime to theory	7.2	12.1	19.07	5.52	34.40	40.49	73.50	.42	.616
3208	Heat Method. Boiled for 30 minutes	22.6	14.0	17.50	5.59	31.52	50.06	81.78	.44	.344
3209	Lime to theory. Heated first	7.7	8.7	16.02	5.65	32.03	48.57	80.59	.45	.596
3210	Lime to half theory. heated first.	15.7	10.8	15.73	5.78	32.20	51.05	80.11	.44	.607
3211	Lime to half theory	14.5	11.4	14.81	5.74	33.02	52.81	85.83	.44	.585
3212	Lime to theory	7.2	10.7	14.91	5.83	31.93	51.45	83.37	.40	.594
3213	Lime to Neutral	neutral	8.8	15.34	6.52	32.21	48.83	81.03	.41	1.068

solids not sugar. In all cases the percent of nitrogen in the solids not sugar increases in the sirup over that in the defecated juice.

During defecation if the nitrogen is in the form of protein coagulated by heat that portion of it will be at once removed. At this point there is not a very marked decrease of solids not sugar. During evaporation the solids not sugars which disappear are mainly non-nitrogenous bodies thus bringing the percent of nitrogen relatively higher in the sirup.

In another column is found the percent of solids not sugar and not ash. In all cases there is a decrease after defecation and after evaporation. This shows the decrease in organic matter not sugar and it shows that the decrease comes mainly during evaporation.

Laboratory Experiments: Table IV shows the results of a series of laboratory defecations. Juice was obtained from cane which was cut and stored for a few weeks before pressing. In this respect the juice is not as

representative of factory conditions as it might be. Several methods of defecation were tried out as indicated in the table. In each experiment 500 c.c. of juice were used. In the phosphate method calcium acid phosphate was added in proportion to the amount recommended. In this case lime was not added to neutrality but the theoretical amount to neutralize both the acid phosphate and the juice was added.

In the heat method the juice was boiled for thirty minutes to observe the results on the inversion of sucrose.

In the rest of the experiments different amounts of lime were added and at different times. Lime to one half theory, to theory, and to neutral was added. In these experiments the theoretical amount of lime was figured on the bases of an analysis made of the lime. This analysis was a determination of the alkalinity of the lime by dissolving in standard acid and titrating the excess with standard potassium hydroxide. In the

case of adding lime to neutral, lime was added until the juice gave a slight alkaline reaction. The amount of lime which it took to cause alkalinity was determined by taking a known amount of lime, adding water to make a suspension and then adding small amounts of this until the juice was alkaline. The excess was then evaporated to dryness and weighed. The difference in weight being the amount used by the juice.

Following is a statement showing the amount of lime required according to a theoretical calculation and also the amount actually required to produce a slight alkalinity to phenolphthalein.

Sample #1 - Calculated theoretical amount of lime to neutralize acidity, .73 gms.; actual amount required 1.60 gms.

Sample #2 - Calculated theoretical amount of lime to neutralize acidity, .88 gms.; actual amount required 1.99 gms.

In both cases the actual amount is about 2.2 times the

theoretical amount.

Notes were taken on the color of the juices after defecation. Where no lime was added the color was of course very light. Where lime was added to alkalinity the juice was a very dark brown color. In cases where lime was added to one half theory the juice remained light colored. With lime added to theory the juice seemed to be just at the point where it had started to turn dark. Where lime was added after heating to boiling the skum instead of rising sank to a large extent. This shows the undesirability of adding lime after the separation of the skum has taken place.

A study of the acidity in table IV shows just what would be expected except that it would naturally be expected that the juice would be neutral after adding the theoretical amount of lime. The increase in acidity in 3206 is due to the acid phosphate. 3207 and 3212 check. 3209 is higher than 3212 because the lime was added after heating. The same thing is noticed in 3210 as compared

with 3211. This would indicate a higher efficiency for the lime added before heat is applied to a defecator. The neutralizing effect of the lime added is almost directly proportional to the amount of lime added up to the theoretical amount. Why it should take over once again as much lime to neutralize about one third the original acidity is a difficult question to answer. It is likely that at this point the lime is combining with the sugar or some other substance. A change of some kind is indicated in the darkening of the juice before alkalinity is reached.

The lead subacetate precipitate shows a decrease as the lime increases, the maximum being reached where lime has been added to theory.

The ash determinations show very little except in the case of 3213 where an increase of ash would be expected. The calcium oxide column shows similar results to what were found in table III.

With regard to the sugars there is the same general

Table V. Composition of Defecator Residues.

Laboratory No.54, Theoretical Lime Used		Laboratory No.3204, Factory Method
% Dry Matter	28.74	25.02
% Ash of D. M.	13.84	5.62
% Sucrose of D. M.		20.86
% Dextrose of D. M.		4.76
% Sugar of D. M.		25.62
% Nitrogen of D. M.	2.46	3.14
% CaO of D. M.	2.63	.61
% CaO in ash	19.00	10.91
% P ₂ O ₅ of D. M.	2.03	.81
% P ₂ O ₅ in ash	14.65	14.33

result as in table III. The results here would indicate that there is no inversion of sucrose on boiling with acid phosphate. Sample 3208 where the juice is boiled for thirty minutes alone shows the lowest percentage of sucrose of any defecated sample. This indicates some inversion.

The nitrogen column shows what was pointed out in connection with table III, namely, that where cane is kept for some time a change in the character of the nitrogen takes place putting it in a form which is not removed by the process of defecation.

Defecator Residue: In the factory study it was noticed that there was a large amount of residue after the juice was removed from the defecator. An analysis of this residue was made from two defecators to serve as a basis for considering the advisability of using a filter press to recover the juice which is at present lost.

Table V shows the composition of defecator residues. This shows that there is at present a considerable loss

of sugar, a great part of which could be saved by the use of a filter press. However, it is a question whether the increase in yield of sirup would justify the added expense.

In an abstract (10) from an article in a Russian Journal the advisability of using defecation residues as a fertilizer is discussed. The author concludes that the use of defecation residues from sugar beet juices increases the yield of beets on heavy soils and enhances the action of superphosphate and nitrate fertilizers.

Another article, (11), also suggests the idea of utilizing these residues as a fertilizer. The use of sorghum juice residues for hog feed has been practiced by some farmers. In the use of the residue for a fertilizer it would have to be dried. This would require considerable equipment and would also require much heat. From the analysis it appears that these residues are high in sugar and nitrogen. From the high sugar and

Table VI. Analysis of Sirups.

Lab.No.	Source	Method of Defecation & Remarks	Dry Matter	Lead sub-acetate ppt. 5cc sample. Original Sirup	Acidity N/10 acid 10 gm. sirup	% Ash Orig. Sir.	% Sol. Ash Orig. Sir.	Sol. Alk. cc N/10 acid 5gr. sample. Original Sir.	% Insol. ash of total sirup	% N. of Total	% CaO of Total	% Sucrose of Total	% Dex-trose of Total
3314	Wessale Bros., Waconia	Phosphate Method	72.7	10.5	15.3	3.15	2.32	5.46	.832	.147	.204	47.86	17.71
3315	Wessale Bros., Waconia	Theoretical Amount of lime added	72.5	10.0	12.3	3.25	2.40	6.94	.840	.145	.245	48.48	17.15
3316	Wessale Bros., Waconia	Lime added until neutral-slightly crystallized	77.2	10.7	12.3	3.45	2.55	7.41	.900	.126	.257	51.26	18.80
3317	Wessale Bros., Waconia	Lime added until nearly neutral after heating	64.7	11.5	5.9	3.17	2.24	6.80	.932	.148	.383	44.32	15.97
3318	BiederBros. Belle Plaine	Factory Method - Lime	76.8	9.8	9.2	3.04	2.18	6.41	.854	.185	.303	36.24	33.65
3319	BiederBros. Belle Plaine	Theoretical Amount of lime added-very slightly crystallized	75.6	9.5	10.4	2.69	2.04	6.24	.648	.172	.305	43.52	26.11
3320	BiederBros. Belle Plaine	Factory Method-Lime-Crystallized	73.8	11.3	13.9	3.23	2.58	7.93	.656	.193	.097	47.30	17.01
3321	W.D.Riley, Amboy	Heat only	78.7	11.3	15.3	4.76	3.92	12.92	.836	.131	.274	37.18	30.73
3322	W.D.Riley, Amboy	Heat only - 1912 - crystallized	82.5	9.0	9.4	1.85	1.48	6.28	.374	.121	.086	55.48	24.99
3323	Loveall, Winnebago	No lime used	73.1	9.8	13.1	2.65	2.03	4.85	.618	.100	.114	35.64	31.20
3324	Thew, Wemon Center	No lime used	73.6	13.3	17.8	4.21	3.30	10.75	.910	.186	.090	39.14	27.43
3325	Jenks, Windom	No lime used	72.1	11.7	11.8	2.49	1.75	5.64	.734	.102	.199	40.06	26.43
3326	Barkley, Pipestone	No lime used	78.8	11.2	15.8	3.39	2.74	6.59	.652	.053	.147	30.44	40.77
3327	A. Roenhildt, LeSueurCenter	No lime used	76.3	10.3	15.3	3.45	2.73	6.06	.724	.125	.143	40.18	28.81
3328	R. Roenhildt, LeSueurCenter	No lime used	70.1	12.0	12.8	2.23	1.68	4.72	.554	.092	.147	37.20	30.62
3329	AugRoenhildt, LeSueurCenter	No lime used - crystallized	81.0	11.8	15.0	3.64	2.84	8.35	.796	.121	.148	38.36	33.65
3330	JohnTraxler, LeSueurCenter	No lime used	76.1	9.7	12.0	2.88	2.17	6.29	.714	.109	.210	41.08	29.04
3331	F. Roenhildt, LeSueurCenter	No lime used - crystallized	78.1	9.7	15.2	3.10	2.39	6.37	.704	.154	.116	43.58	27.83
3332	Ed. Peterson, St Peter	No lime used	76.1	10.7	13.7	2.99	2.20	7.13	.786	.134	.216	43.28	26.64
3333	BiederBros. Belle Plaine	Lime - crystallized	78.7	10.3	8.9	2.82	2.08	5.81	.742	.172	.299	50.68	22.57
3334	Jones, Howard Lake	No lime used	76.9	11.0	15.0	2.66	2.14	6.14	.516	.136	.122	34.20	36.43
3335	Zum Berge, Bongard	Lime - Neutral to litmus - crystallized	80.0	10.0	8.4	5.13	2.19	10.90	.942	.213	.423	44.58	26.41
3336	Jones, Howard Lake	No lime - slightly fermented	78.8	10.7	16.3	3.13	2.56	7.97	.568	.140	.143	33.46	38.07
3337	Jones, Howard Lake	No lime - ("best") - mouldy	71.7	8.0	12.3	1.82	1.20	3.82	.622	.130	.144	46.24	21.53
3338	Cox, Long Lake	No lime - ("best")	76.1	8.7	11.8	2.45	1.80	5.21	.646	.124	.155	39.76	32.66
3339	Cox, Long Lake	No lime - evaporated slowly	75.3	11.0	19.2					.183	.089	25.66	42.36
3340	JohnsonBros. LesterPrairie	Lime used - crystallized	79.9	9.3	8.4	3.58	2.75	6.15	.828	.059	.321	43.78	31.05
3341	JohnsonBros. LesterPrairie	Lime in excess - crystallized	80.3	8.8	7.4	3.02	2.16	4.93	.862	.183	.327	42.74	32.66
3342	J. Smith, Isanti	Bicarbonate of soda	70.0	11.0	12.3	2.03	1.38	4.07	.652	.141	.068	35.34	30.87
3343	BiederBros. Belle Plaine	Lime used -from light soil-crystallized	79.4	10.5	11.8	4.12	3.34	10.02	.778	.187	.130	45.56	25.92
3344	BiederBros. Belle Plaine	Lime used -from heavy soil-crystallized	77.3	9.3	10.0	4.15	3.30	9.35	.852	.170	.224	40.92	28.32
3345	Anderson, Gibbon	Lime used - pan evaporation	77.2	9.7	9.9	2.73	2.23	6.00	.502	.109	.133	37.70	33.65
3346	A. Roenhildt, LeSueurCenter	Lime used	77.7	9.8	13.0	2.86	2.12	5.98	.738	.095	.078	35.06	36.07
3347	Wessale Bros., Waconia	Excess of lime	68.8	9.7	4.9	3.50	2.46	7.19	1.040	.108	.407	40.74	23.39
3348	Wessale Bros., Waconia	Slight excess of lime	75.7	10.2	8.4	3.85	2.29	6.74	1.562	.120	.704	33.86	34.28
3349	Wessale Bros., Waconia	Lime used - left acid	69.9	9.5	8.9	3.52	2.78	8.51	.740	.105	.152	38.34	28.09
3350	Wessale Bros., Waconia	Neutral to litmus	75.5	11.0	6.7	2.88	2.01	5.88	.876	.120	.326	30.86	39.54
3351	Wessale Bros., Waconia	Lime used	75.8	10.3	8.4	3.44	2.61	7.56	.828	.056	.249	41.22	28.81
3352x	Wessale Bros., Waconia	Phosphate Method	66.7	7.8	5.9	3.09	2.25	6.33	.840	.127	.312	27.40	35.61
3352	Wessale Bros., Waconia	Phosphate Method	66.5	8.5	5.4	3.17	2.39	6.39	.782	.107	.290	27.88	35.26
3353	Wessale Bros., Waconia	Lime used to nearly neutral	71.2	7.0	6.4	2.66	1.91	5.38	.746	.103	.302	28.82	38.75
3354	Wessale Bros., Waconia	No lime used	73.0	10.7	14.8	2.90	2.32	5.80	.578	.069	.107	26.04	40.39

(Sugar determinations made by David O. Spriestersbach.)

Table VII. Composition of Sirups from Various Methods of Defecation.

Method	No. Sample	% D.M.	cc lead subacetate ppt. in 5cc DM basis	cc N/10 acid in 10 gms. D.M.	% Ash of D.M.	% sol. ash of D.M.	sol. alk. in cc of N/10 acid in 5 gms. D.M.	ratio of alk. to total ash	ratio of alk. to sol. ash	% insol. ash of D.M.	% insol. ash of total ash	% CaO of D.M.	% CaO of Ash	% sucrose of D. M.	% dextrose of D.M.	% sugar of D.M.	% solids not sugar	% nitrogen of D. M.	% N. in solids not sugar.
No Lime	17	76.2	13.3	18.8	3.90	3.02	8.91	2.99	2.98	.881	22.44	.203	5.35	51.01	40.69	91.70	8.30	.161	1.94
Lime used	11	76.7	12.4	13.6	4.25	3.38	9.65	2.190	2.84	.998	22.98	.265	6.33	55.50	35.98	91.48	8.52	.173	2.05
Lime to Theory	2	74.0	13.2	15.4	4.02	3.01	8.92	2.193	2.98	1.008	24.96	.365	10.70	62.20	29.10	91.30	8.70	.214	2.46
Lime to Neutral	4	72.9	13.7	9.4	4.72	2.89	9.87	2.083	3.44	1.206	26.54	.491	10.85	51.39	41.12	92.51	7.49	.190	2.67
Lime in Excess	5	74.9	12.9	9.1	4.65	3.10	8.50	1.812	2.71	1.549	32.98	.643	13.55	52.43	39.98	92.41	7.59	.181	2.39
Phosphate	3	68.6	13.0	12.7	4.58	3.39	8.87	1.932	2.61	1.193	26.09	.395	9.39	49.60	43.87	93.47	6.53	.095	1.45
Bicarbonate of soda	1	70.0	15.7	17.6	2.90	1.97	5.82	2.020	2.95	.932	32.12	.097	3.35	50.48	44.09	94.57	5.43	.201	3.70
Crystallized	12	78.7	12.7	13.9	4.33	3.15	9.52	2.234	3.08	.963	22.60	.290	6.94	58.12	33.29	91.41	8.59	.199	2.32
Very badly crystallized	1	82.5	10.9	11.4	2.24	1.79	7.61	3.394	4.24	.453	20.21	.104	4.65	67.26	30.28	97.54	2.46	.147	5.97

nitrogen content it appears that these residues have a high feeding value and their use as a hog feed could be made profitable.

- Analysis of Commercial Sirups -

Table VI shows the analyses of forty samples of sirup obtained from various parts of the state.

Table VII shows the averages of all samples made by the indicated method of defecation. The first column gives the number of samples from which the averages are taken. Under the head of "Lime Used" are included all of the samples where lime was used in defecation excepting those where special amounts of lime were used. These are given under special heads. There was one sample where bi-carbonate of soda was used as a neutralizing agent in defecation. Under the head of "Crystallized" are included all samples which showed crystallization regardless of the method used in defecation. The last sample is one made in 1912 and is very badly crystallized. It seems abnormal in many respects. This may be accounted for not so much in the fact that it is

crystallized as to the probability that much of the uncrystallized portion of the original sirup has been used leaving the remaining portion abnormal.

There is nothing exceptional with regard to the dry matter content of the different classes of sirup except that the crystallized samples contain a higher percentage than the others. This is especially true of the badly crystallized sample. It is evident that crystallization is dependent to a large extent on the concentration.

The lead subacetate precipitate is lower in the samples where lime was used than where it was not. The sample where sodium bicarbonate was used shows the largest precipitate. This is due in part to the high acidity.

The acidity decreases with the amount of lime used which is to be expected. The high acidity of "lime to neutral" is due in part to the fact that this series includes the sample mentioned above as having only three

fourths of the theoretical amount. A neutral juice gives an acidity equal to about one c.c. tenth normal acid per gram of dry matter.

The ash content increases as lime is added. The sodium bicarbonate sample is very low in ash as is also the one which is badly crystallized.

There is nothing significant in the percentage of soluble ash and its alkalinity. The increase in soluble ash where lime was used is due to the increase in total ash. The ratio of the soluble ash to total ash shows a definite relation to the amount of lime added. The ratio decreases with the addition of lime.

There is a direct relation between the percentage of insoluble ash and the amount of lime added, the former increasing as the latter is added. The same relationship holds in the case of the insoluble ash calculated on the basis of the total ash and with the calcium oxide calculated on the same basis as well as on the total dry matter basis. The percentage of calcium

oxide is very low where sodium bicarbonate is used.

In a study of the ash it was hoped that a relationship would be found between it and the crystallization. In speaking of molasses Mackenzie,(9), states that formally "the presence of invert sugar was supposed to prevent the formation of crystals, but, on the contrary, it is now known that it has no such effect. Gunning has shown that while concentrated alcohol does not dissolve sugar, an alcoholic solution of organic salts of potassium does dissolve sugar, forming a thick uncrystallizable sirup, which is very easily soluble in alcohol, methyl alcohol or water, but from which sugar is not again directly obtainable. This sirup consists of compounds of potassium saccharate with potassium salts of organic acids found in molasses".

Applying this theory to the crystallization of sorghum sirup it would be expected that the percentage of soluble ash would be small. This, however, is not the case.

In studying the sugar content of the sirups it is at once seen that the sucrose content is less where no lime is used and the dextrose is higher. This of course is due to inversion with the stronger acid. In most of the samples where no lime was used open pan evaporation and slow boiling was practiced. This would also explain the inversion.

The phosphate method shows the lowest amount of sucrose of any. This is due very likely to boiling with the acid phosphate. The high percentage of sucrose and low amount of dextrose in the crystallized samples of course seems normal. This explains in part why these samples crystallized.

The percentage of solids not sugar being less where no lime was added can be accounted for by the fact that in most of these cases slow open pan evaporation, with much skimming was practiced. The long boiling would naturally remove more impurities than rapid evaporation. The phosphate method shows a higher

Table VIII. Showing Quality of Sirups

No. of Sample	Description	Color	In Order of		Crystal- lization	Taste	
			Clarity Light	Dark			
14	Phosphate	37	*50	26	*20	**52	
15	Lime to Theory	24	*49	**52x	*44	**52x	
16	Lime to Neutral	*20	34	*47	29	*19	Best
17	Lime to Neutral	*44	37	**52	*35	34	
18	Lime	*43	36	23	*19	30	
19	Lime to theory	34	*18	*48	31	*14	
20	Lime	*16	38	*53	*16	*53	
21	No lime	*51	24	28	*40	*47	
22	No lime	22	54	*46	*33	*50	
23	No lime	38	32	21	*43	*49	
24	No lime	31		*45	*41		
25	No lime	29		39	22	26	
26	No lime	32		25		32	
27	No lime	*40		30		38	Good
28	No lime	*33		27		*51	
29	No lime	**14		**14		54	
30	No lime	*15		*15		*18	
31	No lime	*49		*17		39	
32	No lime	28		42		*40	
33	Lime	36				*16	
34	No lime	*19				*43	
35	Lime to neutral	54				*33	
36	No lime	21					
37	No lime	27				*41	
38	No lime	*50				*44	
39	No lime	*18				24	
40	Lime	*35				23	Medium
41	Lime in excess	*46				*17	
42	Bicarbonate of soda	42				*15	
43	Lime	30				21	
44	Lime	*17				*48	
45	Lime	25				28	
46	Lime	*53					
47	Lime in excess	39				*46	
48	Lime in excess	*45				*45	Poor
49	Lime	*47				39	
50	Lime to neutral	*48				22	
51	Lime	26				29	
52	Phosphate	23				31	
52x	Phosphate	*41				*35	
53	Lime to neutral	**52x				*20	Bad
54	No lime	**52					
						25	
						30	
						27	
						42	

* Lime used in defecation.

** Phosphate method in defecation.

purity than the samples where lime was used.

The percentage of nitrogen was less where no lime was used, which can be explained on the blow evaporation basis. In the other samples the nitrogen decreases with the amount of lime. In the phosphate method the nitrogen content is less than in any of the others.

On the basis of solids not sugar there is considerable uniformity, however, there is a decrease in the phosphate method.

- Quality of Sirups -

In studying the quality of the sirups they were judged on the basis of color, clarity, and taste. Table VIII gives the number of each sample with a description of how it was defecated. In judging color the samples were arranged in order of their color by reflected light, the lightest being placed first. Those in which lime was used are starred. Those made by the phosphate method are double starred. Two of the samples in which the phosphate was used are the darkest. The

lightest samples were made without lime. A study of the acidity shows that these samples in which lime was used and which appear at the head of the list are still highly acid while those at the foot of the list are not as acid. The darkness of some samples, for example, No. 23 and No. 26, where lime was not used is due to slow evaporation. The darkness of the phosphate samples is due to lime being added to neutrality and to boiling one half hour in the defecator before evaporation. Sample No. 14 made by the same method, excepting that lime was added to three fourths the theoretical amount, is lighter in color. It can then be concluded that a dark sirup is due to much lime and to slow, long continued evaporation.

In determining the clarity of the sirup test tubes were filled with sirup and the transparency noted. There were two distinct types of sirups, the light and the dark. These two were not comparable so each group was arranged separately according to clarity. In the

light sirups those in which lime was used are the clearest. The relationship is not the same in the case of the dark sirups. Two of the phosphate samples are well toward the top of the list there. Sample No.14, the other phosphate sample, was not made neutral with lime but lime was added to three fourths theory. It appears that clarity varies directly with the amount of lime. Nos. 50 and 47, also No. 48, all have had an excess of lime.

Under the head of "Crystallization" are given all samples which show crystallization. Those which show it the least are placed first. It is noticeable that most of them were made with lime defecation. From this it appears that by lime defecation "gummy materials" are removed which if present prevent crystallization. The presence of lime would also prevent inversion of sucrose. The relatively higher percentage of sucrose would favor crystallization. To prevent crystallization well defecated juices should not be evaporated to too great a

concentration.

In studying taste, the sirups were tasted four times. Each time the poorest were eliminated. Those remaining at the end were considered the best. Among the best only two samples were made without lime. All of the phosphate samples remained in this group. No lime samples were found in the last group. From this it appears that the phosphate and lime methods produce the best tasting sirups.

In judging taste two persons worked independently. They both came to practically the same decision as to which were the ten best samples.

- The Phosphate Method -

From the high rating which has been given the samples of sirup made by phosphate defecation it would appear that this is the most desirable method. If quality alone were to be considered it undoubtedly would be. But one of the big factors is the extra cost of production, both in time and in extra materials and

equipment. In the late bulletins by the United States Department of Agriculture a method in which a phosphate is used is recommended. This method advocates the addition of lime to alkalinity and allowing the juice to settle. After settling the clear juice is run off into another defecator and made slightly acid with an acid phosphate. The juice is then heated and allowed to settle for a second time. The clear juice is then drawn off and evaporated. This method is said to give a very clear and bright product.

The big objection to this method is the time required to complete the process of defecation. It would take two or three hours while the method in use now requires only thirty to forty-five minutes. To use the phosphate method and handle the same volume of juice as is now handled would necessitate the installation of four to six times as many defecators.

The added expense for lime and phosphate would be another objection to this method. As was pointed out,

it requires 2.2 times the theoretical amount of lime to give the juice an alkaline reaction. The phosphate used can be obtained at five cents per pound. This would be used at the rate of five pounds per 1000 gallons of juice. The added expense for lime and phosphate would not be serious although on a large scale it would amount to considerable in a seasons run.

- Lime Method ; Factory Control -

A sirup and molasses dealer, (12), who has handled large quantities of sorghum sirup says that the best quality of sirup should be light in color and mild in flavor.

With this in mind a special study of the lime method was made to determine the correct amount of lime to add to produce this result. Since one of the main objects in adding lime is to neutralize acidity it was thought that the theoretical amount was the proper amount to add. To prove this, experiments were conducted at two factories where a 50 c.c. sample of the juice from each defecator was titrated with tenth normal potassium

Table IX. Showing Uniformity of Decrease of Lead Subacetate Precipitate and Acidity where Lime was added to Theory.

Lab. No.	Description	C.c. lead subacetate ppt. to 5 c.c. D.M. Basis	C.c. tenth normal acid to 10 gm.D.M.
3200	Original Juice	11.4	17.4
3201	Defecated Juice	6.2	4.6
3202	Original Juice	11.6	17.7
3203	Defecated Juice	6.7	4.2
Lime added according to factory method			
3103	Defecated Juice	6.8	10.2
3198	Defecated Juice	12.8	5.9
3199	Defecated Juice	10.2	12.0
3177	Defecated Juice	10.7	8.6
3178	Defecated Juice	7.2	3.0

hydroxide and the amount of lime to correspond to this titration was added. Very satisfactory results were obtained. At one factory the system was put into actual operation for the remainder of the season. At the other factory the work was done on their last days run so an opportunity was not available to give the method an exhaustive trial there. However, the manufacturers said that they considered the juice to be properly limed and expressed their desire to investigate the method further next year. According to these manufacturers one of the main things that they are striving for is to get a uniform product. The work in laboratory defecation shows that the theoretical amount of lime will bring the juice to the point where more lime will cause a marked darkening. In other words the theoretical amount of lime should produce a light sirup.

Table IX shows the results of two samples defecated with the theoretical amount of lime. Notice the uniformity of acidity of the defecated juice. The last

Table X. Showing the amount of quick lime to add where 50 c.c. of juice shows indicated acidity with n/10 K O H

Gals. Juice	Acidity in 50 c.c. of Juice																					
	10cc		12cc		14cc		16cc		18cc		20cc		22cc		24cc		26cc		28cc		30cc	
	#	oz	#	oz	#	oz	#	oz	#	oz	#	oz	#	oz	#	oz	#	oz	#	oz	#	oz
100	0	7.5	0	9	0	10.5	0	12	0	13.5	0	15	1	.5	1	2	1	3.5	1	5	1	6.5
200	0	15	1	2	1	4.5	1	8	1	11	1	14	2	1	2	4	2	7	2	10	2	13
300	1	6.5	1	11	1	15	2	4	2	8.5	2	13	3	1.5	3	6	3	10.5	3	15	4	3.5
400	1	14	2	4	2	9	3	0	3	6	3	12	4	2	4	8	4	14	5	4	5	10
500	2	5.5	2	13	3	3.5	3	12	4	2.5	4	11	5	2.5	5	10	6	1.5	6	9	7	.5

part of the table shows the lack of uniformity where the lime was added by the manufacturer in amounts determined by his judgement or by using litmus as an indicator. This shows the need for factory control.

Table X shows the theoretical amount of quick lime to add to known quantities of juice where a 50 c.c. sample requires the indicated number of c.c. of tenth normal potassium hydroxide to neutralize it using phenolphthalein as an indicator. Where calcium hydroxide is used approximately one third should be added to the indicated quantities. Or to use the same table the titration can be considered as one third more and the figures read direct from the table. For example, if the defecator contained 400 gallons of juice and if the titration showed 18 c.c. of tenth normal potassium hydroxide solution used, instead of reading the lime as 3# 6 oz., it would be very simple to run over to the column headed 24 c.c. and read the lime required as 4# 8 oz. The indicated amount of lime should

be weighed out, slaked and added as mild of lime.

- Summary and Conclusions -

1. Acidity decreases during defecation and increases during evaporation, with the lime and phosphate methods. The acidity varies inversely with the amount of lime used.
2. The volume of the lead subacetate precipitate is not a good indication of the efficiency of defecation. With high acidity there is a large precipitate.
3. The ash content increases after lime has been added. In the phosphate method there is a decrease in the amount of ash.
4. Calcium oxide increases during defecation and decreases during evaporation.
5. There is an apparent increase in sucrose during defecation due to a decrease in non-sugar solids and to interfering optically active substances present in the original juice.
6. There is an apparent decrease in reducing sugars

during defecation due to the elimination of other reducing substances which are not removed by lead subacetate. The increase during evaporation is due to inversion and to the hydrolysis of mucilagenous bodies.

7. The nitrogen content decreases after defecation. The largest decrease is apparent in the phosphate method.

8. In cane which has stood after being cut the nitrogen is changed to a non-precipitable form which is not removed by defecation.

9. Solids not sugar decrease during defecation and evaporation, the larger decrease being during the latter process.

10. The color of the juice is not darkened materially until more than the theoretical amount of lime has been added.

11. The amount of lime to produce alkalinity is 2.2 times the theoretical amount.

12. Defecator residues are valuable as a hog feed due

to the high carbohydrate and nitrogen content.

13. In sirups there is little variation in the lead subacetate precipitate with different methods of defecation. It is less, however, where lime and phosphate defecation has been used.

14. Acidity in sirups varies inversely with the amount of lime used.

15. In sirups the total ash is higher where lime has been used.

16. The ratio of the alkalinity of the soluble ash to the total ash decreases as lime is added.

17. The percentage of insoluble ash and calcium oxide increases with the lime added.

18. Sucrose increases with the lime used.

19. Dextrose decreases with the lime used.

20. Crystallization is due to a high percentage of dry matter, a relatively high sucrose content and a juice relatively free from "gummy materials".

21. Crystallization is most frequent in samples where

lime was used.

22. The phosphate method gives the darkest colored sirup.

23. The lightest colored sirups were made without lime.

24. Clarity varies with the amount of lime used.

25. The phosphate and lime methods give the best tasting sirups.

26. The phosphate method is not as practicable as the lime method because of the increased cost of production.

27. The theoretical amount of lime gives proper defecation with little darkening of juice.

28. The titration of juice with potassium hydroxide using phenolphthalein as an indicator is an efficient method of factory control.

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Evaporating House - Primitive Type



Settling Tank



Horse Power Mill
Horizontal Rolls



Horse Power Mill
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Open Pan Evaporator



Steam Evaporator



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