Pilot plant white pan boiling of molasses desugarization extract

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Abstract
Production of sugar from extract or the syrup produced from a molasses desugarization system is a technology that has been used by sugar industry as a means of generating additional revenue from what was previously a low value by-product stream (molasses). Production of sugar from extract has presented challenges that are different in degree than normally seen with the processing of thick juice. This paper is a brief examination of work completed at American Crystal Sugar to quantify the effect of extract on white pan boiling and determine to what extent changes in pH and fondant size and quantity could mitigate those effects. This work measured the effects of pH, fondant size, and fondant quantity on the crystallization of sucrose from extract. White sugar boiling at pH 8.5 was found to provide crystallization rates or pan boiling times in the pilot plant that were similar to those experienced when processing beet derived thick juice.

Introduction
American Crystal Sugar Company has produced sugar from extract for more than ten years from our East Grand Forks, Minnesota, facility and five years from the Hillsboro, North Dakota, plant. East Grand Forks (EGF) has a simulated moving bed separator and Hillsboro uses a coupled loop separator. Both separators were designed by Amalgamated Research Inc. These two separators have met all of the required performance guarantees and have performed very well. Problems that we have experienced with extract boiling have had nothing to do with performance of the separators, but are due to issues with feed provided to the separators and handling of the extract after the separators.

Typical problems experienced by the factory include: extract pH is too high and sufficient sulfur dioxide can't be added to control color or drop pH to the desired level, extract boils too slowly, grain won't grow or the pan will not take seed, color is too high, and molasses purity is too high. From the business side other complaints are heard such as: sugar production rate is too low, sugar color is high, sugar does not store well, and carbonation system is running during extract campaign that results in higher costs. The comments made by both parties are relevant and are non trivial from either the processing or business perspective.

The quality of the extract produced from the two facilities is shown in the table below. Typical values for thin juice are also shown.

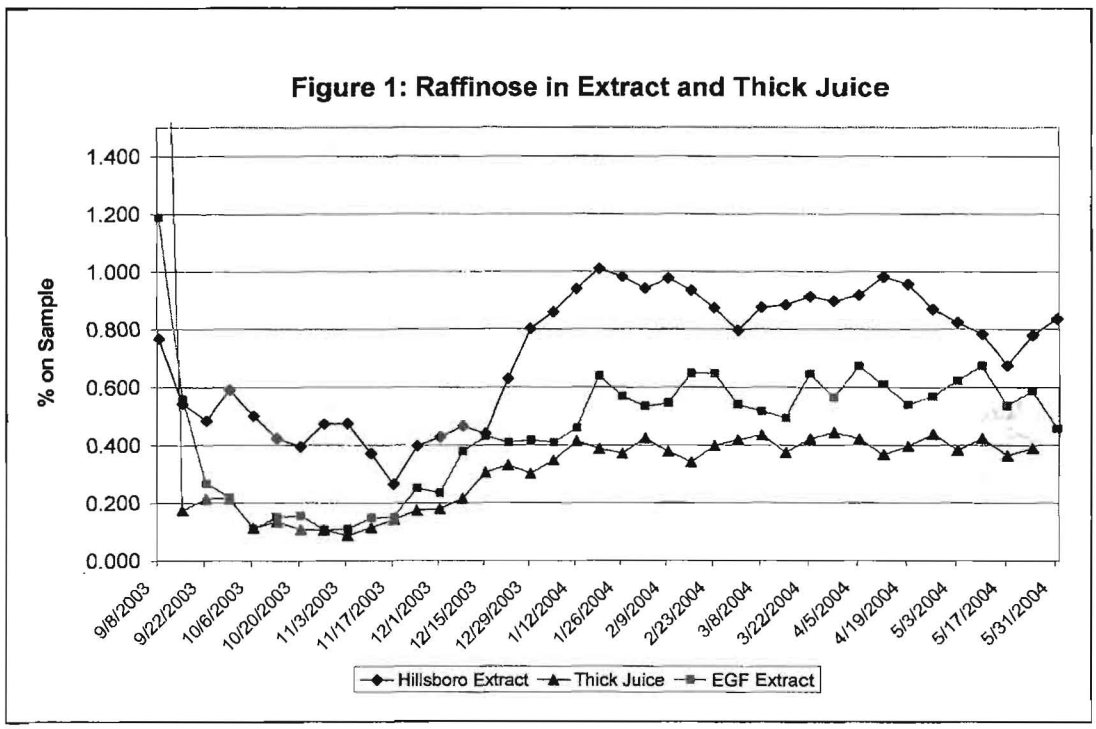
Table 1

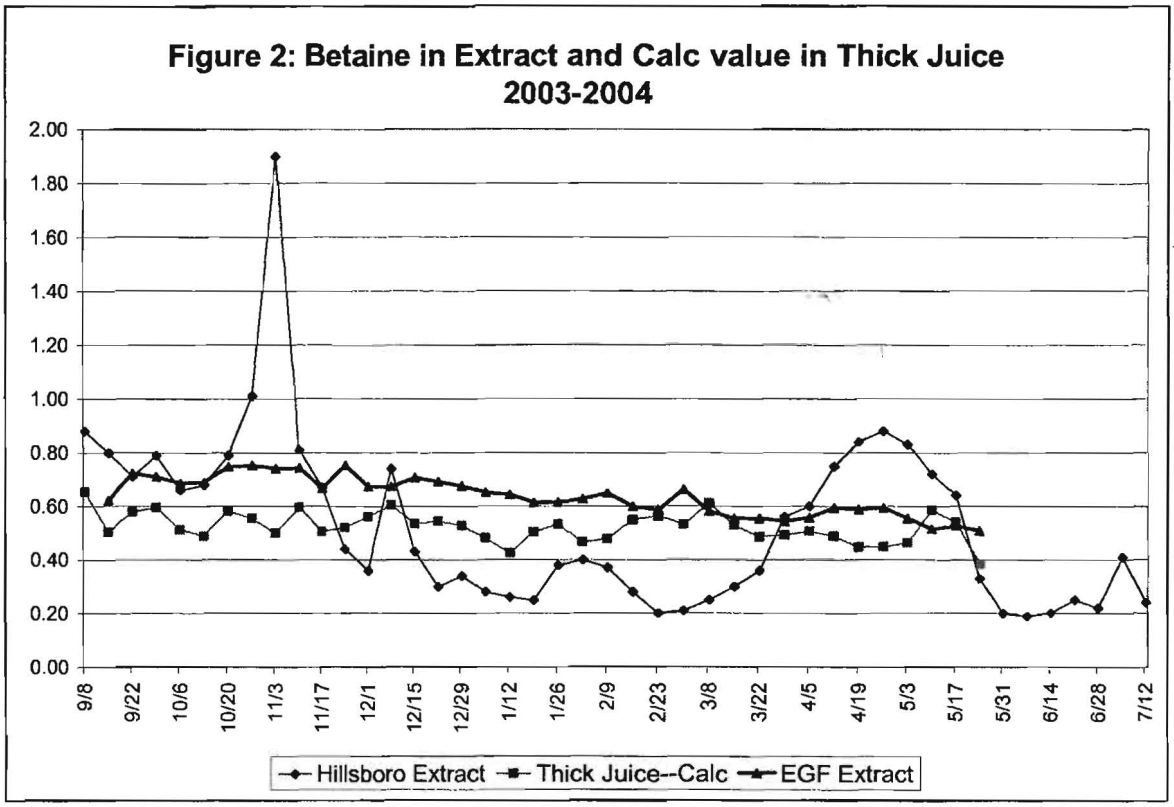
	EGF Extract	Hillsboro Extract	Thin Juice
Purity	89.55	90.79	91.5
Color (pH 7 adjusted)	8860	10256	2770
Raffinose	0.46	0.73	0.093
Betaine on RDS	2.72	0.75	0.9*
pH	9.8	10.2	8.4

* Betaine is not measured on thin juice; this value is calculated from measurements of betaine in molasses.

As may be noted from the table, raffinose levels are somewhat different in extract from the two separators. This difference is due to differences in operation of the separators and not due to differences in molasses feed. The following graph shows the changes in raffinose concentration during campaign. The following graph shows trends for betaine concentration across the campaign. Again, the difference in betaine concentration is due to differences in separator operation, not variation in the feedstock. The pH of the extract from the two separators is typically higher than thin juice. Hillsboro extract is produced at a pH of approximately 9 and then raised to pH of greater than 10 to ensure storage without microbial degradation. The rationale for the higher pH was discussed by Groom in 2003 (*1*).

Raffinose concentration is higher in both extract streams than is normally found in thick juice. The raffinose would be expected to retard sucrose crystallization with a greater effect at Hillsboro than at EGF; an effect noted by numerous authors, including VanHook (*2*). We did not evaluate the effect of raffinose on crystallization in these trials since there was no action that could be taken to mitigate the effect.





In order to determine if the differences in composition noted above or other differences were the cause of the difficult and slow boiling and crystallization of white sugar, a series of pilot plant boiling trials were run. Primary variables for the trials were pH of the extract, fondant size, fondant volume, and boiling rate or water removal rate. In addition extract and factory liquor were compared in boiling trials. In order to most efficiently study any effects due to the different parameter, the trials were run using experimental design methods. Due to only having two variables that were of any significance during the initial trials, factorial designs were used for setting up the experimental runs and for data analyses.

Pilot plant work was completed using a manually controlled batch pan. All operations of the pan from feeding to control of vacuum were completed manually. Water removal from the pan was monitored by weighing water that was condensed from the vapor stream. Time of boiling was controlled by the water removal rate. Water removal was reduced by holding the pan on water or adding water back to the pan in order to control the brix of the mass during the boiling. Boiling time was varied from 2 up to 3 hours by controlling water removal rate. Extract used for boiling trials was from Hillsboro. The pH of the extract was adjusted using 20% sulfuric acid. Sulfur dioxide was added to at the same level to all extract used in the trials.

The first set of trials was completed to assess the effect of pH, purity, and the use of sulfuric acid as an acidulant. There are many anecdotal remarks regarding the effect of pH on boiling rates with specific mention of pH below 9 being desirable. Sulfur dioxide is the primary method of adjusting pH of the extract. Due to the high pH of the extract entering the process, the use of SO_2 alone is not enough to drop the pH to 8.5-9.0 and still

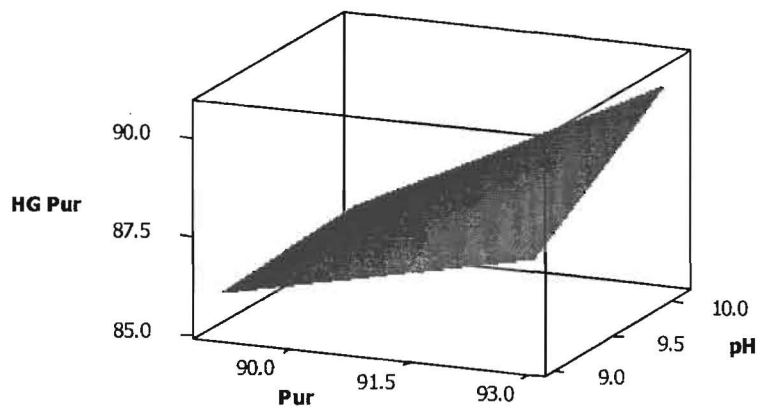
stay within the limits for SO_2 in the final sugar. Variables and levels used are shown in the next table.

Table 2

	Extract pH	Extract Purity
High level	10.1	93
Low level	9	89

The pH was found to have an effect at both purity levels and was found to have significant effects on sugar yield and the color/NS ratio. The effects are shown in the following response surface graphs developed from the experimental data.

Figure 3: Plot of High Green Purity vs pH, Purity



Figures 3,4,and 5 show that purity has the expected effects: high green purity is lower with lower feed purity, crystal yield is greater from higher purity feed, and color/NS ratio is lower with lower purity feed since there are more non-sugars present in the feed. For the most part, lower pH enhances the effects noted due to feed purity. Lower pH in tandem with higher purity feed lowers the high green purity due to higher crystallization rates in a set boiling time (Figure 3). The lower pH during boiling also lowers the color/NS ratio (Figure 5) in the green syrup from the boiling. Presumably this is due to formation of color during boiling at the higher pH. Statistical analysis of the data indicates that pH has the strongest effect on color/NS ratio and high green purity. In all other cases the purity has the greatest effect. Given that the only variable we can control with the incoming feed is the pH, the data help to support the claim that pH adjustment is useful and if SO_2 is not sufficient for dropping the pH, then sulfuric acid ought to be used.

Figure 4: Plot of Yield vs pH, Purity

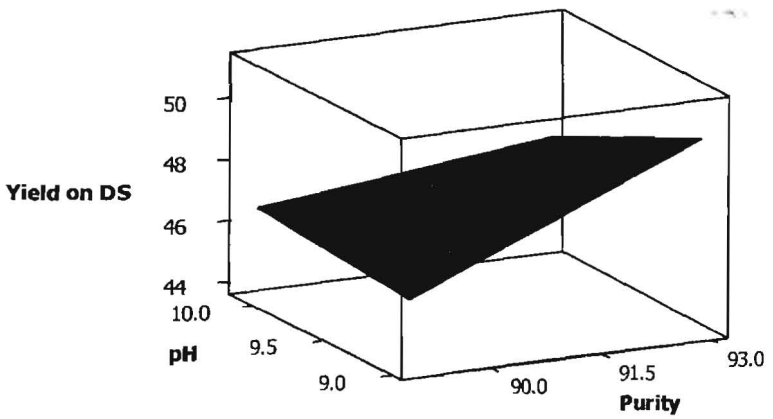
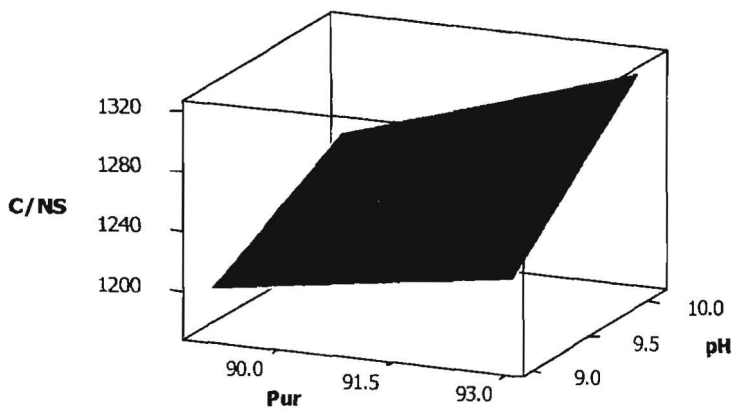
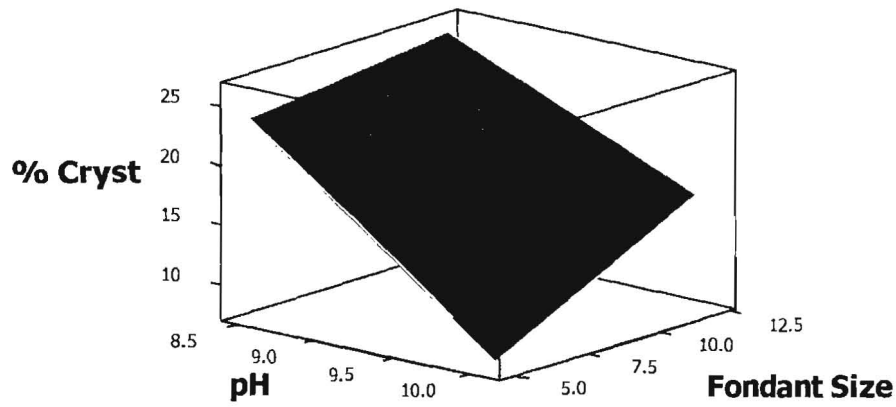


Figure 5: Plot of C/NS vs pH, Pur



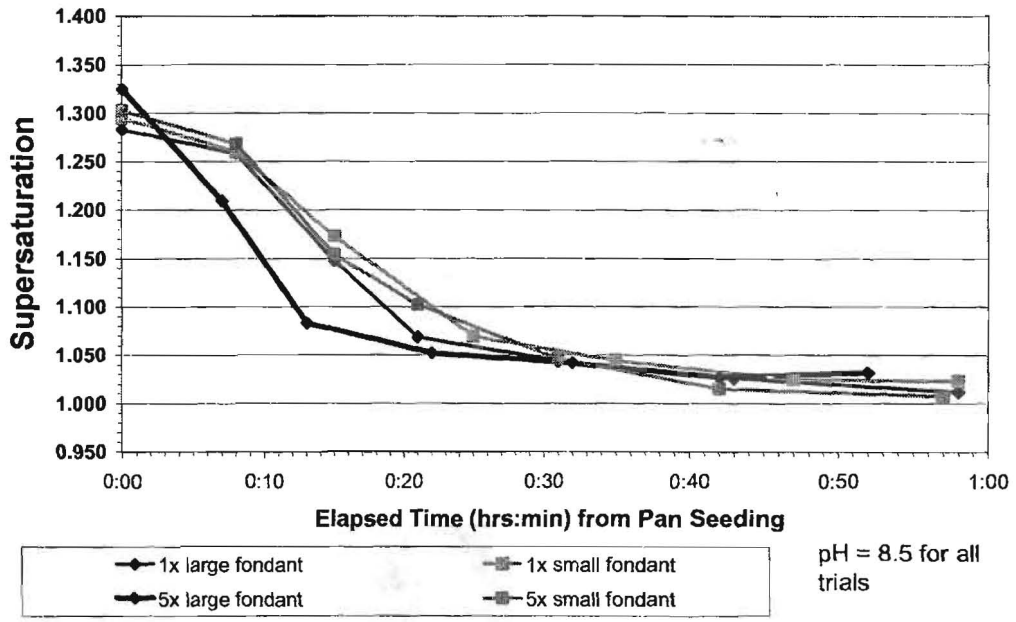
Full pan seeding with milled sugar is standard practice at all of our factories. The purpose of this series of tests was to determine if there was any advantage to using 4.8 or 11.8 micron fondant. Fondant was produced by grinding granulated sugar in isopropyl alcohol. Size of the fondant was controlled by changing the grinding time. All pans were seeded with the same number of nuclei; the quantity of seed was adjusted for the average particle size in the fondant. Size of the fondant given is the volume weighted size as determined using a Coulter particle size analyzer determined by an outside lab.

Figure 6: % Crystallization vs Fondant Size, pH



Four trials were run with the goal of establishing effects due to pH and fondant size. Data were taken from the same time during the pan boiling cycle and show that pH has a more pronounced effect than fondant size. At higher pH there is an indication that there was greater crystal yield with the large fondant; however, due to the limited number of trials, the association will not be given much significance. The primary effect on yield is due to pH and pH affects yield with both 4.8 and 11.8 micron fondant. Those results are shown in Figure 6.

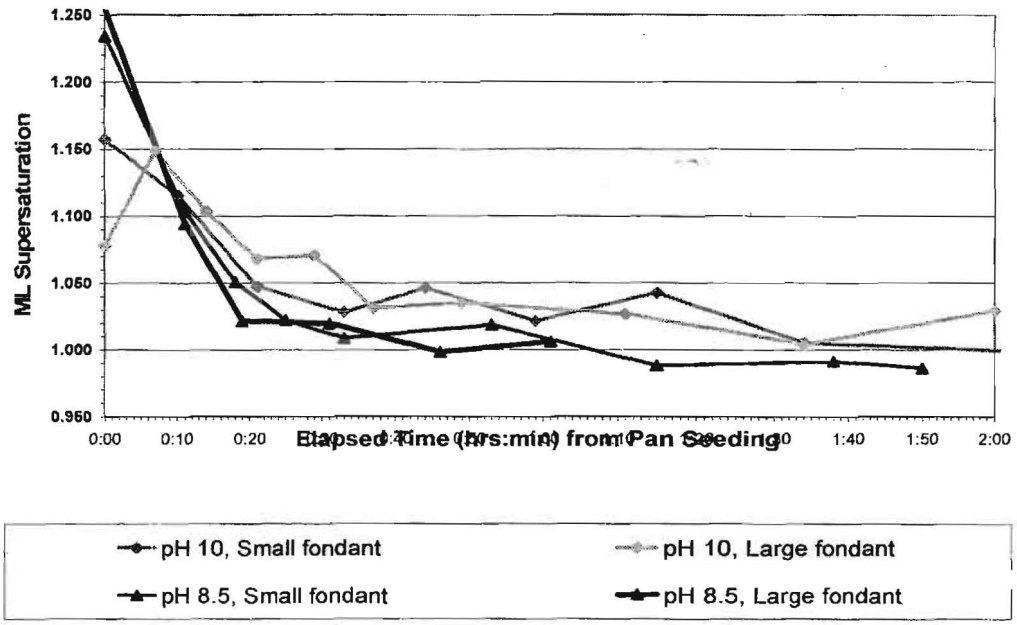
Figure 7: HLB Extract Pan Supersaturation



Another way of looking at the data is to look at the effects of pH and fondant size on the drop in supersaturation during the pan boiling cycle. At periodic intervals during the pan boiling cycle, a sample of mass would be taken from the pan, syrup separated from the mass, and purity determined on the mass and mother liquor. Mother liquor supersaturation curves were calculated from these data. A plot for the case with different fondant and pH is shown in Figure 7. The supersaturation dropped more rapidly to 1 in the case that the pH of the extract was 8.5 compared to extract pH of 10. The effect is not large, but likely is linked to how the rate of crystallization is affected by pH.

An additional set of boiling trials was completed to evaluate the effect of the amount of fondant on pan boiling rate. Extract pH was set at 8.5 and fondant size at 4.8 and 11.8 microns. Volume of fondant needed for full pan seeding was determined for both the small and large fondant and that quantity was designated as 1x. The pan was seeded to five times the calculated mass of fondant and that quantity was listed as 5x seeding. The only noticeable difference in the trials is a somewhat quicker drop in supersaturation early in the pan boiling. Thirty minutes after seeding there was no noticeable difference between the pans. Figure 8 shows the results from those trials. The more rapid drop in supersaturation would not be surprising given the greater surface area of the larger fondant. This trial was completed primarily to counter the operator argument about fondant size and volumes. The results are of course not surprising. Putting in insufficient quantity of seed will eventually result in the pan

Figure 8: HLB Extract White Pan ML Supersaturation



establishing sufficient nuclei in the pan to drive the supersaturation towards one. The result may be a low particle size and a large CV. This trial has more value in establishing that the technique of sampling during white pan boiling gives reproducible data than of learning anything new about crystallization in the white pan. The figure for trial set 8 shows the results.

The last set of trials was run to determine whether extract was truly slow boiling. Three trials were run with water removal rate of the pan controlled so that boiling time was varied from approximately 2 up to over 3 hours. The table below shows the data from those trials.

Table 3

Time from seeding to pan drop	% crystallization on Dry Matter
3:17	54
2:47	56
2:07	55

In these trials, water removal rate was varied such that the rate of sugar release from solution for crystallization was lower in the pans boiled longer. Based on the data shown in the previous figure, if the pan is seeded properly and the pH adjusted, the supersaturation drops toward 1.0 relatively quickly. The result is that the pans can be boiled as quickly as pans operating on normal thick juice. There was no significant difference in yield between pans boiled for 2 hours compared to the pan boiled for over 3 hours. Slow boiling of extract is more likely due to pH and pan seeding practices than to

chemical composition of the extract. It appears that crystallization rates of sucrose from extract are not markedly different than that found in standard liquor from beet.

In the case that extract has been pH adjusted and the boiling follows standard operating practices, the overall capacity of the sugar end may be lowered due to the color/NS ratio of the extract, but not due to the chemical composition of the extract. In the case of Hillsboro the color/NS ratio is greater than 1000. Hillsboro thin juice typically has a color/NS ratio of 400. The high color of the extract will require that pan boiling operation will have to be changed. In the case that a portion of the extract is brought into the intermediate pan, the overall capacity of the pan floor may be decreased as discussed by Thompson in 2003 ASSBT (*3*).

The chromatographic separators used at the two facilities do an acceptable job of rejecting color from the molasses. At EGF the ratio of extract color to molasses color ranges from 0.2 to 0.25. At Hillsboro that ratio is 0.15-0.18. The problem is the color of the molasses introduced into the separator. The table below shows the average molasses feed and extract color for the two facilities.

Table 4

	Molasses Feed Color	Extract Color
East Grand Forks	39,200	8,860
Hillsboro	61,700	10,300

Reduction of the molasses color at Hillsboro to the levels found at East Grand Forks would be expected to reduce extract color from 10,000 down to approximately 7100. The lower color would be expected to reduce problems with boiling extract and may be low enough to take extract directly into the white pan.

The problems with extract boiling can be mitigated by proper adjustment of pH. This may improve high green colors and result in better yield in the pans. It will not resolve the issues surrounding the high color of the extract and the impact that has on pan floor productivity.

REFERENCES

1. D. Groom, T. McGillivray, J. Heggeness, I. S. Samaraweera, paper presented at the Proceedings of the 32nd Meeting of ASSBT, San Antonio, Texas 2003.
2. A. VanHook, *Journal of the A.S.S.B.T.* **22**, 72 (April 1983).
3. P. Thompson, P. Fry, paper presented at the Proceedings of the 32nd Meeting of ASSBT, San Antonio, Texas 2003.