

NOT FOR PUBLICATION

A METHOD TO ASSESS CARBONATION EFFICIENCY

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ABSTRACT

A lime-phosphoric acid purification method has been shown to provide an accurate and direct estimation of second carbonation purity without any of the assumptions associated with the indirect methods of prediction using impurities. The direct method, was also shown to be precise and more expedient than the indirect methods on a small number of samples. The speed of the method was shown to be further increased by using centrifugation, instead of vacuum filtration. The lime-phosphoric acid method not only produced the purity, but also essentially the same non-sucrose composition as that of second carbonation juice.

Combined glucose and fructose concentrations of greater than 1.88% in raw juice, with typical glucose to fructose ratios of 1.48-1.67, were found to have an insignificant effect on clarified diffusion juice sucrose polarization values.

The pH set points, found to be critical to the final purity, were more easily and reliably achieved with liquid phosphoric acid than gaseous carbon dioxide.

The lime-phosphoric acid method, when used as a benchmark for factory carbonation improvements, has been shown to significantly help improve carbonation efficiency and subsequent factory operations.

INTRODUCTION

Purification is a process of impurity removal, which in terms of the beet sugar industry refers to the removal of non-sucrose components, using lime and carbon dioxide. The efficiency of non-sucrose removal, not only is very important to the economy of a beet sugar factory, it is also dependent on many variables, some of which are very complex. McGinnis summarizes this, on page 253 in his book, "This unit process is remarkable for its simplicity of purpose and complexity of chemistry."⁷

The search for an accurate and precise analytical method to show the theoretical maximum impurity removal through lime carbonation, irrespective of the many variables, has led to the development of various de-liming procedures for bench scale juice purification. In 1954, Brown and Serro developed a method involving oxalic acid as the de-liming agent.¹ Carruthers and Oldfield³, following the work of Brown and Serro, found that precipitation of calcium with oxalic acid tended to be inefficient in beet juice, despite the extremely low solubility product of calcium oxalate. Furthermore, juices purified with lime and oxalic acid were found to have inconsistent concentrations of potassium and calcium as compared to those purified by factory processes (conventional lime-carbon dioxide). Ultimately, this led to a modification of Brown and Serro's method, substituting phosphoric acid in place of oxalic acid. The modification incorporating phosphoric acid produced

straw colored juice with non-sucrose distributions similar to those found in beet juice purified using conventional factory processes.²

The method by Carruthers et al.² was further evaluated, refined, and used to determine extractable white sugar as applied to the evaluation of agronomic practices and grower deliveries.³ Dexter et al.⁴ in addition to using an advanced, expandable pH meter, used a higher temperature during the first phosphatation step and a lower lime concentration. Maximum error, using the Dexter et al.⁴ method for "clear juice purity", was shown to be low.

The objective of our current investigation was to develop a relatively quick, robust, direct, and simple analytical tool that would provide an accurate and precise estimation of the theoretical maximum second carbonation purity from a given raw juice. This method is to provide a benchmark value to use as a guide for improving the efficiency of factory juice purification.

METHODS AND MATERIALS

Evaluated variables

Factors affecting lime purification are well known, and include such variables as pH, temperature, lime concentration, rate of recirculation, retention time, density of raw juice, and gas adsorption.^{6,7} These variables contribute to the overall ability to develop a method of estimating second carbonation purity in a laboratory environment. Some of these variables, namely lime concentration, pH adjustment, agitation, temperature, and time, have been shown to have a significant effect on final thin juice purity.^{6,7} Other parameters affect the precision and speed of the analysis, such as, method of phase separation and type of agitation. Our improvements to the method for assessing second carbonation purity was based on previous research on lime purification and beet quality assessment.

Experimental

Individual 200 mL raw juice samples, prepared from large, homogenous composites, were used for evaluations. Due to a sample limitation, Part I used raw juice of a different source as that in Part II. Raw juice samples used for Part III were specific to different factories spread over different weeks. Procedural variations for different considerations are noted at the beginning of each part. All data were collected using good laboratory practice (GLP) guidelines to ensure correct instrument operation and data validation.

All experiments were performed on composite raw juice subsamples. One equipment variable was assessed at a time, while holding others constant. Cations and anions were determined using high performance ion chromatography (HPIC) employing conductivity detection. Carbohydrates were determined by HPIC with pulsed amperometric detection. Polarimetric sucrose values were obtained using an 822 nm "dark solution" polarimeter. Solids were estimated using refractometry. All other components were measured using industry accepted methods. A flow schematic of the method, showing procedural steps, is illustrated in Figure 1.

RESULTS AND DISCUSSION

Introduction

For purposes of simplicity and illustration, the experiments are reported as separate units: Part I. Method development - evaluation of variables to improve precision; Part II. Method validation; Part III. Factory assessment and improvement.

Part I. Method Development - evaluation of variables to improve precision

Lime Addition

Earlier research on lime usage for elimination of non-sugars, under variable quality conditions, suggests approximately 2.0 grams lime per 100 grams beet was adequate for non-sucrose elimination.^{1,2,3,4,8,11} Carruthers et al.^{2,3} used 2.5% CaO on pressed juice or 1.75% CaO on raw juice. Dexter et al.⁴ used 2.0% CaO on pressed juice. We found for our method, the lime concentration was best held constant at 1.80% CaO on raw juice or approximately 2.25% on beet, which assumed a 125 diffuser draft (1.25 dilution factor).

pH adjustment

Brown and Serro¹ and Carruthers et al.² used findings of "optimum" first and second carbonation CaO concentrations (0.08% for first carbonation and 0.021% for second^{6,7}) to adopt pH set points in their laboratory tests. These pH values were determined to be 11.2 for the first purification step and 9.2 for the second. Furthermore, Carruthers et al.³ determined that accurate pH adjustment in a phosphoric acid de-liming procedure is critical to the final thin juice purity. Adjustments in pH is easier with addition of liquid phosphoric acid in contrast to bubbling gaseous carbon dioxide into juice samples. Any excess of acid resulted in a depressed pH and a corresponding reduction in the purity of the clarified juice,

because of the weight of the excess acid.³ Carruthers et al.³ found it necessary to determine the pH values at 60°C that correspond to pH 11.2 and 9.2, as measured at 20°C. With samples of British juice, they observed the two titration endpoints to be at pH₆₀ 11.0 and pH₆₀ 8.9.³ However, these pH adjustment errors for the development of this method were minimized with the use of an automatic temperature compensating pH meter, which referenced the pH readings at a higher temperature automatically to the corresponding values at 20°C.

Raw Juice Density

For development of this method, individual raw juice samples were used for each clarification and raw juice density was not an issue.

Temperature and Retention Time

Temperature and retention time have generally been considered two variables that are critical to juice purification. Adequate purification using lime-carbon dioxide systems requires high temperatures and retention times of approximately 20 minutes in order to complete the complex chemical reactions necessary to provide a clear second carbonation juice of high purity and one which is thermostable for evaporation.^{6,7} However, the objective of this bench method is to predict second carbonation juice purity by impurity removal and is not one of juice thermostability for evaporation. Therefore, temperatures common in factory processes for evaporative juice stability, 80°C and 95°C for first and second carbonation steps, are not essential for prediction of second carbonation purity using laboratory methods.

Brown and Serro¹ observed that temperatures of 60°C and 80°C for their first and second purification steps, using oxalic acid as a de-liming agent, accurately predicted factory second carbonation purity. However, Carruthers et al.^{2,3} found the use of oxalic acid as a de-liming agent did not result in clarified juices having non-sugar distributions consistent with factory purified second carbonation juices. Carruthers et al.² further observed the use of phosphoric acid, in place of oxalic acid, as a de-liming agent and temperatures of 60°C and 80°C for first and second phosphatation produced clarified juices with purity and non-sugar distributions that were more typical of those found in factory purified juices using lime-carbon dioxide. Following their earlier work, Carruthers et al.² further refined these temperatures to 60°C for both first and second phosphatation steps to simplify the method. Dexter et al.⁴ used temperatures of 67°C and 37°C for first and second phosphatation steps to determine extractable white sugar. It should be noted that Carruthers et al.^{2,3} and Dexter et al.⁴ only heated the samples to the set point temperature and made no attempt to hold these temperatures for extended periods of time. These earlier researchers observed that approximately eight minutes were required to reach the first phosphatation set point.

The chemical reactions that occur under hot alkaline conditions require time and temperature to approach completion and therefore greater significance is placed on these variables during the first phosphatation step. The purpose of the second phosphatation, as with second carbonation, is to reduce the concentration of calcium salts. For all practical purposes, the various Ca-phosphate complexes formed are all insoluble in hot water.¹² Thus, successful second phosphatation is dependent upon setting the correct pH with phosphoric acid and raising the temperature to ensure the precipitation of residual calcium. If the pH and temperature are adjusted correctly, the precipitation of phosphate occurs rapidly and the retention time is less important.

Invert Sugar Destruction

In order to develop a robust prediction of second carbonation juice purity using a polarimeter as a measuring tool, polarization effects of invert sugar must be minimized by their destruction under hot alkaline conditions. Carruthers et al.² reported no appreciable invert sugar destruction by the lime-phosphoric acid purification as compared to factory processes using lime-carbon dioxide. They assumed this difference was insignificant, since invert survival did not significantly affect the purity of the clarified juice at the levels encountered during their study (370 mg/100S).

The specific rotation of glucose, fructose, and sucrose are +52.7, -92.4 and +66.5, respectively. Therefore, the effects of these carbohydrates at defined concentrations can be determined. The correction for glucose of polarimetric sucrose concentration was calculated to be 0.792 times the glucose concentration. The correction for fructose of the sucrose concentration was -1.389 times the fructose concentration. Thus, not only is the combined concentration of glucose (dextrorotatory) and fructose (levorotatory) important, but so is the ratio of glucose and fructose. The greater the fructose concentration relative to glucose, the more levorotatory will be the combined rotation effect, thereby reducing the polarimetric sucrose concentration value. Typical glucose to fructose concentration ratios in our raw juice have been observed to range from 1.48-1.67. Applying these ratios and the above polarimetric sucrose corrections suggests that the combined glucose and fructose concentrations in the original raw juice could exceed 1.88% on juice without showing a significant effect on sucrose polarization. This assumes no glucose and fructose destruction occurs through the purification process, which would place even less significance on the glucose and fructose content in raw juice to effect final clarified juice purity. For the development of our method, temperatures and heating times consistent with the method defined by Carruthers et al.³ were adopted for evaluation. These conditions resulted in greater than a 30% reduction in the combined glucose and fructose concentration.

Agitation

Basic knowledge of chemical reactions provides a means of predicting the effects of agitation of a sample during heating. Samples prepared with agitation were more reproducible, having purities with a standard deviation of 0.26; whereas those prepared with no agitation had a standard deviation of 0.65. The type of agitation, mechanical stirrer versus magnetic agitation with a stir bar, had no apparent impact on sample preparation. It should be noted that Dexter et al.⁴ suggested only occasional stirring was adequate to produce reproducible clear juice purities (S. E. mean = 0.066).

Phase separation

In order to provide more rapidly analyzed samples, gravity filtration, vacuum filtration, and centrifugation were evaluated as potential techniques for removing the sludge produced from the first phosphatation step.

Gravity filtration with E & D 615 filter paper (coarse) was found to be ineffective. It took over two hours to generate enough filtrate (approximately 100 mL) to proceed with the second phosphatation step.

Vacuum filtration with E & D 615 filter paper was considerably more efficient, requiring 15-20 minutes to generate enough filtrate to proceed with the method. This included the 5-10 minutes of cooling required to sufficiently lower the sample temperature in order to prevent sample boiling under vacuum conditions.

Centrifugation was found to be the most efficient, requiring no cooling time. Samples from the first phosphatation step, centrifuged at 3000 rpm for three minutes, yielded a tightly compacted pellet of insoluble calcium complexes, from which the clear supernatant was easily transferred to the second phosphatation step.

Gravity filtration through E & D 615 filter paper was efficient for the second phosphatation step providing samples of low turbidity for polarimetric evaluations. If turbidity occurs, VWR 413 filter paper (fine) or equivalent could be used to improve samples for polarimetric analysis. Due to the low particulate material inherent in second phosphatation samples, filtration proceeded rapidly.

Part 2. Method Validation

Subsamples of a homogenous raw juice were used to test our method and to compare it with other methods of purity assessment. Non-sucrose compositions were determined on ten replicate raw juice subsamples. This was to provide data

for predicting a second carbonation purity ("target thin juice purity"-TTJ) using selected non-removable non-sucrose impurities and to assess the consistency of non-sucrose distributions of lime-carbon dioxide and lime-phosphoric acid purified raw juice samples.

The prediction of second carbonation purity, using selected non-sucroses, has its origin in work performed by Carruthers et al.^{2,3} Research^{2,3,6,7,8,13} has shown that selected non-sucroses are not eliminated in carbonation and they carry through to thin juice. These non-sucroses reduce thin juice purity and therefore their concentrations can be used to predict the purity of thin juice. Following this early work by Carruthers et al., Batterman¹³ expanded Carruthers et al.'s list of non-sucroses, incorporating chloride, nitrate, glucose, fructose, raffinose, and betaine, in an attempt to improve the precision of the "indirect" method for predicting thin juice target purity. Batterman minimized the number of assumptions about the average anion character of juice, Carruthers et al. had used, by actually measuring these components instead of predicting them using mathematically derived constants. Batterman¹³, however, did not refer to organic anions, which have been shown to significantly contribute to the total ionic balance of juice. It is important to note that organic anions (primarily lactate) were not omitted from the early work of Carruthers et al., but were used, along with other anions, to develop "constants". These were used in an empirical equation together with the assumption of an average anion distribution, to obtain a total impurity value.

McGinnis⁷ categorized non-sugars into classes based on their ability to be removed by carbonation. These classes were non-removable non-sucroses (NRNS), storage sensitive non-sucroses (SSNS), and removable non-sucroses (RNS). Making up the NRNS category were the non-sucroses: sodium, potassium, α -amino nitrogen, chloride, nitrate, and betaine. The SSNS category was comprised of glucose, fructose, raffinose, kestose, dextran, levan, galactinol, and inositol. The RNS category had oxalates, saponins, citrates, phosphates, sulfates, and malates as components. As stated previously, Carruthers et al. included lactate as a NRNS for purposes of impurity prediction.

Also, incorporated into the TTJ value is an arbitrary 2.00 grams per 100S constant to adjust the total impurity value. This empirically derived constant reflects the calcium salt solubility product principle. However, the 2.00 grams per 100S value, as stated by Batterman¹³, is arbitrary, fine tunable, and was based less on theoretical calculations than on a desire for the target to be a realistic value for the factories' capabilities. An example of the calculation is shown for purposes of illustration. The NRNS value includes lactate.

NRNS	5.55% on sucrose
SSNS	2.22 " "
Other	2.00 (assumed)
Total NS	9.77
Purity (TTJ)	91.10

$$\text{where Purity} = \frac{1}{(1 + ((\text{NS}/100\text{S})/100))} \times 100$$

The 2.00 grams per 100S allow for some removables left in the thin juice and compensates for non-removables, which are not included in the NRNS list. These include a variety of organic compounds, which are removed incompletely or not at all, as well as the anions required to ionically balance the cations.

As stated previously, non-sucrose distributions of a batch of raw juice samples and those from two different purification methods were determined (Table 1). Shown in Table 2 are purity comparisons from bench scale purifications using lime-phosphoric acid, lime-carbon dioxide, and TTJ determined from original raw juice impurities listed in Table 1.

Carruthers et al.² concluded that the simplest and most direct method of estimating beet quality for extractable sugar is derived from the association between high purity clarified juice and high sugar content in beet. However, they further noted that a more precise estimate of beet quality could be obtained from the concentration, in meqs per 100 sugar, of potassium, sodium and amino nitrogen in lead digest. It is important to note that this procedure of indirect purity prediction represented the best compromise between precision and expediency in the tarehouse, with the technology of their time. It is further noted that impurity prediction provides not only an estimate of second carbonation purity, but a measure of the nonsucroses which have an impact on subsequent factory operations.

To this end, it is very important to not only provide a precise estimate of high purity clarified juice, but an accurate and direct measure of the degree of removal of non-sucroses from a given raw juice. Accuracy, for purposes of this paper, is defined as the purity that provides the best prediction of second carbonation purity.

As shown in Table 2, there was no significant difference between the second carbonation and the second phosphatated juice purities. However, greater precision is afforded by the lime-phosphoric acid purified raw juice as compared to the lime-carbon dioxide purified juice. In agreement with Carruthers et al.,² purity prediction using selected impurities (TTJ) was found to be more precise than either carbonation or phosphatation. However, our experiments show, using

the 2.0 gram per 100S constant, that the purity derived by the indirect method of impurity prediction (TTJ) was statistically different from the second carbonation purity and did not accurately predict this purity (Table 2).

Table 1 shows the average non-sucrose distributions for lime-carbon dioxide purified raw juice as compared to the same raw juice purified with lime-phosphoric acid. There are statistically significant differences between the two methods for glucose, fructose, organic acids, and calcium concentrations. The lime-phosphoric acid procedure, as stated previously, destroyed approximately 30% of the original invert sugar; whereas, greater than 60% was destroyed in the lime-carbon dioxide procedure. This resulted in a greater concentration of organic acids in the lime-carbon dioxide purified samples and a noticeably higher color. Residual calcium concentrations in the purified raw juice samples were significantly higher in the lime-carbon dioxide procedure, owing to the much lower solubility products of phosphates as compared to carbonates.

Part 3. Individual Factory Assessment and Improvements

Individual samples of raw juice from four different factories over different weeks were used to assess the lime-phosphoric acid method's applicability for estimating factory purification efficiency. Figure 2 shows the pooled correlation between the factory carbonation purified and phosphoric acid purified samples for all factories and weeks. TTJ values were also correlated with data from the factory carbonation purification, shown in Figure 3.

Both the phosphoric acid and TTJ methods correlate well with factory carbonation purification. However, there was a much better correlation between the benchmark and factory purities. Table 3 shows there was a significant bias in the TTJ prediction of second carbonation purity at Factory E. This is the subject of further investigations to define missing non-sugars making up the TTJ prediction.

The lime-phosphoric acid method has been used as a benchmark in a program to improve factory purification efficiency. By using the benchmark value, determining the variables of carbonation operation, measuring and reducing process variation, significant improvements have been achieved in overall purification efficiency. In Figure 4 factory carbonation improvements are illustrated by a reduction in the difference between factory thin juice purity and the benchmark values. These improvements are also shown in terms of improved non-sugar elimination (Figure 5). The improved carbonation efficiency has also improved juice stability for evaporation. Figure 6 shows reduced evaporator sucrose destruction, as measured by the difference in the concentrations of thin and thick juice organic acids and invert sugar. Improved carbonation efficiency and reduced evaporator sucrose destruction has also reduced color formation in the evaporators and sugar end (Figure 7).

SUMMARY

The described lime-phosphoric acid purification method provides an accurate and direct estimation of second carbonation purity without using the assumptions associated with the indirect methods based on impurities. Furthermore, our method is a precise and expedient means for estimating second carbonation purity with varying beet quality. This method not only produces the purity, but also essentially the same non-sucrose composition as that of second carbonation juice.

In agreement with previously reported information², combined glucose and fructose concentrations of greater than 1.88% in raw juice, with typical glucose to fructose ratios of 1.48-1.67, were found to have an insignificant effect on sucrose polarization values.

The method of second carbonation purity prediction using selected impurities (TTJ) compared favorably for most locations and beet qualities. However, a bias was seen in the TTJ prediction of purity for at least one location.

This lime-phosphoric acid method for predicting the purity of second carbonation juice is more rapid than the TTJ method on a small number of samples and its speed can be further increased by using centrifugation, instead of vacuum filtration.

The pH set points (critical to the final purity) were more easily and reliably achieved with liquid phosphoric acid than gaseous carbon dioxide. Other researchers also addressed variability in pH set point results in variable purities.^{2,3,6,7,8}

This method when used as a benchmark for factory carbonation improvements, has been shown to significantly help to improve carbonation efficiency and subsequent factory operations.

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Figure 1

METHOD FLOW DIAGRAM

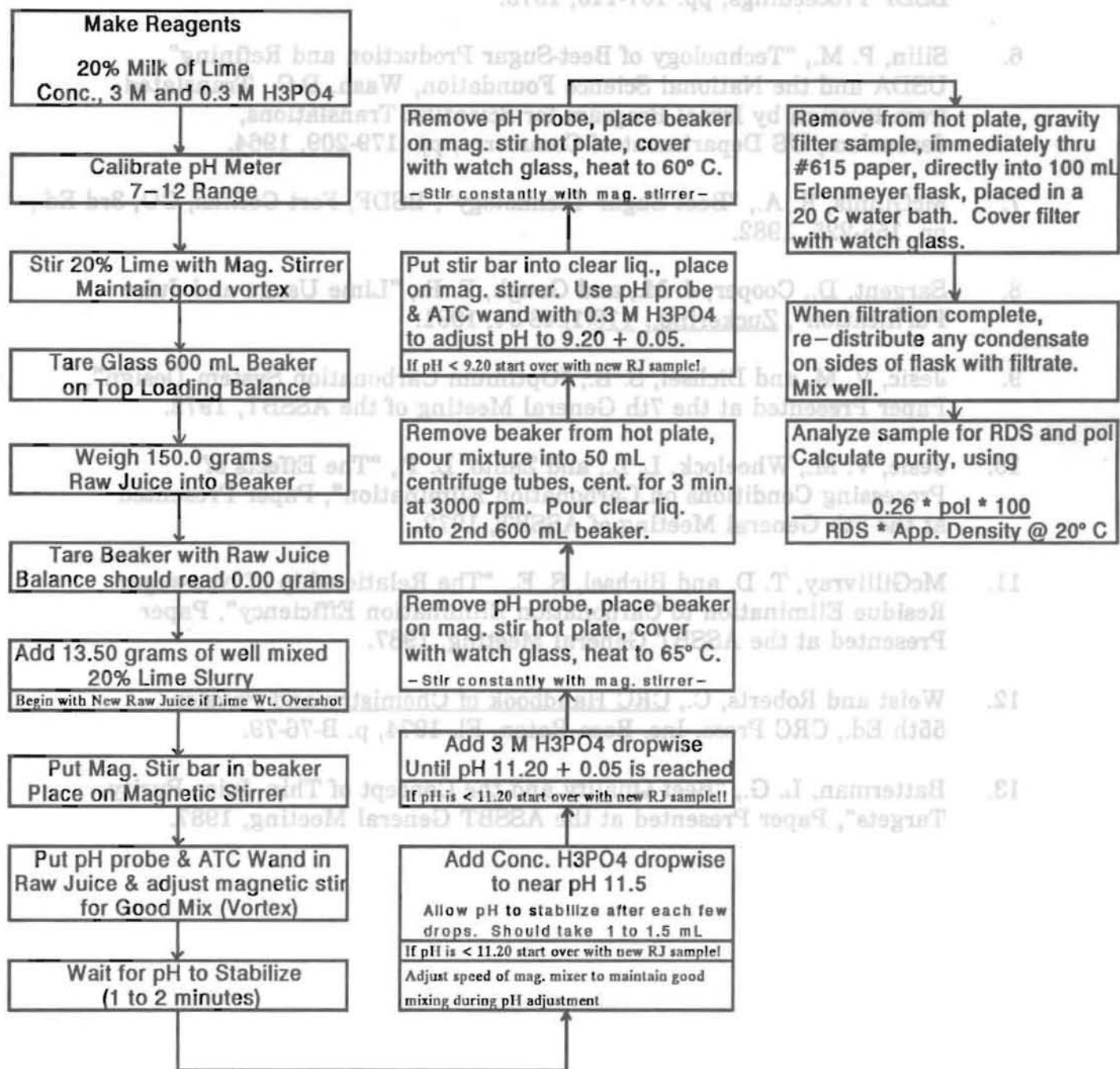


Table 1

Non – sucrose Comparisons

Mean of ten replicates

Analysis	Raw Juice		Carbonation		Phosphatation		Mean Diff.
	Mean	S.D.	Mean	S.D.	Mean	S.D.	Carb. – Phos.
RDS	13.48	0.00	13.40	0.03	13.18	0.04	0.22
Sucrose	88.79	0.02	90.05	1.00	90.97	0.32	-0.92
Raffinose	1203.00	10.05	1232.00	6.00	1212.70	13.25	19.30
Glucose	844.40	34.52	360.20	136.76	600.70	8.63	-240.50
Fructose	569.20	7.43	227.10	100.87	347.70	7.29	-120.60
Betaine	1317.00	6.40	1324.00	11.14	1326.00	9.17	-2.00
Sodium	349.00	6.12	348.90	5.52	349.90	3.48	-1.00
Potassium	945.30	5.98	948.50	6.23	948.90	5.84	-0.40
Calcium	23.10	2.77	14.70	2.45	9.60	0.80	5.10
Chloride	182.00	5.83	181.70	3.74	182.60	2.42	-0.90
Nitrate	115.40	2.87	114.70	2.69	114.60	3.44	0.10
Organic Acids	742.00	29.93	1629.00	384.82	1284.30	3.58	344.70
Amino nitrogen	1198.00	67.05	1241.00	61.55	1230.00	32.25	11.00

- Organic Acids are the total of lactic, acetic, formic, glycolic, and pyruvic acids.

- (mg/100 S) except for sucrose, which is on RDS

Table 2

Purity Comparisons

Laboratory Purifications

Replicate	Carbonation	Phosphatation	TTJ
1	88.60	90.86	91.47
2	90.30	91.39	91.27
3	88.86	90.55	91.41
4	90.48	90.76	91.39
5	90.31	90.70	91.29
6	91.04	91.45	91.23
7	90.82	91.22	91.31
8	90.62	90.67	91.45
9	91.19	90.78	91.40
10	88.30	91.28	91.32
Mean	90.05	90.97	91.35
S.D.	1.01	0.32	0.08

Organic Acids are the total of lactic, acetic, formic, glycolic, and pyruvic acids.

(mg/100 g) except for sucrose, which is on RDS

Table 3

Figure 3

Factory Purity Assessment

Mean of All Weeks

Purification Type	Factory A	Factory B	Factory C	Factory D	Factory E
Impurity Prediction (TTJ)	91.58	90.79	91.44	89.95	87.36
Benchmark Value	91.07	90.55	91.32	89.61	84.47
Factory Purity	90.21	88.75	89.72	89.10	82.99
TTJ – Factory Purity	1.37	2.04	1.72	0.85	4.37
Benchmark – Factory	0.86	1.80	1.60	0.50	1.48



Figure 2

PURIFICATION COMPARISON

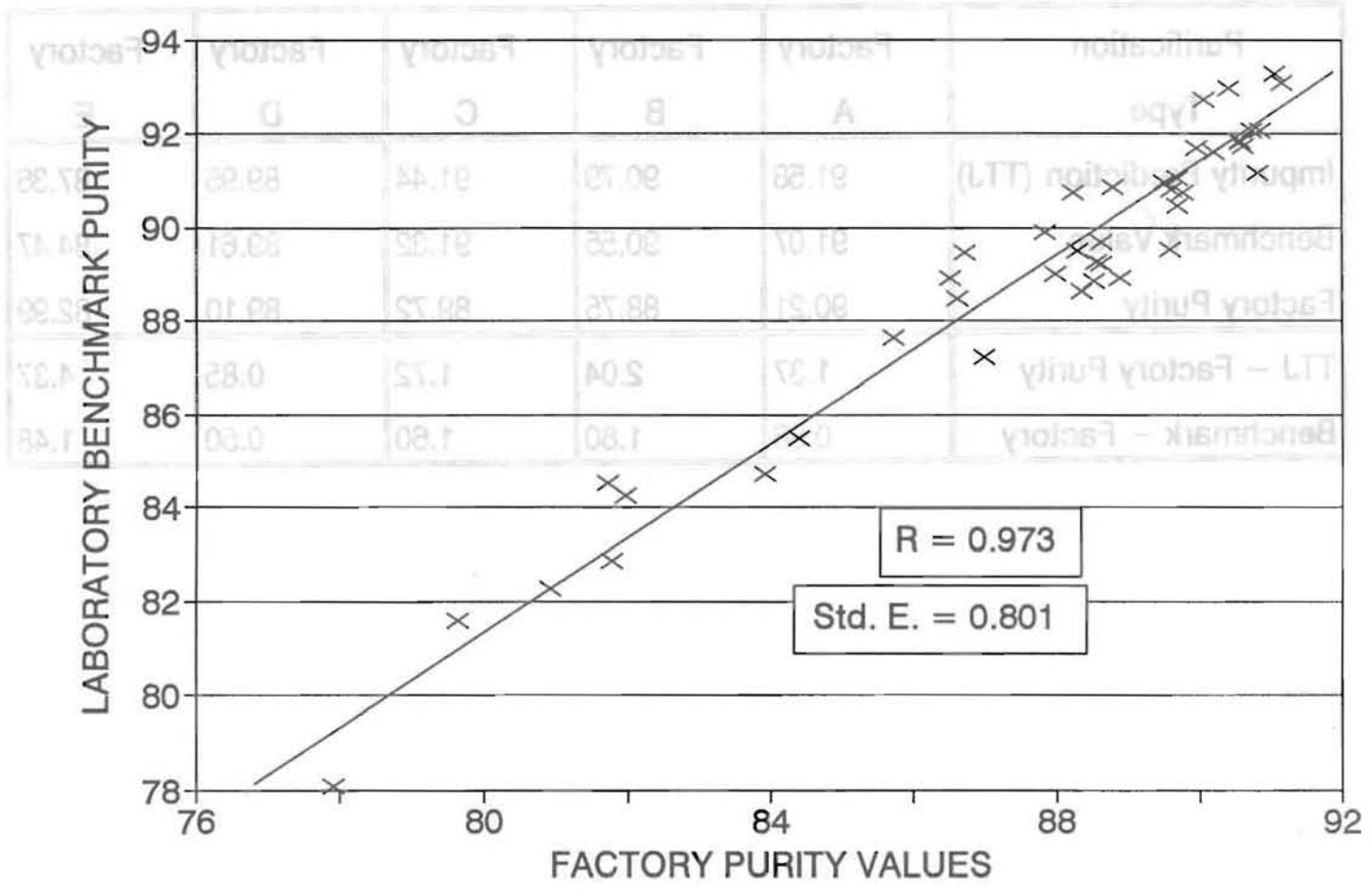


Figure 3

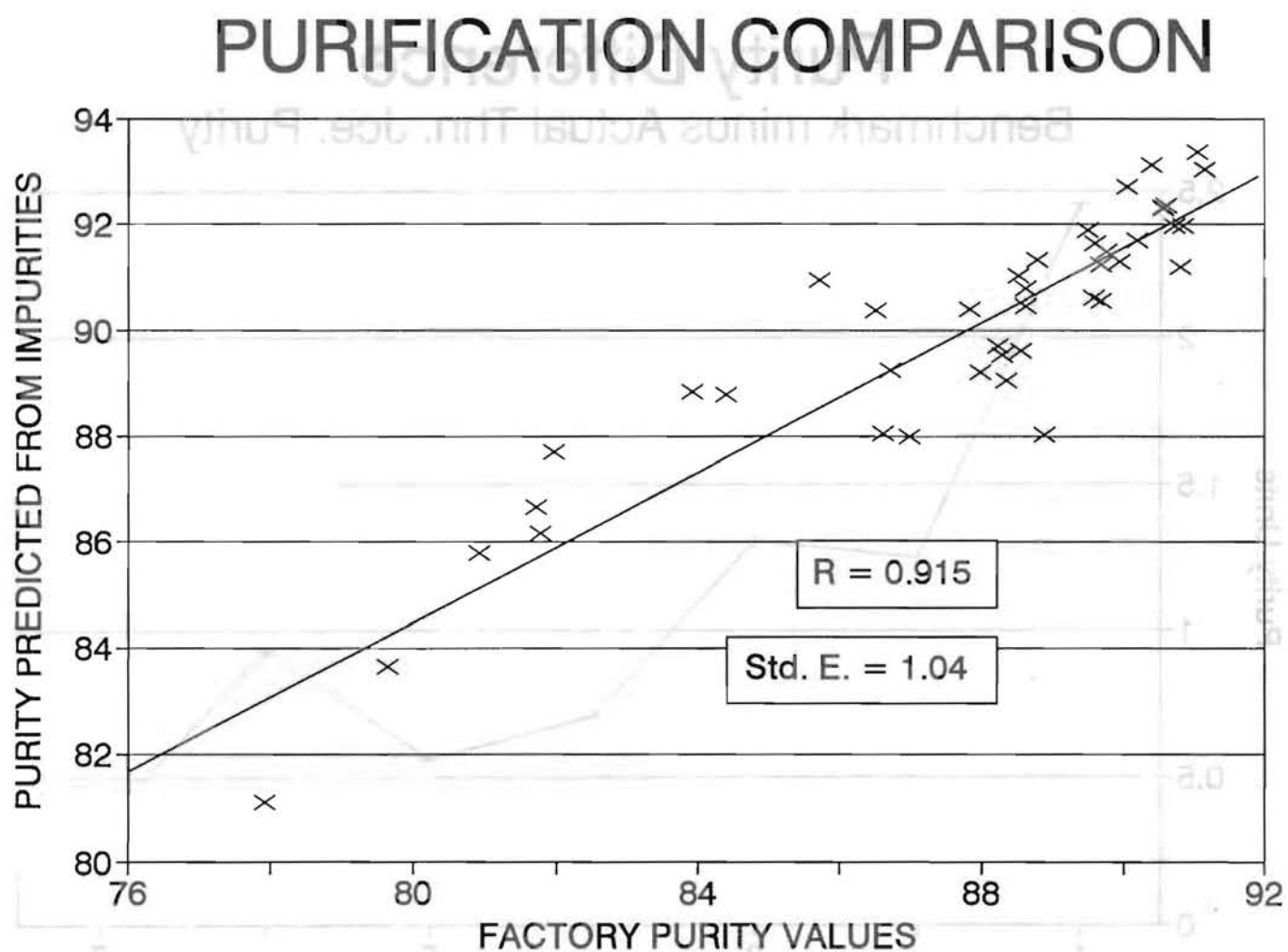


Figure 4

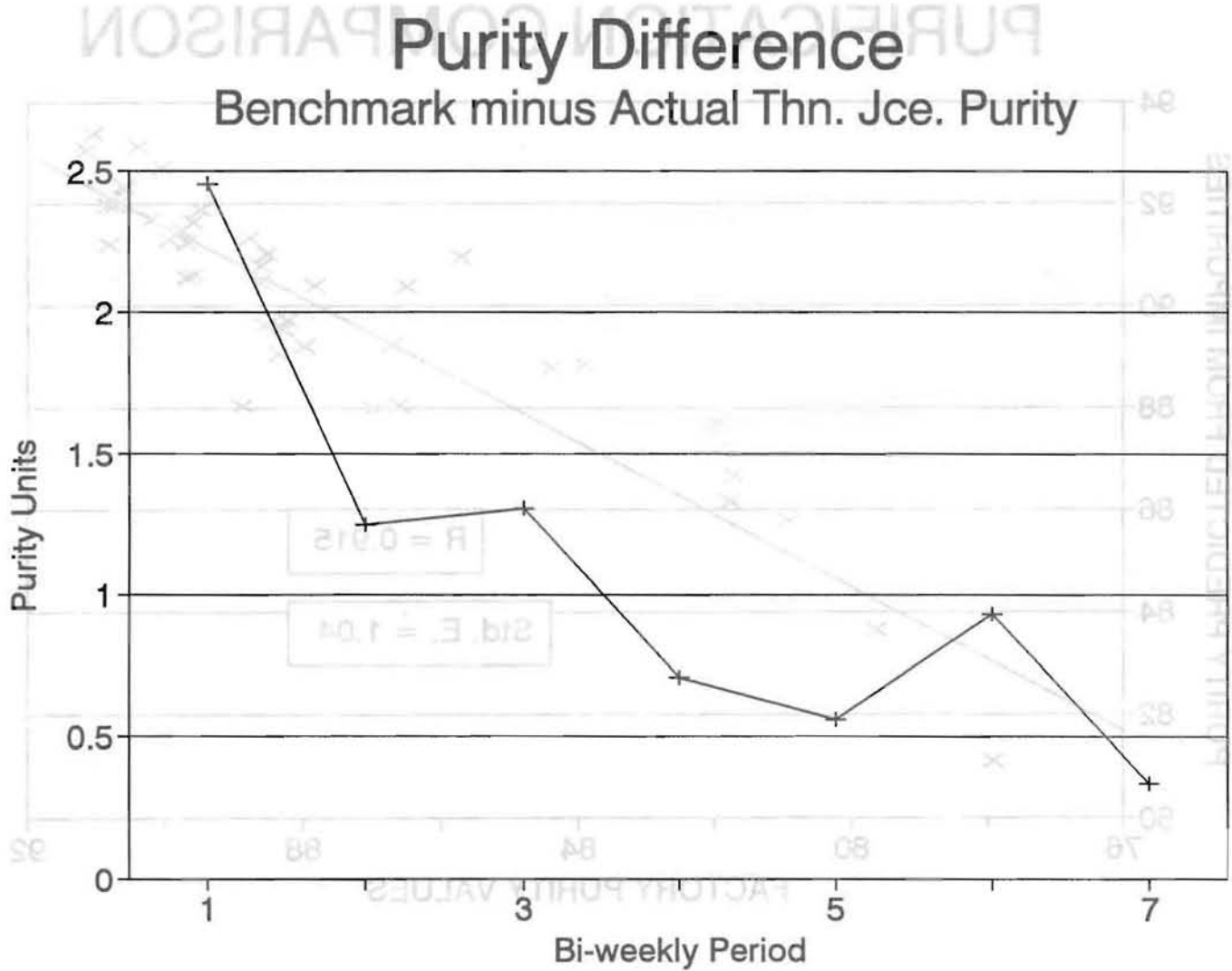


Figure 5

Carbonation Non-sugar Elimination

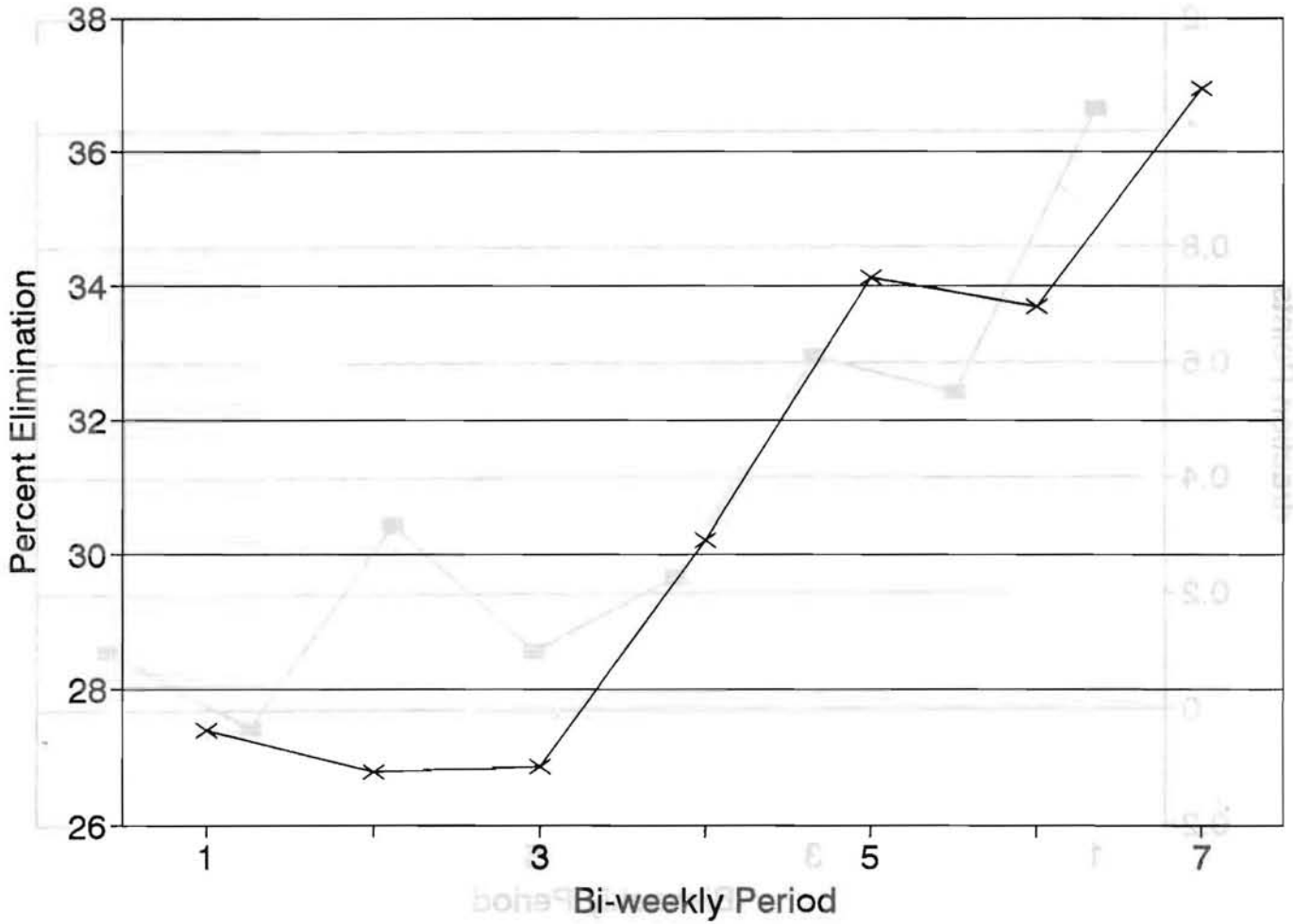


Figure 6

Evaporator Sucrose Destruction

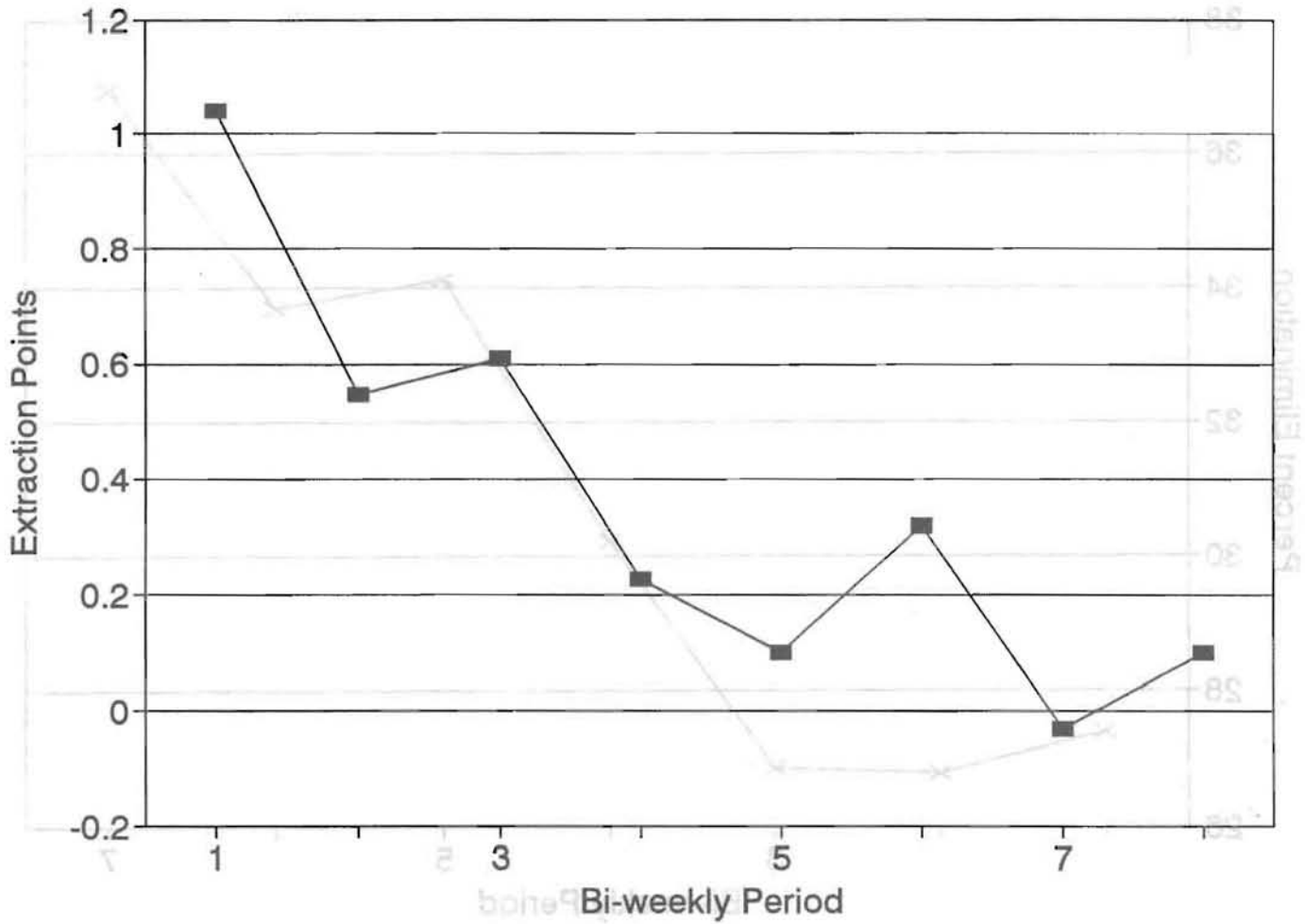


Figure 7

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Color Formation

