A Method for Determination of Thin Juice Purity from Individual Mother Beets

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It has long been recognized that there is a good correlation between sugar content and juice purity in sugar beets. Largely because of this parallelism, and the difficulties involved in the determination of purities of juices from individual mother beets as well, the beet breeder has contented himself with selection for quality on the basis of sugar content alone. Another factor in discouraging the use of beet juice purity as a measure of beet quality is the knowledge that the actual criterion of quality is the purity of the defecated beet juice and not the purity of the raw juice.

Because of the desirability of possession of a method capable of giving the purity of thin juice obtainable from individual mother beets, this subject was investigated at our Research Laboratory and the method to be described was developed.

This paper details the procedure and presents comparative results showing the purities of juices obtained by the new rapid method, applicable to volumes of beet juice as small as 50 ml., and the standard lime and carbon-ation method which requires relatively large volumes of beet juice. Application of this method to experiment station operation, and results obtained in variety test studies, will be presented in another paper by Mr. Ralph Wood, Agronomist, Longmont Experiment Station.

The new method gives the apparent purity of thin juice, agreeing with that obtained by the standard lime defecation and carbonation procedure within the limits of analytical error, is very rapid, requires only a small sample, and is adaptable to mass operations. It involves defecation of the beet juice sample with milk of lime and removal of the lime in two stages with oxalic acid, simulating standard sugar house techniques at first and second carbonations. This procedure may be referred to as oxalation. It has been tested in the laboratory and has demonstrated its ability to produce satisfactory results with proper attention to details of procedure. It fails in the same manner as does standard carbonation procedure. That is, when insufficient lime for defecation of the juice is used, and when pH is not properly controlled at the first and second oxalation steps (equivalent to 1st and 2nd carbonation), an off quality juice may be expected, with accompanying error in juice purity.

Method of Purity Determination

Juice pressed from rasped beet pulp is the starting material. Commonly, plenty of pulp will be available for the test. When individual mother beets are being tested, a special rasp is required, which will provide a quantity of rasped pulp sufficient for the necessary tests. The requirements are generally met by use of a rasp which cuts a 36° sector from the root. This

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will provide approximately 100 grams of pulp from a mother beet weighing] kilogram. Thirteen grams of the pulp are used for the sugar and other tests and the remainder is used for the purity test. This residual pulp is placed in a small piece of duck filter cloth and the juice is quickly pressed, using a Carver laboratory press. If 50 ml. of juice are not obtained, the necessary quantity of water is poured onto the press cake, and the pulp is re-pressed. The writers recognize that this simple procedure will not be satisfactory if the mother beets are of small size, and a process for preparation of ca 100 gms, of suitably finely divided pulp, representative of the whole beet root, will need to be devised.

A 50-ml. sample of press juice is treated at room temperature with 1.1 gm. CaO in the form of a 20 percent milk of lime, mixed thoroughly and allowed to stand about five minutes. More lime may be required if green beets are being tested. Great excess of lime is undesirable because of the dilution of the sample later associated with its removal by oxalic acid.

The limed juice is titrated to 11.2 pH with a practically saturated solution of oxalic acid, using a titrimeter standardized at 11.0 pH, with a standard buffer. Juice quality suffers from overrun in this titration, just as in overcarbonation at the first carbonation station. The quantity of oxalic acid required at this step is ca 18 ml. and any large departure from this quantity shows error in lime addition, oxalic acid concentration or pH at the end point of titration.

After the first titration the mixture at 11.2 pH is heated in a steam or hot water bath to 60°-70° C. and filtered on a 9-cm. Buchner funnel, using a small quantity of celite filter aid. The filtration requires 3-5 minutes, giving a pale straw-colored juice. Off color is indicative of improper manipulations or spoiled beets and such samples are discarded.

The first oxalation filtrate is titrated with the oxalic acid solution to 9.2-9.3 pH, using a titrimeter standardized with a standard buffer at this point of the scale. This second titration generally requires 0.8 to 1.4 ml, of oxalic acid. The juice at 9.2-9.3 pH is heated to 60°-70° C. and may be very readily filtered by gravity, using a small amount of a celite filter aid, and filter paper such as Whatman No. 5.

The sparkling pale yellow filtrate is read directly, without lead acetate treatment, in a 400-mm. tube, and the refractometer dry substance of the juice is read as accurately as possible. The Precision Model Refractometer is recommended, or a dipping type, if suitable provision is made for accurate temperature control. Under these conditions, an error of 0.1° V in the polariscope reading is equivalent to an error of somewhat over 0.1 point in purity, but an error of 0.1 in refractometer reading is equivalent to an error of about 1-1.5 percent in purity. The purity is taken from the proper purity tables, or calculated, employing the refractometer solids as brix and taking the corresponding specific gravity of the solution.

Results

Most of the results to be presented give comparisons of purity determinations on thin juices prepared from large batches of press juice by standard carbonation and by the new oxalation method. To date, only a few tests have been made on individual roots.

Table 1 gives the comparison of apparent purities obtained by the two methods on samples of rasped pulp representing five different varieties grown in 1952.

Table 1.—Thin Juice Apparent Purities—1952 Beets. Thin Juices Prepared by Carbon ation and Oxalation.

Variety No.	Apparent Purity Thin Juice Standard Carbonation	Oxalation
1	88.8	88.1
2	90.7	90.2
3	90.0	90.4
4	92.5	92.2
5	90.1	90.1

In this set of results the sugar content of the carbonated thin juice was determined by polarization of 52.0 gms. of thin juice made to 100 ml.

The beet pulp representing Variety No. 4 was preserved in the deep freeze for about six weeks and then rerun by the oxalation method. The purity shown in the second test was 92.3, showing no change on long storage of the pulp.

Table 2 lists comparative results, obtained by the two methods of preparation, on press juices from, beets of the 1953 season.

Table 2.—Thin Juice Apparent Purities—1953 Beets.

Description of Beets	Apparent Purity Thin Juice Standard Carbonation	Oxalation
Commercial—Brighton	90.8	91.5
Commercial—Longmont	93.1	93.7
Experiment Station—		
Variety No. 1	87.7	88.1
Variety No. 2	90.5	90.9
Variety No. 3	90.1	90.0
Variety No. 4	91.8	91.4
Variety No. 5	90.6	91.8
Variety No. 6	91.0	91.9

The results on 1953 beets suggest a tendency toward higher purity of the juice prepared by oxalation, especially noted in varieties 5 and 6. Previous experience with laboratory carbonation operations on press juice had demonstrated a tendency toward variable results when only 2 percent CaO on beets is used at carbonation. Therefore, three of the above samples were treated with both 2 percent and 3 percent CaO in both methods of thin juice preparation. The results are given in Table 3.

Table 3—Thin Juice Apparent Purities Using 2 and 3 Percent CaO on Beets.

Description of Beets	% CaO on Beets	Apparent Purity Thin Juice Standard Carb.	Oxalation
Experiment Station—			
Variety No. 4	2.0	91.8	91.4
Variety No. 4	3.0	92.1	92.0
Variety No. 5	2.0	90.6	91.8
Variety No. 5	3.0	92.4	92.2
Variety No. 6	2.0	91.0	91.9
Variety No. 6	3.0	91.5	91.9

Increasing the amount of lime used increased the purity of the juice prepared by oxalation an average of 0.3 points, while that of the carbonated juice was increased by 0.9 points. The point to be noted is not that more lime should be used in the oxalation procedure, but unless sufficient lime is used in the carbonation procedure to insure complete defecation, a low purity of juice may be found. Apparent purities of carbonated juices listed in Tables 2 and 3, were run by the method used for measuring the purity of oxalated juice.

Table 4 shows the purities of juice obtained from individual beets of the 1952 season and stored in the root cellar about four months before test.

Table 4.—Apparent Purities of Thin Juice from Individual Beets.

Root Description	% Sugar	Thin Juice Purity
Billings No. 1	16.7	90.6[1]
Billings No. 2	18.7	95.1
Billings No. 3	18.6	94.1
Billings No. 4	15.8	94.4
Gering—Variety A		
Gering No. 1		91.8
Gering No. 2		91.0
Gering No. 3		88.8[1]
Gering—Variety B		
Gering No. 1		93.5
Gering No. 2		91.8
Gering No. 3		87.9[1]
Sterling No. 1	15.3	94.9
Sterling No. 2	15.0	93.3
Sterling No. 3	15.1	94.6

[1] Off color thin juice showed deterioration of roots during storage.

Discussion

Probably the principal question concerning the acceptability of the new method will arise from a question as to how closely the purity of defecated press juice is related to the purity of defecated diffusion juice obtained from those beets in factory operations.

A number of years ago our Research Laboratory had occasion to investigate effects of varying the conditions of digestion of cossettes on the purity of thin juice obtained from the extract, as compared to the purity of standard carbonated press juice. The effects of varying the quantity of water used (ranging from 50 percent to 100 percent on cossettes), the temperature of digestion (ranging from 60° C. to 90° C.), the time of digestion (ranging from 1 hour to 2 hours), and the final pH of the extract (ranging from 6.5 to 5.5), were surprisingly small except when 2-hour digestion at 90° C. was used. The average apparent purity of carbonated press juice from cossettes used in 21 tests was 92.5. The average purity of carbonated diffusion juices obtained under the varying conditions of the 21 tests was 92.2. It can hardly be questioned that the purity of carbonated press juice obtained from beets shows the purity of the thin juice which the factory may be expected to obtain from those beets.

The data in Tables 1, 2 and 3, demonstrate that the oxalation technique provides a quick, accurate method for determination of thin juice purity.

The few results given in Table 4 show significant purity variations in beets of a single variety, and they also indicate that the sugar content of the beet, and the purity of thin juice obtainable from it, are not necessarily parallel. Selection of roots for thin juice purity is feasible and apparently has much to offer to the industry.

When it is desired to select beets for low content of raffinose and other constituents, such as amino acids and non-sugar carbohydrates, the thin juice provides an excellent sample for this purpose. No preparation other than dilution to a standard concentration is required for tests by paper chromatography.

Summary

A rapid method for obtaining the purity of defecated thin juice from the small quantity of press juice available from individual mother beets is detailed.

Data are presented to show that the purities obtained by the new method called oxalation, and by standard carbonation, are essentially identical.

A few results are given to demonstrate the desirability of employment of thin juice purity, as well as other factors, in selection of mother beets.