



# Audubon Sugar Institute

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## Annual Report

### 2003 – 2004

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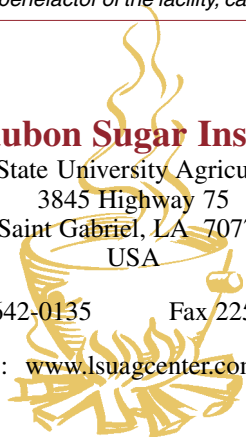
*On the cover: View of Audubon Sugar Institute from River Road. Syngenta Crop Protection, Inc., generous benefactor of the facility, can be seen in the background.*

**Audubon Sugar Institute**

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**Louisiana State University Agricultural Center**  
 William B. Richardson, Chancellor  
 David J. Boethel, Vice Chancellor and Director of Research  
 Paul D. Coreil, Vice Chancellor and Director of Extension

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## Vice Chancellor's Foreword

*The Audubon Sugar Institute (ASI) had its beginnings in the late nineteenth century, actually preceding the formation of the Louisiana Agricultural Experiment Station. In its storied history, the Audubon Sugar Institute has undergone many changes including relocation from New Orleans to Baton Rouge and an administrative transfer from LSU and A & M College to the LSU Agricultural Center. The transition that took place in late 2003 will rival those of the past, however. Although the facility is not completely operational, the relocation of the ASI faculty, staff and equipment to Syngenta Crop Protection, Inc.'s former R & D facility in St. Gabriel is nearing its final stages. It is a credit to the entire ASI team that it continued to serve the sugarcane industry during this transition.*

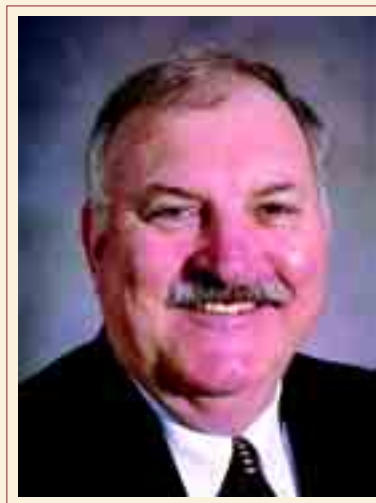
*Sugarcane remains one of Louisiana's most important agricultural commodities. Among the traditional row crops grown in 2003, it ranked first in farm gate value at \$359 million and value-added income at nearly \$230 million. When one considers the number of people involved in production of sugarcane, those that operate and work in the sugar processing mills and those that are employed in the infrastructure that support the crop, it is obvious that sugarcane is vital to the economic well-being of our state.*

*Recently, international trade agreements and discussion of allotments and proportionate share acreage have caused concern for the sugar industry, and these events highlight the challenges that remain for research to develop knowledge and technology to allow the industry to be competitive. ASI scientists strive to find ways to help sugar factories become more efficient through research and transfer of the developed technology to the mills. Under the leadership of Dr. Peter Rein, the faculty at ASI including Dr. Rein, have made a conscious effort to visit the sugar mills throughout the state in an attempt to make their expertise available. In addition, the ASI faculty have a commitment to discover value-added properties associated with sugarcane. Even though sugarcane leads most other commodities in value-added income, enhanced effort in this arena appears worthwhile.*

*This past year, ASI partnered with Michigan Biotechnology Institute (MBI), a not-for-profit company recognized as a leader in biomass conversion, microbial fermentation, and bioprocess research and development. Collectively, these two organizations will seek to develop integrated technologies that will convert bagasse, molasses and cane leaf matter to high value-added products. This collaborative venture will be supported in part by a special grant from the United States Department of Energy.*

*MBI represents a new partner, but we are grateful to our long-time partners, the members of the American Sugar Cane League, for their financial support of projects deemed high priority by the Dedicated Research Committee of the League. Although the LSU AgCenter provides the base of operational support for ASI, the grants awarded by the ASCL are critical for focusing the unit's research on the most important needs of the sugar processing industry. Also, the ASCL funding has provided leverage for ASI scientists to pursue other grant funds. And the ASI scientists have been aggressive and successful in doing so! For example, in the latest round of the Louisiana Board of Regents competitive grant programs, ASI scientists received four of the 14 grants awarded to AgCenter scientists. This is a remarkable success rate, considering the number of faculty members at ASI.*

*As I begin my duties as vice chancellor and director of the Louisiana Agricultural Experiment Station, I am excited about the direction and future of Audubon Sugar Institute. When you read this ASI annual report, I believe you will share my enthusiasm. With support of the sugar industry, the programs are focusing on issues relevant to the industry, value-added research seeks to complement exciting production and processing programs and educational programs are being launched to return ASI to a position of prominence in the training of sugar engineers. Finally, the unit will be housed in a much improved facility, which should stimulate and facilitate greater productivity. We welcome you to visit the new "home" of ASI and learn of the contributions being made to maintain a sustainable sugar industry in Louisiana.*



David J. Boethel



## Mission Statement

To foster a center of excellence for applied and original sugar research, which exceeds the expectations of our stakeholders in Louisiana and the international sugar industry, through innovative research, technology transfer and education.



## Goals of the Audubon Sugar Institute

### Goal 1

To enhance the productivity and profitability of the Louisiana sugar and other sugar process-related industries.

### Goal 2

To improve the practice of sugar manufacture through education and technology transfer.

### Goal 3

To conduct research toward a diversified sugar process industry.

### Goal 4

To attract, retain and develop a world-class staff to serve our stakeholders.

### Goal 5

To encourage use of low environmental impact technologies in sugar processing.

## Audubon Sugar Institute Advisory Board

Dr. William Brown – LSU AgCenter  
Michael Daigle – LULA-Westfield  
Neville Dolan – Raceland Raw Sugar Corporation  
Trevor Endres – Enterprise Factory  
Barry Forse – Cinclare Central Factory  
Ronald Guillote – St. Mary Sugar Coop., Inc.  
Roddy Hulett – South Louisiana Sugar Coop., Inc.  
Windell Jackson – American Sugar Cane League  
Dr. Benjamin Legendre – LSU AgCenter  
Greg Nolan – Lafourche Sugar Corporation

Anthony Parris – Iberia Sugar Coop., Inc.  
Rivers Patout – Sterling Sugars, Inc.  
Robert Roane – Jeanerette Sugar Co., Inc.  
Dr. Peter Rein – Audubon Sugar Institute  
Chip Savoie – Westfield Sugar Factory  
Charles Schudmak – Cora-Texas Mfg. Co., Inc.  
David Stewart – Alma Plantation  
Jackie Theriot – Louisiana Sugar Cane Coop., Inc.  
Tommy Thibodeaux – Cajun Sugar Coop., Inc.  
(As of March 2004)

## Head of Institute's Report

*A chapter in the life of Audubon Sugar Institute ended and a new one began with the move from the old factory building on the LSU campus to our new facility in St. Gabriel. The last remaining useful equipment from the Audubon Sugar factory, the three 3-roll mills and their gearing, were salvaged and taken to St. Gabriel. I have a vision for the future installation of the mills in a new Audubon Sugar factory on our new site at St. Gabriel with steam supplied from Syngenta. Such a facility would set us apart as a sugar institute with unequalled teaching, research and training opportunities.*

*The move has been more disruptive than any of us imagined; we continue to put in extra effort to get our total capability operational again because it is obvious we will have a world-class facility of which we can be proud. We are most grateful to Syngenta Crop Protection Inc. for this most generous donation of its R & D facility. It has everything we need and suits us very well.*

*Dr. Bill Brown, vice chancellor of the AgCenter until his retirement in the spring of 2004, put in considerable effort to secure our new premises. This was typical of the way he has supported, helped and promoted Audubon in all of our activities. We are most grateful for what he has done for Audubon Sugar Institute. We all wish him well in his retirement and look forward to working with Dr. David Boethel, who succeeds him.*

*Audubon continues to make progress. We welcomed Dr. Luis Bento, a world-class sugar technologist, as a new professor. We have also been strengthened by our new staff members Julie King, Melati Tessier and Stella Polanco. The Audubon team is growing in caliber and capability.*

*Our collaboration with Cenicaña in Colombia is progressing well, and during the year we signed another collaboration agreement with the Mauritius Sugar Industry Research Institute. We also accommodated visiting research scholars from Portugal and the Czech Republic, both of whom spent productive time with us. Working contacts with more LSU departments have been fostered, with new collaborative effects with Mechanical Engineering, Biological Engineering and Chemistry bearing fruit.*

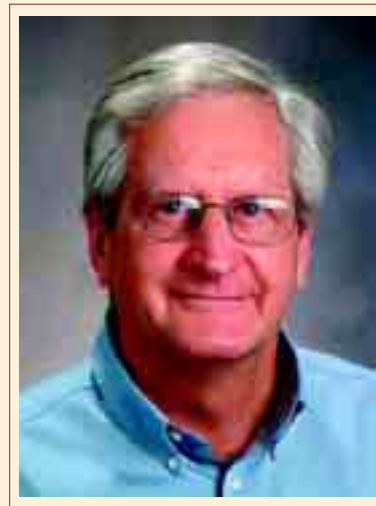
*We continue to receive good financial support from the American Sugar Cane League. We are very grateful for this and are aware of our need to deliver value in return.*

*We have been trying hard to improve our extension efforts for the Louisiana industry. We started a new series of scheduled off-crop and mid-season visits, in an attempt to keep our faculty in touch with the requirements of the industry. In most cases, this has benefited us and the Louisiana mills. We intend to make our Web site more useful for the Louisiana processors, too.*

*Our short course program has run well. We continue to engage with international experts to augment our capabilities in covering a range of courses we believe are of value to the industry. The courses in the College of Engineering for undergraduate and graduate students are now in their third year. We continue to attempt to provide education and training opportunities that are relevant and useful – but we could use a bit more feedback, and, in some cases, support from the Louisiana industry.*

*We still have too few faculty members to cover all the areas and achieve everything I would like to see Audubon do. Our efforts to get an endowed professorship looked promising but did not materialize. Expanding the number of faculty needs to be an important goal in the next year.*

*We look to the future with considerable optimism. We have enhanced potential in our new premises, although we still need to spend more of our resources on getting fully operational. We have been successful in a number of research grant applications that will allow us to expand our graduate research programs. We have productive and energetic people who will help us climb to new heights. I thank them for their efforts in the past year, particularly in the extra work involved in moving to St. Gabriel.*



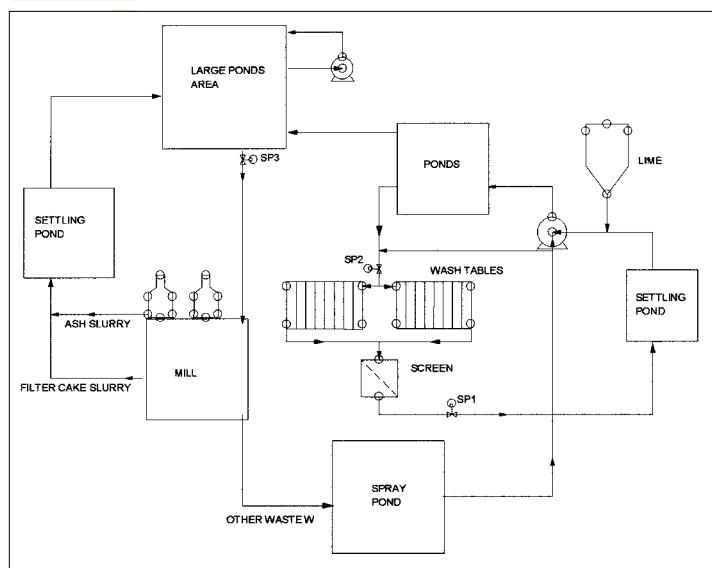
Dr. Peter Rein

## A Survey of Wastewater Handling and Composition at Louisiana Sugar Mills

The goal of this survey, based on visits to the mills in the course of the 2003 processing season, was to describe the layout and main elements of the wastewater system and provide data on water composition.

The dry weather throughout the 2003 season allowed mills at times either to avoid completely or at least reduce washing of cane for most of the season. Special attention was given to the wash-drum cane cleaning system at Enterprise because of its unique design and the general interest of the industry in improving cane-washing operations. Because of the layout, washing in the drum could not be stopped, so that it was always in operation, providing a dependable source of wash water for sampling and analytical work, for measurements of mud sedimentation velocity with and without flocculants, and measurements of efficiency of hydrocyclones for mud removal.

**Figure 1. A typical Louisiana sugar mill wastewater system.**



**Table 1: Sugar and Ion Composition of Samples in Sugar Mill Wastewater Survey.**

Location	Date	pH	Sucrose	Glucose	Fructose	Na	NH <sub>4</sub>	K	Mg	Ca
SP1	10/22/2003	8.4	390	20	150	138	n/d	183	86	237
SP2		8.5	30	0	150	137	4	204	95	255
SP3		8.7	0	0	280	143	10	264	112	187
SP1	12/11/2003	7.5	180	70	60	116	14	232	98	192
SP2		7.6	10	0	0	129	13	226	98	203

**Table 2: Anionic Concentrations in Wastewater samples.**

Location	Lactate	Acetate	Propionate	Formate	Butyrate	Chloride	Malate	Sulfate	Oxalate	Phosphate
SP1	10	476	318	3	224	193	n/d	35	n/d	22
SP2	5	508	325	2	238	210	n/d	34	n/d	23
SP3	8	253	173	3	32	238	n/d	38	n/d	28
SP1	91	370	284	32	142	228	2	18	1	13
SP2	66	356	275	23	125	223	20	25	1	12

An example of the results is shown in Tables 1 and 2. Sugar and ion composition is given in mg/L. Of the anions, chloride, malate, sulfate, oxalate and phosphate come from the cane and the soil, but lactate, acetate, propionate, formate and butyrate, products of sugar degradation, originate primarily in the wastewater loop and should therefore be indicative of the condition of the pond system. Some of the more volatile short-chain anions, formate and acetate could also come from the condenser water if some of it enters the wash-water loop.

Typically, the solid filter cake is diluted and pumped to the wastewater ponds, together with the fly-ash slurry. Most systems fully or partially recirculate the cane wash water, and the water is released from the ponds after the BOD levels have reached acceptable levels through natural degradation after the grinding season.

## A Dip Assay for Dextran

The presence of dextran in sugar process juices normally indicates stale cane, because much of the dextran is generated in damaged plant (sugarcane) material. The proper operation of a sugarcane processing facility would benefit from targeted exclusion of loads of stale cane from the process. A rapid, low-cost method for dextran analysis can be applied to screen loads of sugarcane before delivery to the mill yard.

The isolation of an anti-dextran phage suitable for use in a simple assay format, such as a paper dipstick was targeted as an appropriate reagent for dextran analysis in this application. A phage library (Fab 2lox) was screened for dextran-binding phages using five different methods: thin layer chromatography (TLC), Sephadex column, enzyme-linked immunosorbent assay (ELISA) screening, a combination of ELISA and Sephadex column screening and a technique developed in the course of this research, Sephadex bead agarose electrophoresis (SBAE). Immunonephelometric assays were used to determine the dextran-binding affinity of all new phage preparations.

Protein-coated polyvinylidene difluoride membranes (PVDF) dipped into dextran solution (T2000) and dried at room temperature were used as the matrix. Test solution is applied to the membrane, and it is dried at room temperature. Then phage is applied to the paper, incubated for 1 minute, washed once and dried. A diluted solution of HRP-anti-M13 is applied and then washed three times. TM Blue™ substrate (0.5 ml) was applied to develop color. Dextran concentrations higher than 50 ppm in sugar juices could be visually detected using a paper-dip assay.

Saturation with dextran was achieved below 500 ppm of T2000. Saturation of antigen on the PVDF membranes limited the upper range of the assay. The assay showed high specificity against dextran but not cornstarch, sucrose, glucose or chitin. Table 3 summarizes the specificity of phage (AE-M1114-m74-2R) for polysaccharides. A phage (AE-M1114-m74-2R) based paper dip assay was tested for specificity against dextran T2000, cornstarch, sucrose, glucose, and chitin at 1000 ppm. Phosphate-buffered saline (PBS) was used as a control. The developed color was scanned by NucleoVision scanning densitometry system. Standard deviations were obtained from triplicate experiments (n = 6).

Dextran concentration in mixed sugar juices was estimated by comparing the sample color with that from a standard that showed similar color intensity. The results were compared to those obtained with the Midland SucroTest™.

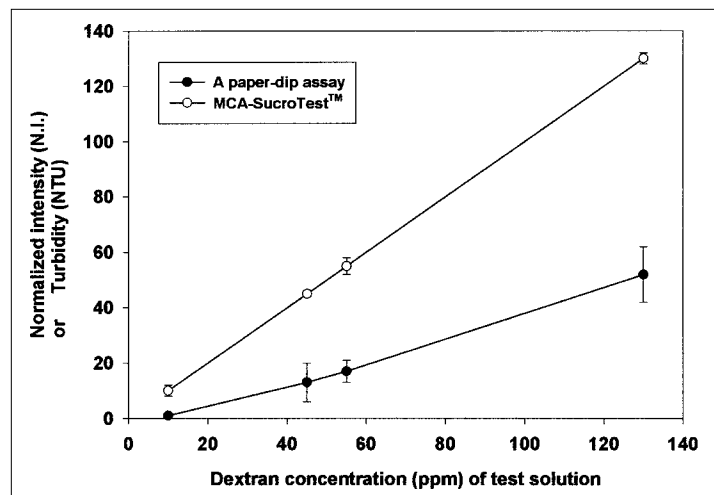
Analysis of dextran in mixed juices from four different sugar mills showed

high correlation with Midland SucroTest™. With some simplification, this paper-dip assay system has potential to become a cost-effective method suitable for routine screening of sugarcane coming to the mill.

**Table 3: Specificity of phage (AE-M1114-m74-2R) for polysaccharides.**

Carbohydrate	Normalized intensity (N.I.)	Standard deviation
Dextran	27	7.1
Corn starch	6	8.5
Sucrose	1.5	2.1
Chitin	1.5	0.7
Glucose	6	8.5
PBS (Control)	3.5	4.9

**Figure 2. Detection of dextran in sugar juices by paper-dip assay and MCA-SucroTest™. Each point represents a sample obtained at a different Louisiana sugar mill. The error bars represent standard deviations of triplicate experiments (n=6).**



*Dr. Day preparing fermentation media.*

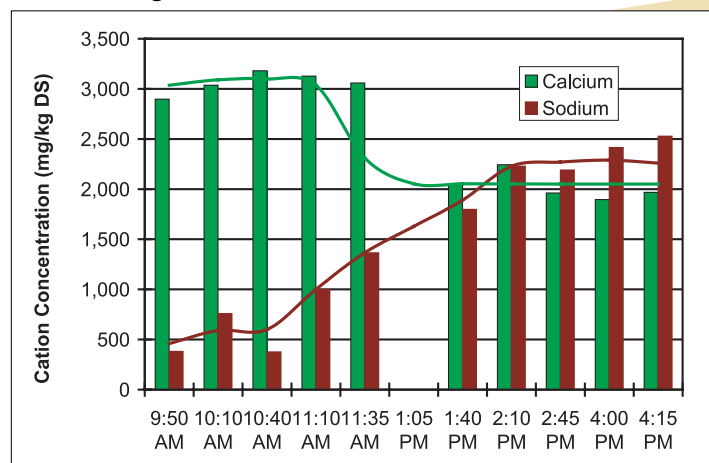
## Replacement of lime with soda ash in cane juice clarification

After laboratory studies of juice clarification, full-scale trials were organized to study the potential of full or partial replacement of lime with soda ash. In two factory tests, the soda ash liquor was continuously added to cold mixed juice and hot liming then operated as usual. In the first test, the soda ash addition was about 70 kg Na<sub>2</sub>CO<sub>3</sub> per hour, or about 0.02% on cane. In the second test, the soda ash addition was doubled to about 135 kg Na<sub>2</sub>CO<sub>3</sub> per hour, or 0.04% on cane, and was high enough that, after heating the mixed juice, pH reached the factory pH set point and therefore no milk of lime was added for the duration of the four-hour test.

The samples of mixed juice (at a location downstream of the soda ash addition), clarified juice (at the exit of the clarifiers) and syrup (from the syrup pump) were taken at various intervals before, during and after the addition of soda ash liquor, cooled to 25 to 45°C, and their solid content, conductivity and pH measured within 15 minutes after being taken. Turbidity of the clarified juice was measured on hot juice with a standard Hach ratio-nephelometer. Small sub-samples of about 20 ml to 50 ml were heat-sealed in plastic pouches, immediately frozen in a low-temperature freezer, then transported frozen to ASI and analyzed later for sugars, anions and cations.

No processing problems were noted in either test when soda ash was being applied. The turbidity of clarified juice, though, more than doubled, and was, with the soda ash addition, at the upper limit of clarified juice turbidity range (100 to 200 NTU) usually seen in Louisiana. Whether increased turbidity of clarified juice affects the boiling house operation is unknown, but it is likely that a judicious change or adjustment of flocculant type and/or dose would be able to compensate at least partially for the increase. As a result of partial or full replacement of lime with soda ash, neither potassium nor magnesium levels were affected. Sodium increased as expected, and calcium was reduced essentially down to the levels in the incoming mixed juice (Figure 3).

**Figure 3. Calcium and sodium profiles in clarified juice prior to and during the soda ash clarification.**



Although this represents a substantial drop of calcium levels in comparison with liming (30% to 50%), even the higher dose of soda ash (Test 2) did not reduce the hardness of clarified juice below that of the mixed juice. Either the relatively low pH or other reasons prevented or inhibited precipitation of calcium carbonate. One possible complicating factor might be that the calcium determination is by HPIC of a highly diluted solution (app. 0.1 Brix) and if the calcium carbonate were present as a fine precipitate (causing perhaps the increased residual turbidity), it could have been re-dissolved upon dilution and detected as soluble calcium.



Removal of none of the measured anions appears to have been significantly affected by the switch from lime to soda ash. Phosphate was higher in Test 2 during soda ash addition, but that may have been caused by the somewhat lower average pH of clarified juice during rather than before soda ash addition. Although the number of syrup samples was limited, it appears again, as in our previous testing in 2001 and 2002, that when soda ash is used, pH of syrup is higher than pH of clarified juice, unlike in liming.

Interestingly, the levels of phosphate and oxalate, known components of evaporator scale, are lower in the syrup than in the clarified juice (unlike those of the other anions), presumably reduced by the amount retained in the scale, but the number of samples is too limited for a definite conclusion.

Use of soda ash in clarification appears feasible, reducing calcium levels in clarified juice by 30% to 50% in comparison with liming, and more if higher pH levels of clarified juice are maintained. Whether this translates into reduced evaporator scaling can be determined only in an extended factory operation, which should also involve optimization of flocculant dose and type in juice clarification when soda ash is used. With the lower dose required, the cost of soda ash may be comparable to the cost of lime treatment, but with an advantage that soda ash is a solution that is easier to handle than the milk-of-lime suspension and faster to react in neutralizing the cane juice acids.

Yet untested but potentially significant could be the benefit from reduced silica ( $\text{SiO}_2$ ) levels. While typical  $\text{SiO}_2$  levels in the industrial grade soda ash are about 100 ppm, even good quality hydrated lime has about 1% or 10,000 ppm  $\text{SiO}_2$ , which may at least partially lead to silica scaling in the low pH environment of the evaporators.

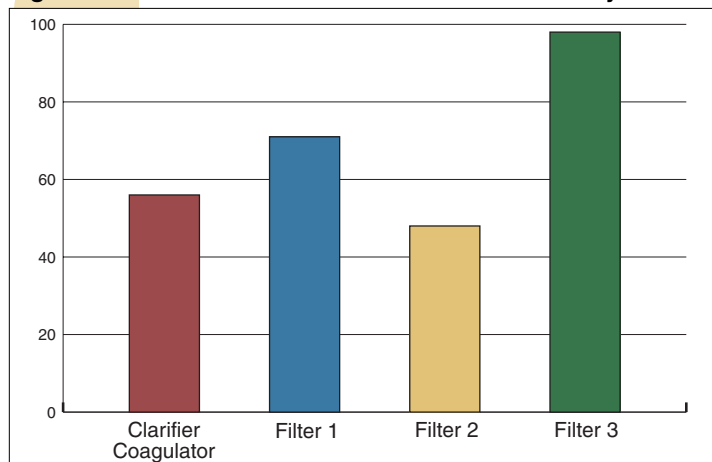
### Organic Acid Loading as a Measure of Filter Losses

Previous research has shown that the presence of specific volatile and/or non-volatile organic acids could be used to pinpoint areas of microbial generated loss of sugar in mill operations.

Questions were asked as to whether specific mud filter systems were sources of microbial contamination. The use of organic acid levels as a measure of microbial activity was used in an approach to this problem.

A previous survey indicated that most Louisiana sugar mills were operating their filters at temperatures (inlet and outlet) high enough to preclude microbial contamination. Even so, in some instances increases in organic acid levels were detected (Figure 4). In some cases the change in organic acids was between the clarified juice and the mud filter. This investigation will be continued next season.

Figure 4. Increase in lactic acid across a mud filter system.



The southwest corner of the new location of Audubon Sugar Institute.

### Anaerobic digestion and production of biogas from sugar mill effluents

An estimated 30 to 40 tons of sugar in filter cake and 10 to 30 tons in cane wash water are disposed of in wastewater ponds every day in an average Louisiana mill.

At a theoretical yield of 10,000  $\text{ft}^3$  gas (65% methane) per ton of sugars, a potential exists to produce up to 550,000  $\text{ft}^3$  of biogas a day at a mill, equivalent in Btu terms to some 350,000  $\text{ft}^3$  of natural gas. This could eliminate need for natural gas at the mills, drastically reduce the land area now required to hold the wastewaters and also reduce air and water pollution from uncontrolled degradation of sugars and other organics in the mill effluents.

Results from a non-optimized laboratory reactor were encouraging, but it is realized that substantial improvements in the organic load, gas production rate and gas composition are possible.

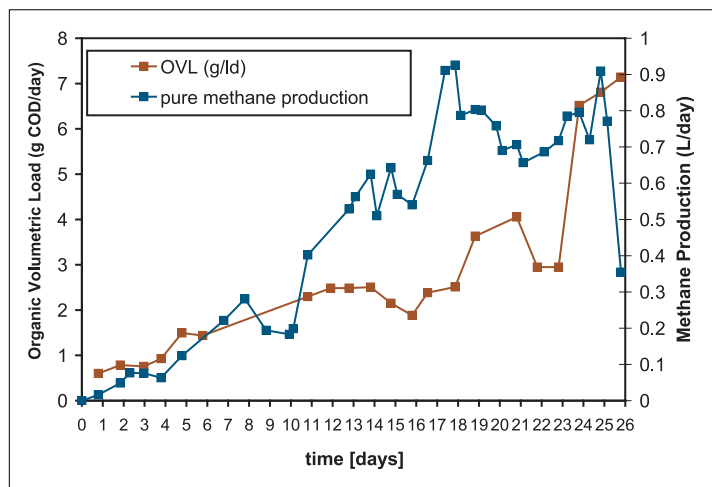
Figure 5. A 4L laboratory anaerobic reactor for dilute filter cake slurry.





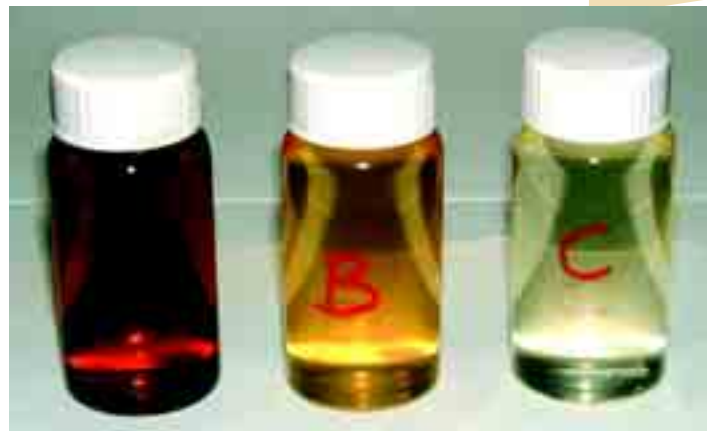
Plans are under way for construction and testing of a pilot anaerobic reactor with longer residence time, automatic pH control and a design that will allow decoupling of the hydraulic and sludge residence times.

**Figure 6. Results from a laboratory anaerobic reactor fed with dilute filter cake. Hydraulic and sludge residence times were approximately 24 hours, at a temperature of 50°C.**

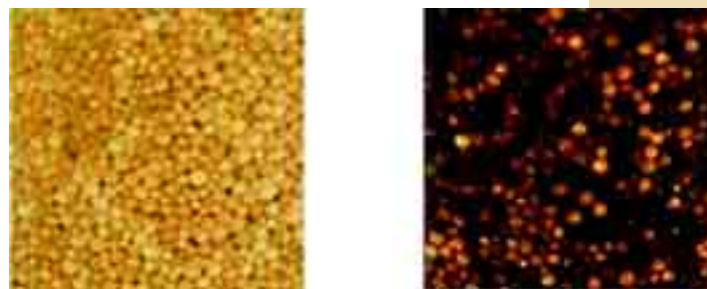


liquor is used (Figures 7 & 8). The longer working cycles imply that regeneration costs in chemicals and utilities will decrease. Less effluent and sweet water will be generated as a result as well. The regeneration effluent color will be lower compared to normal conditions, thereby requiring less treatment and reduced costs. These preliminary results justify further studies.

**Figure 7. Raw cane sugar liquor (A); after oxidation with hydrogen peroxide (B); after decolorization with resin (C).**



**Figure 8. Resin after 160 BV with oxidized liquor (left); and resin after 79 BV of non-oxidized liquor.**



## Decolorization studies

Sugar colorants are comprised of a variety of compounds of different origin and chemical nature. Their presence in final white sugar is detrimental to sugar quality.

The study of colorants in sugar production and refining is key to the research goals at Audubon Sugar Institute to establish their origin, formation and removal. The study will help understand the impact of sugar colorants on processing, the mechanics of how these compounds bind with sugar crystals, their behavior during sugar storage, and their effect on sugar quality during final use. The properties of these compounds can be further investigated to provide the information needed in identifying colorants that will add value to products such as their use as antioxidants and to produce colored sugars.

The production of white sugar directly at the mills is another important objective of this research. Studies have already begun to develop a simple and economical decolorization process to remove colored impurities in cane syrups as a step toward this goal.

Ion exchange resins have been used successfully in sugar decolorization for more than three decades. Resins used for decolorization are polystyrenic divinyl benzenic resins with quaternary ammonium positive groups bonding to a mobile ion, normally a chloride. These resins have great capacity to remove cane sugar colorants; however, some colorants attach to resins so strongly that their removal with normal regeneration can be very difficult.

An approach to alleviate this problem is the introduction of oxidants to the process before decolorization with resins. The use of oxidants such as hydrogen peroxide and ozone is known to the sugar industry. The oxidation reaction transforms high molecular weight colorants into colorless compounds of lower molecular weight. Organic acids also are formed by this reaction. This phenomenon provides favorable conditions for ion exchange resin decolorization.

Results of tests with raw cane sugar liquors previously decolorized with hydrogen peroxide indicate that longer working cycles are achieved with final resin color lower than when non-oxidized



*New professor at Audubon, Dr. Luis Bento, running some ion exchange decolorization trials.*

## Modeling Adsorption of Cane Sugar Juice Colorant in Packed-bed Ion Exchangers

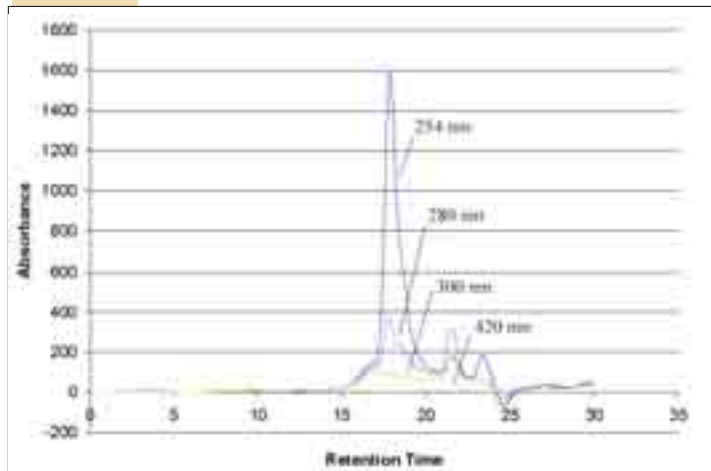
The removal of cane sugar colorants using packed-bed ion exchangers has become an area of interest with the prospect of the direct production of white sugar at the mill. Processes such as the WSM process (White Sugar Milling) have been developed, because of the attractiveness of the possibility of producing white sugar directly, with the simultaneous production of high-grade molasses.

To optimize the process design, it is necessary to be able to model the colorant removal and to predict the operating parameters of the ion exchangers. Using different pretreatment methods, a predictive model is being developed based on model parameters established by Broadhurst (2002) and extended by further experimental work.

The premise of both these models is that the color in the sugar juice can be described by breaking the overall color into defined pseudo-components using gel permeation chromatography (GPC). These colorant responses can then be measured and modeled independently.

Colorant response from GPC data is obtained using absorbance detection. Previous work was done in the visible spectrum (420nm), but it has been found that more accurate responses for individual components can be obtained in the ultra-violet region. Using various color formation tests and measuring the consequent color increases, it was concluded that 280nm was the most appropriate wavelength for quantifying the colorant. This is illustrated in Figure 9, where the magnitudes and resolution of the responses can be seen, with 254nm having the largest response but having too much emphasis on one peak.

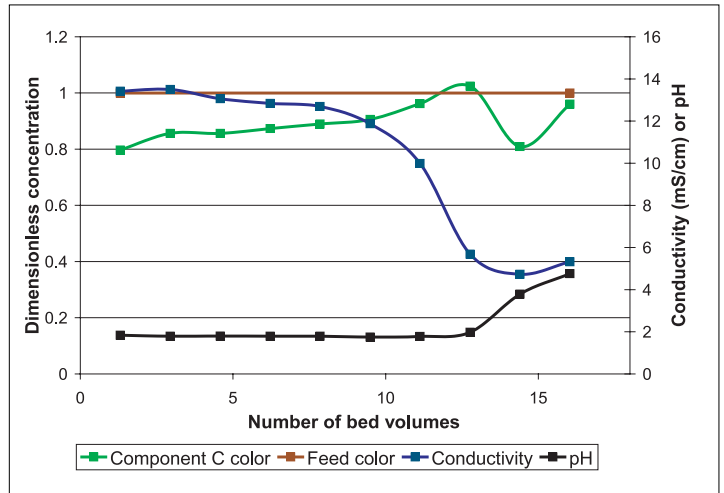
**Figure 9. Absorbance versus retention time for St. James mill syrup for all wavelength considered.**



The current process involves a strong acid cation (SAC) followed by weak base anion (WBA) ion exchange. The complication in the modeling is the strong dependence of adsorption upon the pH, particularly for the SAC. As the pH increases, the adsorption parameter of the system experiences a large change requiring complicated expressions to predict its behavior. This behavior is illustrated in Figure 10, with the colorants being desorbed as the pH increases, affecting the colorant removal.

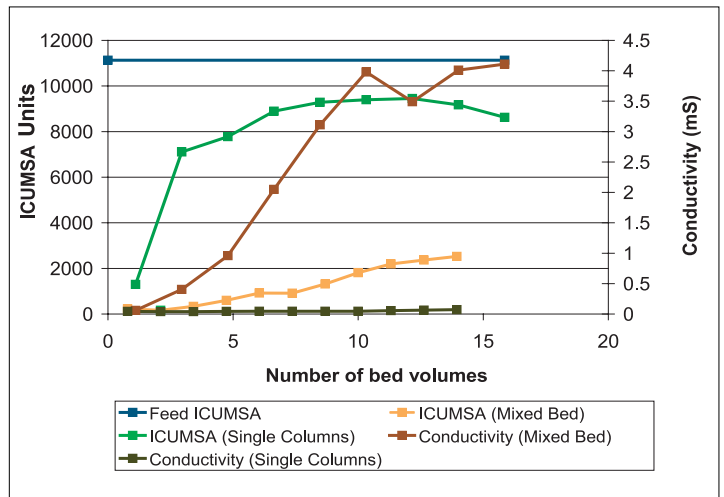
Mixed bed ion exchange columns were investigated in an attempt to obviate the refrigeration required for the SAC stage, to inhibit inversion at a low pH. It was found that a neutral pH could not be maintained because of the low buffering capacity of syrup, even with various permutations of cationic to anionic resin ratios. More serious was the loss of the ability to remove the same amount

**Figure 10. Breakthrough curve for SAC resin.**



of colorant in the solution, and a reduction in color removal was recorded as demonstrated in Figure 11. This suggests that the low pH region in the SAC column is important in achieving a higher color removal.

**Figure 11. Comparison of ICUMSA color leaving mixed bed to SAC and then WBA column run.**



## Online Measurement of Evaporator Heat Transfer Coefficients

The availability of good heat transfer coefficient data is scarce so, to capture valid and useful data, an attempt was made to use an online calculation of the heat transfer coefficient.

The sugar mill chosen was St James, which has a quadruple effect Robert evaporator train with no vapor bleeds and no condensate flash recovery. An online mass and energy balance computes the heat transfer coefficient for each of the vessels, assuming steady state conditions.

The computation is completed in Microsoft Excel, using the solver and iterate functions as well as a macro to converge to the steady state solution. The controller (Honeywell UMC800) takes data from the mill in the form of nine temperature measurements from RTDs, two volumetric flows from magnetic flow meters (flows of juice into the train and syrup out the final effect) and a microwave Brix probe for the Brix measurement of the final syrup. Once the computation is completed in Excel, the data is sent to the con-



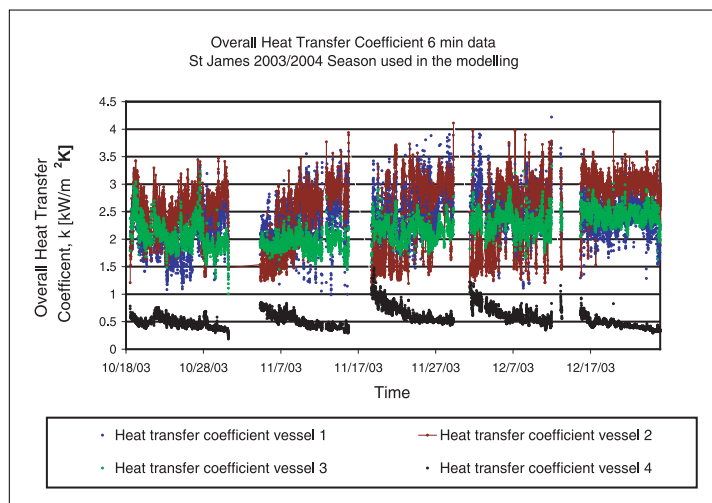
troller program for logging and data storage. The data is all logged at one-minute intervals and the model in Excel is run every five seconds to ensure the calculation is always at the converged solution when the heat transfer coefficient data is captured.

A number of technical difficulties meant that data capture began only about a month into the season, starting in the middle of October 2003. Once these problems were corrected, the program interface of Excel and the control program was excellent and the online computation proceeded successfully. Later in the season, however, the microwave Brix probe gave problems and was taken offline and replaced. The replacement then caused the same problem, so any subsequent data from the mill had to be rerun with lab values for Brix. An RTD measurement in the third vessel calandria was found to be erroneous because of a heat conduction error and a corrected offset for that temperature was applied.

A major advantage to measuring the heat transfer coefficient online is that mills are able to tell when a particular vessel is underperforming and requires attention to repair or clean that effect. The use of the model also means that the computed mass flows can be used in a mass balance check for the mill.

The trended data for the 2003 season is shown in Figure 12. This data is computed as an effective heat transfer coefficient and does not include the boiling point elevation caused by hydrostatic pressure for the differential temperature. Figure 12 shows that the only effect that is scaling up to any significant degree is the final effect. When the mill performs its chemical cleaning, all four vessels are cleaned. To save both downtime and money, the data indicates that the mill may not have to clean all the vessels on each stop but rather just clean the last effect or the last two effects. This would reduce the cleaning time from 24 to 12 hours and at least halve the chemical usage.

**Figure 12. Heat transfer coefficients for the St. James Sugar Mill quadruple effect evaporator in 2003.**



The St. James mill is making adjustments to the feed distribution into the evaporators. Over the next season, we hope the calculated heat transfer coefficients will increase with the changes. This should be evident in the calculated values.

The scale in the final effect was clearly visible when photos were taken at the end of the season, even after an initial clean of the vessel. In Figure 13 the scale is clearly seen in the tubes and on the lower tube sheet of the calandria in the final effect. Samples of the scale were collected and will be analyzed at Audubon.

A model is being developed to predict the scaling of the effects based on dissolved components in the juice fed to the evaporator train.

**Figure 13. Scale in the final effect tubes and on the lower tubesheet at the St. James Sugar Mill.**



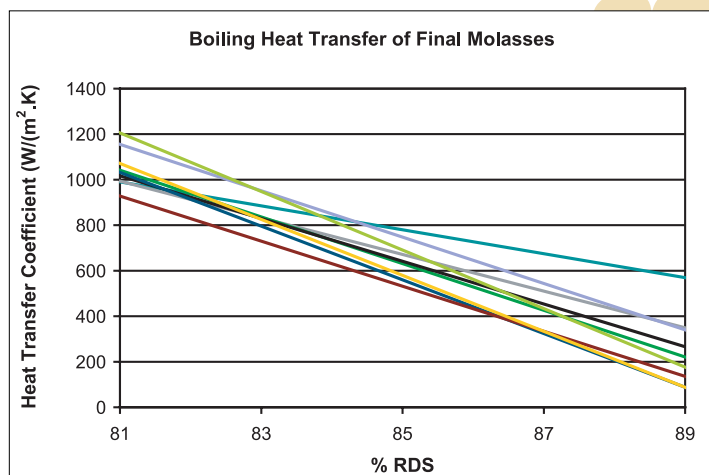
### Hard to boil masecuite: Effect of viscosity, polysaccharides and ionic composition on boiling heat transfer rate of molasses.

The weather was mostly dry during the 2003 processing season, and no boiling difficulties were experienced by the mills. For a better understanding of the factors that may affect heat transfer and rate of boiling in the pans, a series of measurements were made. The final molasses samples were further characterized by their viscosity vs. RDS profiles, polysaccharide concentrations and cation composition.

With one exception, the heat transfer coefficients vs. RDS profiles fell within a fairly narrow range and were nearly linear in the 80% to 90% RDS range.

As expected, a good correlation was found between the heat transfer characteristics and molasses consistency in the 80% to 90% RDS range, measured at standard conditions. This reflects the effect of dry solids on both heat transfer and consistency of molasses. Correlations between the molasses viscosity at constant solids levels (in the 80% to 90% RDS range) and their contents (on dry solid basis) of dextran, starch, potassium, sodium, magnesium and sodium were studied. Surprisingly, no significant correlations were

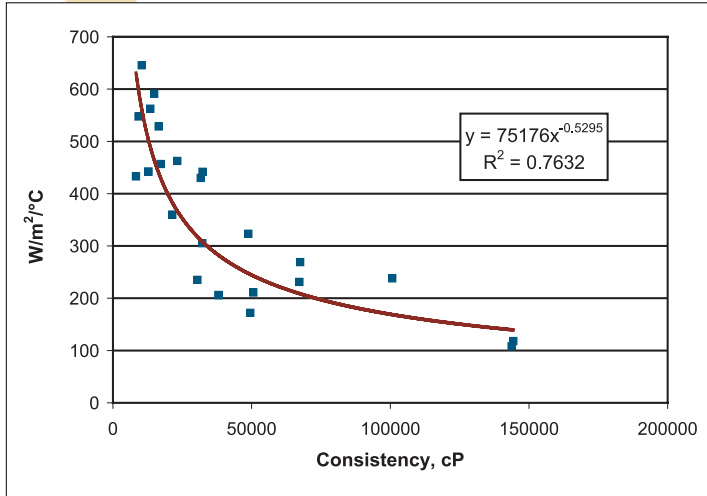
**Figure 14. Boiling heat transfer coefficient of ten Louisiana final molasses from 2003 season. ASI pilot vacuum pan, 10 psi steam pressure, 4.5" Hg absolute pressure.**



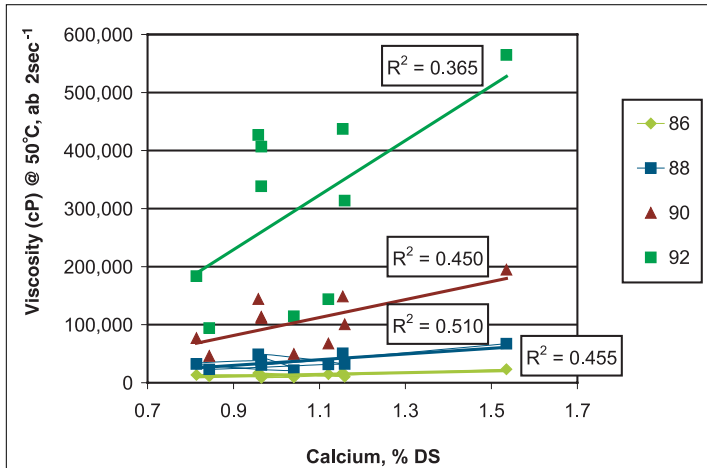


observed between viscosity and dextran or starch concentrations, and only calcium appeared to correlate with any statistical significance at the 86%, 88%, 90% and 92% RDS levels. Whether this is somehow related to possible complexing between calcium, polysaccharides or other molasses components at high RDS levels is under investigation.

**Figure 15. Boiling heat transfer coefficient of Louisiana final molasses vs. consistency measured at 50°C and shear rate of 2 sec<sup>-1</sup>.**



**Figure 16. Effect of calcium content on viscosity (consistency) of final molasses measured at standard conditions, at four levels of refractometric dry solids (RDS).**

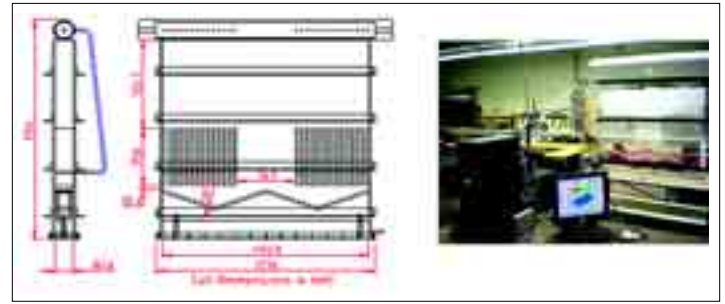


## Analysis of Fluid Flow in the Sugar Crystallization Process

This project developed with the LSU Mechanical Engineering Department involves the application of computational fluid dynamics (CFD) and modern anemometry techniques in the analysis of the fluid flow in vacuum pans, in an attempt to enhance the performance of pans by improving circulation.

A scaled test rig has been constructed to represent the major features of the fluid flow in vacuum pans (Figure 17). To emulate the process in a simplified and controllable manner, spargers are used to inject air into vertical channels that correspond to the calandria tubes. The injected air represents the water vapor, and the buoyancy resultant from density differences between liquid and gas phase is the circulation driving force. Water has been used initially to represent the liquid phase.

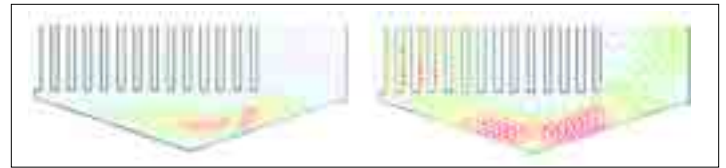
**Figure 17. Test rig and PIV system.**



To determine the flow field, Particle Image Velocimetry (PIV) has been applied. This anemometry technique is based in the use of a pulsed laser sheet and a special high resolution digital camera to follow tracer particles in the fluid; from their change of position, the flow velocity field can be determined. Accurate results for the liquid velocity have been obtained in the bottom region, while for the rest of the domain the high void fraction makes it difficult to measure the results corresponding to the gas phase flow (Figure 17). The average rise velocity of the bubbles has been determined to be 0.23–0.26 m/s, values consistent with the reported range of terminal velocities of air bubbles in water.

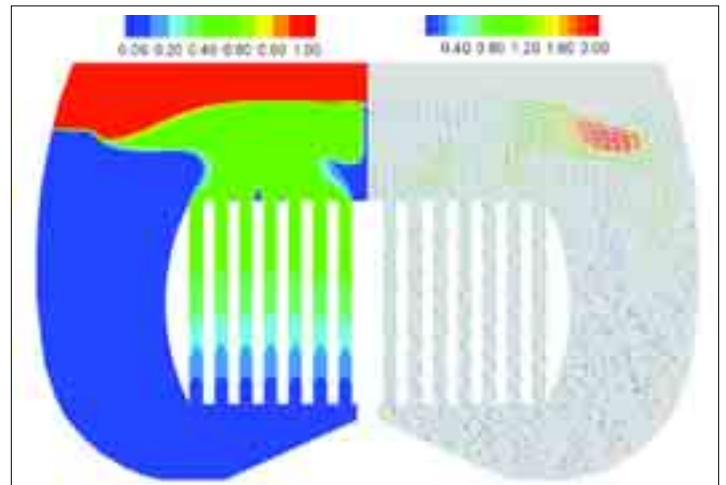
The fluid flow in the test rig is simulated using a commercial CFD code (Fluent). The comparison between the experimental and computational results shows that the simulations are overestimating the flow, predicting higher velocities and circulation than measured, although from a qualitative point of view similarities in the flow patterns are identifiable (Figure 18). Further development of the CFD analysis is in progress to improve the accuracy of the predictions.

**Figure 18. Velocity vectors measured with PIV (left) and liquid velocity vectors predicted with CFD (right).**



The application of CFD for the analysis of batch and continuous vacuum pans has been explored (Figure 19), obtaining results that help understand the process and demonstrate the capability to evaluate the effect of different geometric and operational parameters on circulation. The application of CFD is expected to play a significant role in the optimization of the design of vacuum pans.

**Figure 19. Void fraction and massecuite velocity in a continuous vacuum pan (CFD prediction).**



## Evaluation of C Crystallizers and C Massecuite Reheaters

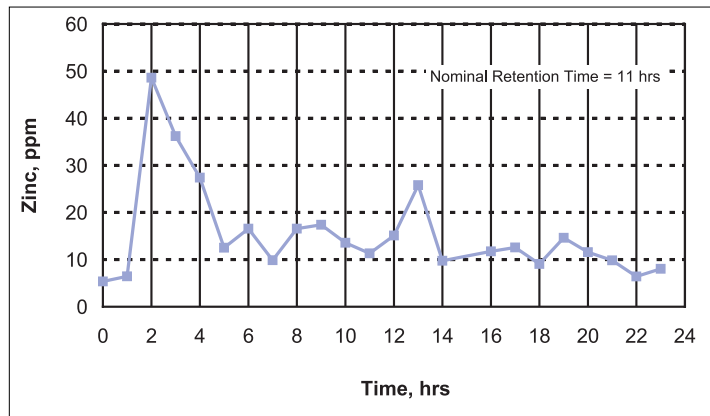
The data gathered in 2003 is a continuation of a project started two years earlier to evaluate continuous and batch pans, vertical and horizontal crystallizers and various types of massecuite reheaters. In 2003 data was obtained on pan crystal yields and crystallizer residence times using tracer tests as well as purity loss in reheaters and the low grade centrifugal station.

### Crystallizer Tracer Tests

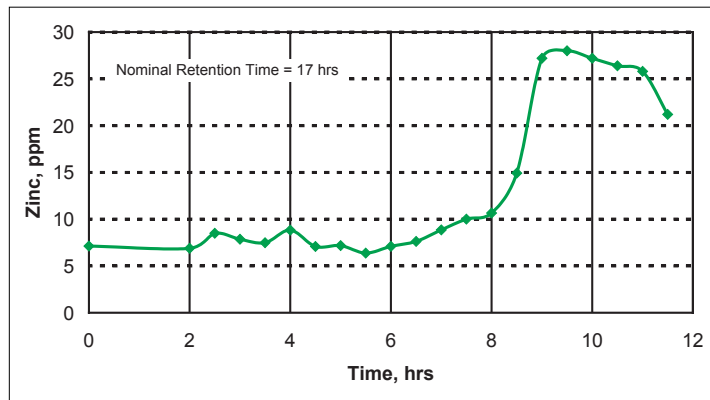
Tracer tests were conducted on three types of vertical crystallizers (Honiron, Silver and Fletcher Smith). The concentration of the tracer (zinc) in the massecuite leaving the crystallizer as a function of time after addition to the crystallizer inlet is shown in Figures 20, 21 and 22.

In the case of the Honiron crystallizer, the tracer peaked in 2 hours (nominal retention 11 hours). In the case of the Silver crys-

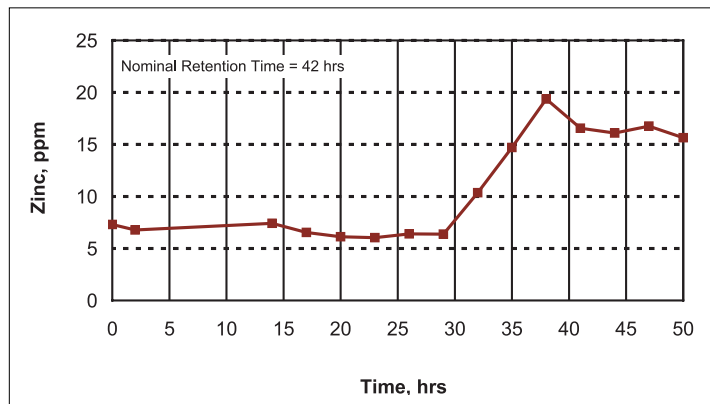
**Figure 20. Tracer Test Data on Honiron Vertical Crystallizer.**



**Figure 21. Tracer Test Data on Silver Vertical Crystallizer.**



**Figure 22. Tracer Test Data on Fletcher-Smith Vertical Crystallizer.**



tallizer, the tracer peaked in about 10 hours (nominal retention time 17 hours). The Fletcher Smith crystallizer tracer peak was reached after 38 hours (nominal retention time 42 hours). This shows evidence of severe short-circuiting in some crystallizers, which is ascribed to differences in crystallizer design.

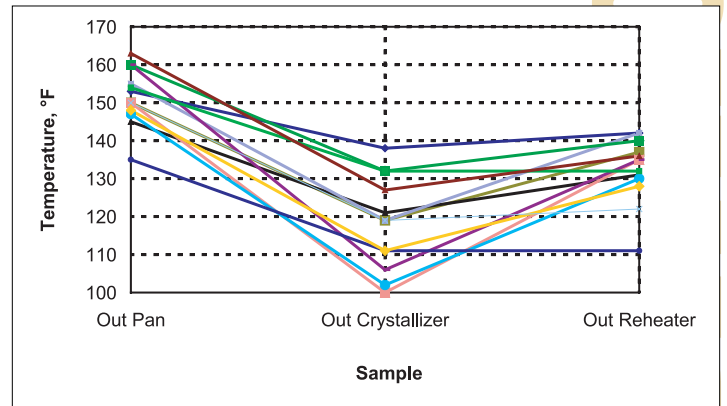
### Temperature Profile

Figure 23 shows massecuite temperatures out of the pan at strike, out of the coldest crystallizer and out of the reheater for a number of different units during the 2003-04 crop. Of note is the high average massecuite temperature leaving the pan of 152°F with a range of 135° to 163°F. The average massecuite temperature leaving the crystallizer of 118°F is also high.

### Purity Profile

Figure 24 shows the apparent C massecuite purity and the cor-

**Figure 23. Massecuite Temperature Profile.**



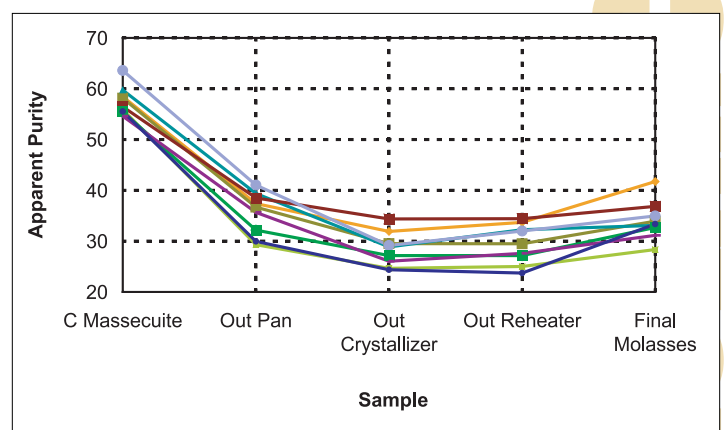
responding cyclone (Nutsch) purities of the pan mother liquor, mother liquor out of the coldest crystallizer, mother liquor out of the reheater and the final molasses purity from the factory centrifugals. Of interest is the very high average massecuite purity at striking (57.5) with a range of 54.6 to 63.6. The cyclone (Nutsch) purity at the pan averaged 35.6, 28.5 out of the crystallizers, 29.5 out of the reheater and 34.0 out of the factory centrifugals.

Crystallizer cooling lowered the mother liquor purity by an average of 7.1 purity points, while there was an average purity gain of 1.0 across the reheater and a further average purity rise of 4.5 across the centrifugals.

### Viscosity Profile

Viscosities were measured using a Brookfield model RVDV-

**Figure 24. Apparent Purity Profile of C Massecuite.**



II+, spindle number 7 viscometer. Figure 25 shows the viscosity of the C massecuite at striking at pan temperature, the viscosity of the C massecuite out of the coldest crystallizer at the crystallizer temperature and the viscosity of the reheated massecuite at the reheated massecuite temperature. Massecuite viscosities at striking varied from 42,000 to 724,000 cP. The viscosity of the massecuite leaving the coldest crystallizer varied from 195,000 to 2,600,000 cP, while the viscosity of the reheated massecuite varied from 68,000 to 1,200,000 cP. The increase in mother liquor purity across the centrifugals appears to be highly dependent on the viscosity of the massecuite being centrifuged.

#### Massecuite Reheater Performance

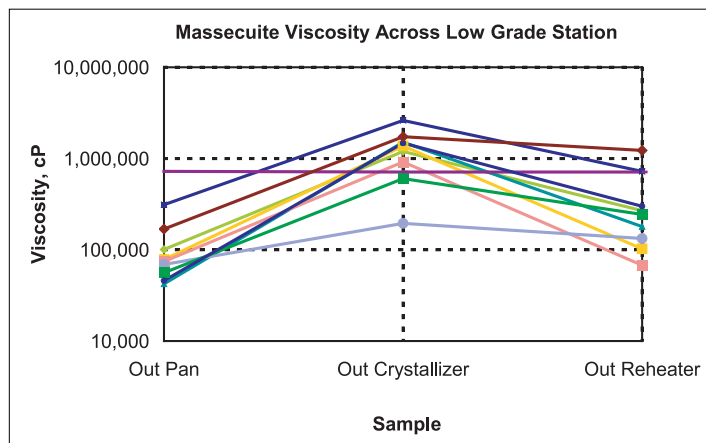
Data on the performance of three large stationary reheaters (one Honiron and two Fletcher Smith units) and one small Stevens coil reheater are shown in Table 4.

All reheater types resulted in small purity rises in the mother liquor (0.4 to 0.8 purity points). Of interest is the limited re-solution for the Stevens coil in spite of a very high reheating water temperature (167° F). The heat transfer coefficient for the Stevens coil was more than twice as high as for the stationary cooling element reheaters. These results are single tests and further measurements under varying conditions still need to be done for the comparison.

Table 4: Summary of Massecuite Reheater Performance.

Type	Heating Surface, Sq.Ft	Massecuite Cu.Ft./Day	Massecuite In °F	Massecuite Out °F	Water In °F	Water Out °F	Heat Transfer Coeff. Btu/hr/ft <sup>2</sup> /°F	Purity Rise
HON	7,534	15,785	113	138	155	150	3.9	0.7
F S	5,436	10,500	104	134	150	134	4.9	0.8
F S	7,350	9,859	132	140	146	142	2.7	0.4
STE	595	9,179	117	130	167	164	9.1	0.5

Figure 25. Viscosity Profile of C Massecuite.



Views of the workshop at Audubon Sugar Institute.



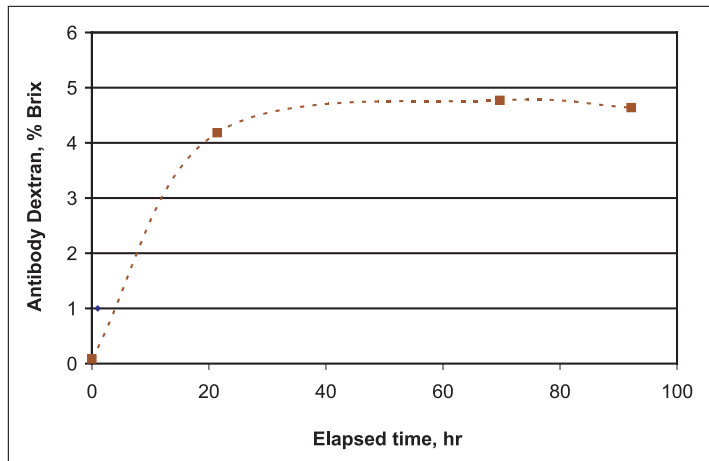
## Pol-Sucrose Measurements and Dextran

With the advent of non-lead juice clarifiers, pol measurement of raw sugar occasionally read higher than is theoretically possible. Dextran possessing an optical rotation of  $[\alpha]_D = +199$  can cause polarimetric sucrose assays to appear artificially high.

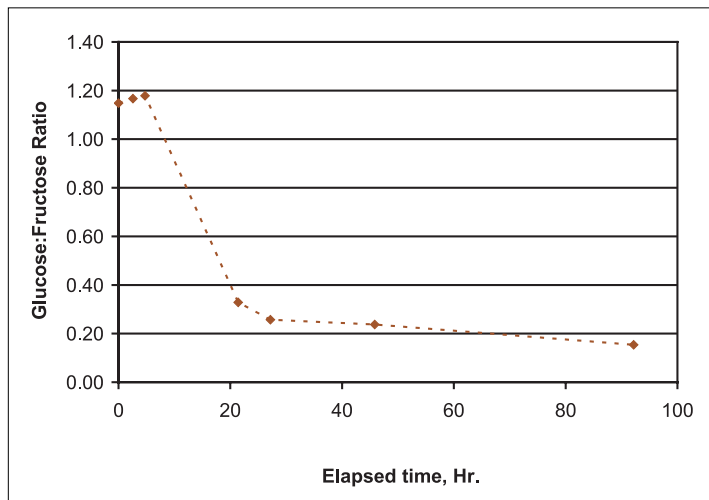
The molecular weight of the dextran has little effect on the optical rotation. The deviation in optical rotation for dextrans ranging from 10 to 2000 kDa was  $\pm 0.002^\circ Z$ . On average, the overestimation of pol in raw sugar due to dextran was  $0.10^\circ Z/100\text{ppm}$ .

Raw sugars can contain significant amounts of reducing sugars (glucose and fructose) that influence the pol. At full mutarotation, the contribution of glucose to optical rotation can be given as  $\Delta^\circ Z_{gl} = 0.0003 \text{ glucose}(\text{ppm}) - 0.0117$ , and, for fructose,  $\Delta^\circ Z_{fr} = 0.0005 \text{ fructose}(\text{ppm}) - 0.0036$ . When these terms are included, a good prediction of pol error can be calculated. The same calculation does not work for juice. This is not surprising, because constituent-reducing sugars most frequently meet or exceed 1%. The ratio of glucose to fructose is  $\sim 1:1$  in non-deteriorated sugar cane juices. This ratio changes inversely with dextran in deteriorating juices (Figures 26 & 27).

**Figure 26. Dextran synthesis with respect to time, as observed in unpreserved juice samples held at 30°C. Results were acquired using an antibody method.**



**Figure 27. Behavior of the glucose: fructose ratio with respect to time. The response is seen as fructose accumulates during dextran synthesis.**



*Audubon Sugar Institute sign at building entrance.*

It was possible to correlate the quantities of glucose and fructose to the amount of dextran and derive an equation to correct the pol values in juice.

$$CP_{appx} = OP + \Delta^\circ Z_{Fru} - \Delta^\circ Z_{Dex} - \Delta^\circ Z_{Glu}$$

Where:

CP = Corrected Pol Sucrose, % Juice

OP = Octapol Sucrose, % Juice

$$\Delta^\circ Z_{Fru} = -0.0012x - 1.3904$$

$$\Delta^\circ Z_{Dex} = (A_1 e^{(-x/t_1)} + A_2 e^{(-x/t_2)} + y_0) - 99.676$$

where x is dextran (ppm on Brix); and  $y_0$  is a constant

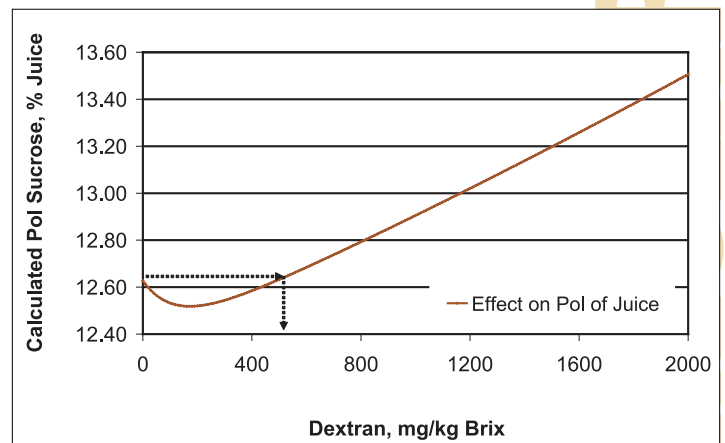
$$\Delta^\circ Z_{Glu} = 0.0005x + 0.9193$$

This equation was validated against laboratory acquired data for composite juice samples.

Using this equation, the “true” sucrose may be closely approximated if Octapol sucrose % juice and dextran ppm/Brix are known. An HPLC is not required. The equation appears to lose utility as dextran quantities exceed 2000 mg/kg Brix.

As glucose is consumed by microorganisms, pol will fall. As micro-organisms form dextran from sucrose, the pol will start to rise. The pol sucrose should break even with the “true” sucrose at  $\sim 500$  mg dextran /kg Brix. Beyond this concentration, pol overestimation occurs. Further investigations are under way and include possible corrective terms for mannitol and chemical loss, further replicates and a general simplification of the equation. An example of the effect of increasing dextran concentration on pol gives a plot as shown in Figure 28.

**Figure 28. Behavior of pol sucrose in juice as dextran concentration increases.**



## Mixed Juice and Syrup Analyses

Audubon conducted analyses on four mixed juice and four syrup composite samples each week. Three factories provided both a juice and a syrup sample; two other factories provided only syrup or juice. The Raceland mill is using the data to establish a more accurate recovery based on sucrose, rather than pol. Work was done with St. James and Raceland to calculate heat transfer coefficients on evaporator trains using this data and data provided by the factories.

Table 5 shows the averages of all data for the season for both syrup and mixed juice. It can be seen that the true purity is higher in the syrup than in the mixed juice; this is a result of C magma recirculation. All of the ratios displayed in the table are the same, within experimental error. Glucose/sucrose ratio is an indication of inversion, but the recirculation of C magma masks any change. Next season, efforts will be made to acquire syrup samples before C magma is added.

**Table 5: 2003 Juice and Syrup Data – season averages.**

	Ref. Brix %sample	App. Purity %	True Sucrose %sample	Ratio pol /sucrose	True Purity %	Fructose F %sample	Glucose G %sample	F/G Ratio	Cond. Ash %sample	(F+G)/ Ash
Juice	14.2	83.6	12.4	0.965	86.7	0.35	0.32	1.08	0.58	1.16
Syrup	64.7	87.5	58.5	0.967	90.6	1.41	1.35	1.04	2.46	1.14

## Final Molasses Survey Results for the 2000-2003 Grinding Seasons

Audubon reintroduced the final molasses survey for the 2000 season to assess actual losses in final molasses more accurately. The normal measurements used in a sugar mill laboratory are not accurate enough at the low purities associated with final molasses to determine losses acceptably. Also the measurement of reducing sugar levels is essential to determine achievable exhaustion.

Over the past four seasons, Audubon has evaluated how it handles and analyzes these samples and worked to improve every aspect of the final molasses survey to increase both accuracy and turn-around time. Part of this evaluation has been the continued practice of running all analyses blindly with weekly check samples. Table 6 contains standard deviation data on select analyses and calculated results on the check samples, which show progress made.

**Table 6: Standard Deviations of Key Components of Check Samples.**

Year	Ref. Brix	App. Purity	True Sucrose	True Purity	Target Purity	T. P. Diff.
2000	1.01	0.89	1.14	1.19	0.37	1.36
2001	0.44	0.66	0.49	0.69	0.14	0.68
2002	0.27	0.77	0.32	0.40	0.11	0.44
2003	0.15	0.68	0.42	0.54	0.14	0.56

The average weekly TPD values for the 2000-2003 seasons are plotted with the weekly averages from the past seasons in Figure 29. Expected trends due to startup and liquidation and cane maturity continued. Table 7 summarizes the average target purity, TPD, F/G ratio, F+G, (F+G)/ash ratio and ash for the past four

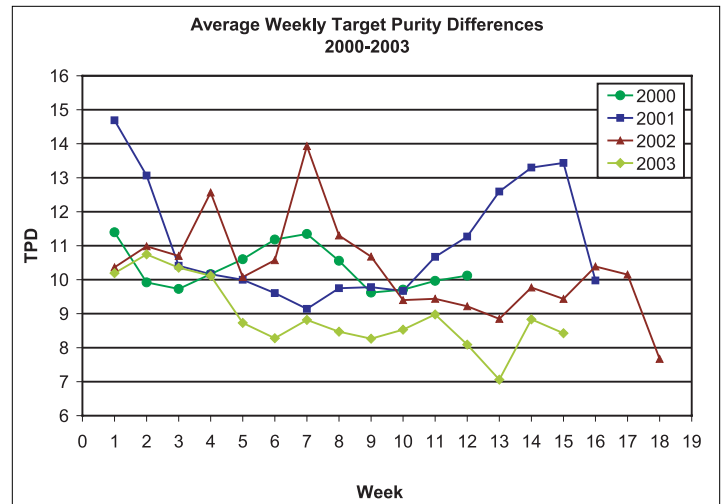
**Table 7: Final Molasses Survey Data Summary for 2000-2003.**

	2000	2001	2002	2003
TP	35.5	33.3	34.0	33.6
TPD	10.2	10.5	10.4	8.9
F/G	1.68	1.41	1.44	1.33
F+G, %	11.8	15.3	14.3	14.7
(F+G)/Ash	0.78	1.14	1.00	1.07
Ash, %	15.4	13.7	13.8	14.0

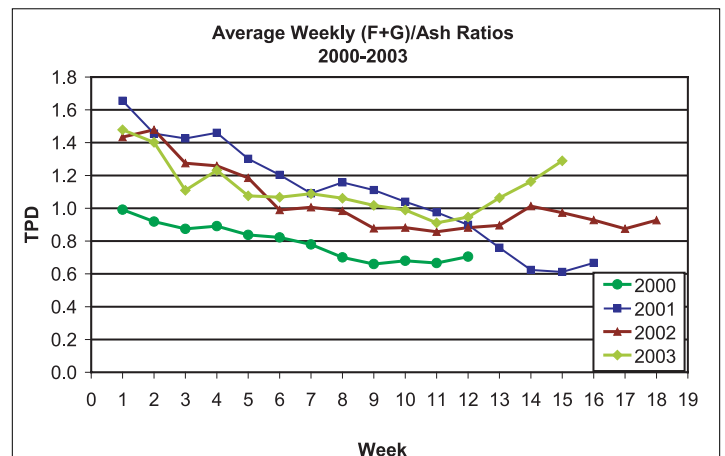
seasons. The average TPD for 2003 was significantly lower than in the previous three seasons. In Table 7, it can be seen that the F/G ratio has dropped from 1.7 to 1.3 from 2000 to 2003. 2001 and 2002 were basically the same, but this is not surprising considering the extreme conditions for 2002. Plotted in Figure 30 are the average weekly reducing sugars to ash ratios for the past four seasons. The average weekly Pol/sucrose ratios are shown in Figure 31. Lower TPD values illustrate that the factories are extracting more of the available sugar and lower F/G ratios indicate that Maillard reaction has decreased, improving exhaustion conditions.

The ash percent molasses and total reducing sugars have

**Figure 29. Weekly Target Purity Difference for the last 4 years.**



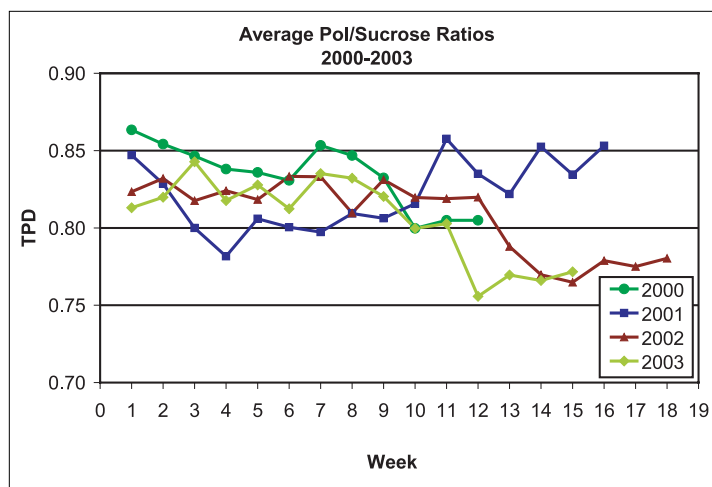
**Figure 30. Weekly Average Reducing Sugars to Ash Ratios for the last 4 years.**





Stella Polanco, research associate, in the Analytical Laboratory.

**Figure 31. Average Pol to Sucrose Ratios in Molasses for the last 4 years.**

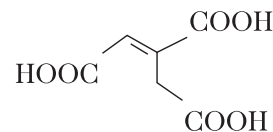


remained relatively constant for the past three seasons, resulting in basically the same target purity. Note the significant difference in target purity from 2000 to the other seasons in Table 7. In 2000 reducing sugars were lower and ash was higher, resulting in a target purity that is one and a half to two points higher than in any of the three following seasons. This point demonstrates the need to use target purity as a benchmark, since seasonal variations caused by different climatic and growth conditions can cause significant difference in the amount of sucrose that can be extracted from cane juice.

The 2003 season is a stark contrast from the 2002 season. 2003 was dry compared to all of the last four. The reduction in both TPD and F/G ratio is evidence of improvements in the Louisiana sugar industry. Certainly favorable weather contributed to these reductions. It can be concluded that mill personnel are starting to take an interest in C station operation. Pan automation is also a contributor to these reductions. The final molasses survey and other services have assisted in these improvements and will continue to help in the future. Although TPD values have dropped, considerable room for improvement still exists.

## Extraction of aconitic acid from cane waste biomass and its application in flexible PVC formulations

There is an opportunity to develop a market within the chemical industry for bio-derived (trans-) aconitic acid (1,2,3-propenetricarboxylic acid).



*trans*-aconitic acid

The acid is a major component of sugarcane, and its recovery from molasses has been a subject of a number of studies, pilot and even semi-commercial production. Last year, in cooperation with other researchers at LSU, a program was initiated to explore a new route for its recovery from waste biomass from cane production, its esterification and application in flexible PVC formulations.

No information is available on the production costs of the acid from cane biomass using this route, the production costs and markets for the aconitic acid intermediates (esters) or the properties of the aconitate acid ester - PVC blends. However, the proof-of-concept work has been very encouraging. The feasibility has already been demonstrated of extracting the waste biomass, ethanol fermentation of the extracted juice, esterification of the acid to tributyl aconitate (TBA) and formulating TBA with PVC. Formulations with up to 1:2 TBA:PVC were produced in our laboratory. For comparison, blends were also prepared with commercially available esters of phthalic and citric acids to assess benefits from substituting those with sugarcane-based plasticizers.

Of the targeted applications, the most imminent could be as a replacement in specialty products for the conventional phthalate-based plasticizers benzyl butyl phthalate, diethyl phthalate, di-n-butyl phthalate, diisononyl phthalate and diisodecyl phthalate. There is a growing debate about the potential health implications of the chemicals that leach from plastics. The most frequently used plasticizer, bis (2-ethylhexyl) phthalate, migrates at a constant rate to the environment. It has been detected in water, soil and food and is therefore considered a widespread environmental contaminant.

Preliminary results indicate that tri-butyl aconitate is more effective in lowering the glass transition of PVC than tri-butyl ester of phthalic acid.

**Figure 32. PVC with about 30% of the tributylaconitate plasticizer.**





## Extension Work

### Ash % Bagasse

Ash % bagasse was determined for samples collected by the American Sugar Cane League as part of a harvester fan speed study. Ash % bagasse was determined for numerous boiler efficiency tests.

### Boiler Tests

Boiler efficiency tests were performed at several factories where natural gas consumption was higher than normal and/or where higher efficiency was desired. An official boiler emissions test was monitored at one factory that installed a new boiler for the 2003 crop.

### Preparation Index and Milling Tests

The preparation index was measured at four factories this year. Mill pol extraction was determined for each mill in the tandem at one of those mills. Two factories built their own preparation index tumbling units in 2003.

### Core Lab

Two factories requested help in reviewing their core lab procedures. Direct cane analysis at one core lab verified the factory's core lab results; suggestions were given to the other factory for improving its procedures. A presentation and tour was given to county agents at another factory on understanding core lab data and core lab procedures.

### Undetermined Losses/Overall Chemical Control

A few factories requested a review of their chemical control and undetermined losses. Independent checks on key analyses were performed, as well as determination of losses in cane wash water and in condenser water.

### Cold Tolerance Tests

Audubon collaborated with LSU AgCenter Extension and USDA personnel to monitor the rate of deterioration of various cane varieties after the freeze in January.

### Effect of Harvester Operations

Audubon helped to analyze samples for theoretical recoverable sugar from harvester pour rate tests conducted by USDA and American Sugar Cane League personnel. Assistance was also given at Raceland mill in a trial to compare clean vs. trashy cane.

### Billet Preservation

Audubon participated in billet preservation tests conducted at one factory where the effect of biocide treatments to preserve billeted cane was measured.

### Enterprise Mill Equipment Modifications

A new design of evaporator feed ring on a pre-evaporator was tried with success.

Assistance was given in the design of pan condensers and an aftercooler to improve the vacuum in the C continuous pan.

Modifications to a number of the batch pans were suggested to improve performance.

### Evaporator Simulations

Calculations investigating different options for expanding the Raceland evaporator capacity were undertaken. The most appropriate options involved installing a new first effect and changing the duties of some of the other vessels. Increasing vapor bleed from the second effect was recommended to improve capacity.



*Dr. Harold Birkett, associate professor, presenting his work to the Louisiana sugar millers at an annual factory operations seminar. Birkett has long been involved in Audubon's extension services.*



*Chromatography equipment in the Analytical Laboratory.*

## Audubon Sugar Institute Analytical Capabilities

ASI has seven operational HPLC systems, three of which are ion chromatography units.

One unit is set up for cation analyses and two others for anion analyses including organic acids. Another system is a GPC unit for dextran analyses with both a UV-visible and a refractive index detector and is used primarily for a project to study color in sugar solutions. ASI also has an HPAEC-PAD anion exclusion chromatography unit used for

alcohol, sugar and oligosaccharide analyses. One HPLC is used for routine sugar analyses on juice, syrup and molasses and other process samples. Another HPLC is dedicated to a specific project requiring extensive sugar analyses.

Other instrumentation includes a refractometer with 0.01 Brix resolution and temperature compensation capabilities, a polarimeter with 0.01°Z resolution and both 589 and 880 wavelength for sucrose by Pol and a conductivity meter with temperature compensation for conductivity ash. A gas chromatograph (GC) was purchased and installed and used mainly in analyses of alcohols and biogas. Additionally, three GCs were donated to ASI. These instruments will be installed or traded for other equipment.

ASI purchased a Near-Infrared (NIR) transmittance spectrophotometer for the analysis on molasses samples in the final molasses survey and factory calibrations. The addition of a new Shimadzu 6650 GF AA has also augmented ASI's analytical capability. This modular instrument comprises a spectrophotometer, graphite furnace and an auto-sampler, making the sequential analysis of up to 60 samples possible. It is used for the detection of silicon and calcium in the study of evaporator scaling and clarification studies using soda ash. There is also potential for other uses such as tracing residence time in sugar process equipment.

With the added space at our new facility, Audubon can use equipment and accessories that were previously stored. One of these is a chiller that was donated to provide the facility with a constant temperature water bath for conductivity analysis. Although the new conductivity meter has a temperature compensation feature, analyzing samples at the proper temperature is still preferred. Column ovens will be acquired for the anion units to maintain a more consistent column and detector cell temperature.

The analytical lab personnel at ASI are experienced and capable of maintaining and operating all the equipment. Continuous effort is made by the team to improve the quality of the analytical equipment, to improve reliability and quality control, and to maximize the capability at ASI.



*The newly acquired Shimadzu Atomic Absorption spectrophotometer was set up to analyze Si and Ca in samples of evaporator scale and mixed and clarified juices.*

## LSU AgCenter, Audubon Sugar Institute Short Courses

In 2003, the Audubon Sugar Institute short course program offered both spring and fall sessions. These courses qualify for Continuing Professional Development for registered professional engineers but are designed to increase knowledge of people at all levels in the sugar industry. The participants represented not only the Louisiana sugar mills but also came from the sugar industry in Central America as well as sugar industry suppliers and customers.

Two courses were given by international experts from outside the USA. A vacuum pan instrumentation and control course was conducted by Dr. David Love from South Africa, and a course on cane preparation and milling by Rod Cullen of Australia.

All courses offered at Audubon Sugar Institute can be viewed online at [www.lsuagcenter.com/audubon/](http://www.lsuagcenter.com/audubon/)

### **Vacuum Pan Instrumentation & Control – One day**

The main objective of this course was to provide a good understanding of the practical principles of automatic control of vacuum pans to maximize the performance and capacity of the pan station.

### **An Introduction to the Technology of Sugar Production – Two days**

A comprehensive overview of all cane-processing operations is covered in this introductory course to provide an understanding of the process from growing cane to final product. It also covers the important aspects that affect sugar production and factors taken into account in running a mill efficiently.

### **Polysaccharides and Dextran**

Sugar mill chemists and factory operations managers were targeted in covering the range of carbohydrates referred to as polysaccharides that affect sugar processing and their removal and control. The latest analytical techniques to measure these polysaccharides, in particular dextran, were introduced.

### **Continuous Vacuum Pan Boiling – One day**

This course provided an understanding of continuous pan boiling and how it compares with batch processing. It was intended for factory operations personnel and those involved in the design and planning of factory modifications and expansions.

### **An Introduction to Sugar Refining – Two days**

A comprehensive coverage of the important aspects in sugar refining was offered for individuals new to the sugar refining industry and for suppliers to or customers of the sugar industry. The course materials offered participants better understanding of issues related to capacity, costs, losses and product quality.

### **Technology Refresher: Improving Raw Sugar Factory Operations and Profitability – Two days**

This refresher course was introduced in 2002 for individuals involved in the operation or the design of raw sugar mills to enhance their technical skills and get up to date with important issues that affect the efficiency and profitability of raw cane sugar production.

### **Cane Preparation and Milling – Three days**

Modern cane preparation and milling technology was introduced in this course for engineers involved in running a sugar factory or project engineers associated with the industry to gain an understanding of the principles that can be applied to improve their own factory extraction, or make the correct choice of plant and equipment. Participants also had the opportunity to discuss problems and identify possible solutions.

### **Bagasse Boiler Operation – Two days**

The course objective was to provide boiler plant operations personnel with an understanding of the role of the boiler plant in the raw sugar factory and the factors that affect boiler capacity, efficiency and air emissions. Emphasis was given to bagasse boilers although discussions also covered natural gas boilers. Combustion calculations and use of charts and tables to estimate boiler efficiency were incorporated in the course materials.

### **Chief Chemist – Four days**

This course was designed for chemists, process engineers and managers who need to understand how factory manufacturing reports are calculated, interpreted and checked against theoretical considerations. The training involved hands-on report calculations, use of computers in the preparation, correction and checking of the reports and use of the report data to analyze factory operations.

### **Bench Chemist – Four days**

This course was developed to suit newly employed bench chemists in a raw sugar factory who are unfamiliar with sugar mill analyses. A brief overview of the process involved in raw sugar manufacturing and why the analyses done are important was given. Hands-on training included handling of a sample mill to obtain juice and bagasse samples, performing the various analyses, calculating the results and maintaining and calibrating the lab instruments.

### **Introductory Sugar Boiling – Three days**

This class offered individuals with little or no experience in sugar boiling an overview of the sugar boiling process. It not only provided an understanding of the principles involved but taught how to boil pans and allowed hands-on trials in making sugar on ASI's pilot plant.



## Degree Courses for Sugar Engineers at LSU

Audubon Sugar Institute offers both graduate and undergraduate education with sugar engineering as an area of specialization or a minor in conjunction with other engineering degree majors.

The Institute also provides part-time work in the analytical lab and factory for undergraduate students minoring in sugar engineering and graduate assistantships to students pursuing a master's degree in chemical engineering, biological engineering, mechanical engineering and food science. With the increased number of research projects approved for funding and added space at the new facility, Audubon has enhanced its capability for more graduate students to attain expertise in a variety of aspects of sugar processing.

### Sugar Engineering Courses

Two courses introduced two years ago have continued to be offered by the College of Engineering. They are a course in Sugar Process Engineering, which teaches all the background to sugar processing (BE 4342), and Sugar Factory Design (BE 4347), which introduces the student to the detail of equipment design in a factory. Prerequisites are listed on our Web site at [www.lsuagcenter.com/audubon](http://www.lsuagcenter.com/audubon).

### Minor in Sugar Engineering

The objective of this program is to produce a graduate ideally suited to the operation and management of a sugar-producing facility. In all cases, the student is encouraged to work as a part-time student at ASI, with maximum exposure to sugar processing at every opportunity.

A set of courses has been prescribed so that students studying in biological, chemical or mechanical engineering may earn a minor in sugar engineering by choosing the two sugar courses described above and their electives to meet the requirements. If possible the student should also choose a design project in a sugar engineering related topic overseen by the staff of the Audubon Sugar Institute. Sugar Process Engineering should be taken in the junior year.

To earn the minor, students in the College of Engineering must complete 18 hours of required courses with a grade of "C" or higher: Visit our Web site at [www.lsuagcenter.com/audubon](http://www.lsuagcenter.com/audubon) for the list of required courses.

Another option involves spending the fall semester at an operating sugar mill, using this period as an internship and earning 3 credit hours (allowed for in BE 3249). This could substitute for one of the required courses, but would probably require a slightly longer time in which to complete the degree.



*Giovanna Dequeiroz, graduate student, conducting experiments on her new biocide.*

### Master's in Chemical, Mechanical or Biological Engineering Specializing in Sugar Engineering

These master's degrees are targeted at people who have a first degree and wish to gain specific sugar processing expertise through further study. Following the thesis option, the master's academic requirement is 24 credit hours in the nominally 21-month program. The course requirements include the Sugar Process Engineering and Sugar Factory Design courses listed above. Depending on the first degree of the individual concerned, he or she would be encouraged to choose electives appropriate to supporting a strong sugar expertise.

A thesis is required related to a relevant sugar-processing topic. In all cases, the student is encouraged to take on a research assistantship in ASI for the duration of the course and is given maximum exposure to sugar processing at every opportunity.

### Sugar Engineering Courses as Condensed, Intersession or Continuing Education Courses

As an alternative or in addition to the normal course arrangement, consideration will be given to condensing the sugar engineering courses into full-time intensive courses held over a three-week period during the summer semester. These courses could be opened to outsiders through a continuing education program.



## Library

The Audubon Sugar Institute Library received book donations in 2003-2004 to add to its growing collection of books from St. Mary Sugar Co-op in memory of the late W. Bradley Kimbrough Jr. and from Cornelius J. Laan of Sugar Knowledge International Ltd. (SKIL). The library now offers a good selection of the most up-to-date books on sugar processing and engineering fundamentals. It also acquired a wide range of chemical engineering and chemical journals and abstracts that came with the new facility in St. Gabriel donated by Syngenta Crop Protection Inc.

## Publications

Birkett, H.S. (2003) Core Lab Sugar Prediction Factory Performance and Liquidation Factor. *Sugar Bulletin*. 81,12: 14-16.

Broadhurst, H.A., Rein, P.W. (2003) Modeling Adsorption of Cane Sugar Solution Colorant in Packed-bed Ion Exchange. *AIChE Journal*. 49, 10: 2519-2532.

Day, D.F., Ott, C.M., Mayo, J.A., Kim, D. (2004) A Multi-Component Safe Biocidal Complex. US Patent 6,692,757. Feb 17, 2004.

Day, D.F. (2004) A New Biocide. US Patent pending, filed Oct 2003.

Day, D.F., Chung, C. H. (2004) A Neutraceutical. US Patent pending, filed May 2003.

Day, D.F., Cuddihy, J., Rauh, J. (2003) Versatility of the Antibody Dextran Test Method. *Journal of Amer. Soc. of Sugar Cane Technol.*, 23: 108.

Day, D.F., Kampen, W. (2003) Organic Acids in the Sugar Factory Environment. *Journal of Amer. Soc. of Sugar Cane Technol.*, 23: 111.

Kang, H.K., Lee, J.H., Kim, D., Day, D.F., Robyt, J.F., Park, K. H., Moon, T.W. (2004) Cloning and expression of *Lipomyces starkeyi*  $\alpha$ -amylase in *Escherichia coli* and determination of some of its properties. *FEMS Microbiology Letters*, 233, 1: 53-64.

Kim, D. and Day, D.F. (2004) Determination of Dextran in Raw Sugar Process Streams. *Food Science and Biotechnology*. 13, 1: 1-5.

Rein, P.W. (2003) Optimum Use of Water and Condensate in a Sugar Mill. *Sugar Bulletin*. 82, 1:11-14.

Rein, P.W. (2003) Cane Preparation - Knifing vs. Shredding. *Sugar Bulletin*. 81, 11:16-18.

Rein, P.W. (2003) The importance of Achieving a High Crystal Content in High Grade Masseccutes. *Sugar Bulletin*. 81, 8: 12-17.

Rein, P.W., Acharya, S., Echeverri, L.F. (2004) Circulation in Vacuum Pans. *Journal of Amer. Soc. of Sugar Cane Technol.*, 24: 1-17.

Rein, P.W. (2004) Instrumentation and Automatic Control. *Sugar Bulletin*. 82, 7: 11-12.

Rein, P.W. (2004) Education and Training of Engineers for the Sugar Industry. *Sugar Bulletin*. 82, 6: 10-12.

Saska, M. (2003) Heat Transfer Rates in Boiling of Cane Syrups and Molasses & the Phenomenon of 'Hard to Boil' Masseccutes. SIT Conference. Hamilton Island, Australia, May 4-7.



*Dr. Michael Saska was one of the 11 LSU AgCenter inventors honored at a recognition reception at the Lod Cook Conference Center on the LSU campus Sept. 11 for patents or plant variety protection certificates received in 2002. The ceremony was part of LSU AgCenter Patent and PVP Club that was created to showcase the achievements of inventors who develop exciting and promising new innovations. Dr. Saska was recognized for receiving a patent on "Process for Separation of Sugar."*



*Drs. Chang Ho and Day were awarded the best paper in the manufacturing section at the ASSCT conference held in Destin, Fla., June 25-27, 2003. Their research paper was titled "Glucopoligosaccharides from Leuconostoc mesenteroides B-742 (ATCC 13146): a potential prebiotic."*

## Representation on Technical Societies and Research Institutes

### ISSCT (International Society of Sugar Cane Technologists)

Executive Committee and Immediate Past Chairman: P.W. Rein  
Co-Products Section Committee: D.F. Day

### SPRI (Sugar Processing Research Institute)

P.W. Rein (Member of Board of Directors)

### International Sugar Journal

M. Saska (Referee), L. Bento (Referee)

### American Chemical Society

D.F. Day, L.R. Madsen, B.E. White

### American Society of Microbiology

D.F. Day, C.H. Chung

### American Institute of Chemical Engineering

P.W. Rein, M. Saska, M.A. Tessier

### American Society of Mechanical Engineers

J.W. King

### American Society of Advancement of Science

D.F. Day

### Society for Industrial Microbiology

D.F. Day

### Southern Region Development Committee – USDA

D.F. Day

## Meetings, Conferences and Workshops Attended

### April 2003

ASI Annual Conference (all staff and faculty)

### May 2003

SIT, Hamilton Island, Australia (P.Rein)  
Australian Society of Sugar Cane Technologists (P.Rein)

### June 2003

ASSCT, Destin, Fla. (P. Rein, D. Day, H. Birkett, J. King, D. Wood, L. Madsen, J. Stein)

### October 2003

MSIRI 50<sup>th</sup> Anniversary, Mauritius (P. Rein)

### November 2003

Southern Region Development Committee, Washington, D.C. (D.F. Day)

### February 2004

ASSCT (La. Division) Baton Rouge, La. (all staff and faculty)

## Foreign Visitors to the Institute

### April 2003

Mario Fremllay (Canada); Dr. David Love (South Africa)

### May 2003

David Beddie (Germany); Tobias Wirth (Germany)

### June 2003

Rod Cullen (Australia)

### July 2003

Klaus Niepoth (Germany); M. Narendraneth (India); G.V. Prasad Rao (India)

### October 2003

M. Kebede (Ethiopia); Tesfaue Ashete (Ethiopia); Deresi Gutema (Ethiopia); Elias Teofaye (Ethiopia); Haige Girmay (Ethiopia); Eduardo Munhoz (Brazil)

### December 2003

Aurelio Bezerra (Brazil)

### January 2004

Mike Getaz (UK); Bill Wiseman (UK)

### February 2004

Minister of Trade and Commerce & Ambassador (Mali); Yuan-ai Wei (China); Terry Kirkpatrick (UK)

### March 2004

Jose Barraza (Colombia); Fernando Gamez Rodriguez (Mexico); Dr. Jose Fernandez Abella (Mexico)





## Faculty and Staff

### Administrative Staff

**Dr. Peter Rein**, Professor and Head, B.Sc. and M.Sc. Chemical Engineering (University of Cape Town, South Africa), Ph.D. Chemical Engineering (University of Natal, South Africa)

**Jane Crawford**, University Administrative Specialist

**Maggie Matherne**, Secretary

**Melati Tessier**, Research Associate Specialist, B.S. Chemical Engineering (Louisiana State University)



### Analytical Lab

**Brian White**, Research Associate –Analytical Chemist, B.S. Chemistry (Freed-Hardeman University)

**Stella Luz Polanco Duque**, Research Associate, B.S. Chemical Engineering (Universidad del Valle, Colombia)

**Lee Madsen**, Research Associate, B.S. Chemistry (Louisiana State University)

**Cheryl Swearingen**, Temporary Analytical Lab Assistant, B.S. Zoology (Louisiana State University)

**John Daigle**, Student Worker, B.S. Biology (Nicholls State University)

### Factory Staff

**Julie King**, Research Associate – Factory Manager, B.S. Mechanical Engineering (Louisiana State University)

**Scott Barrow**, Research Associate – Electronics/Instrumentation Engineer, B.S. General Studies (Alpena Community College)

**Lamar Aillet**, Maintenance Foreman

**Michael Robert**, Student Worker

**Scott Louque**, Student Worker

## Faculty and Staff

**Dr. Donal Day**, Professor, B.Sc. Biochemistry (University of New Hampshire), Ph.D. Microbiology (McGill University, Canada)

**Dr. Michael Saska**, Professor, B.S. Chemical Engineering (Prague Institute of Chemical Technology, Czechoslovakia), M.S. Chemical Engineering (Louisiana State University), Ph.D. Chemical Engineering (Georgia Institute of Technology)

**Dr. Luis Bento**, Professor, B.S. Chemical Science (Coimbra University, Portugal), Licenciante in Engineering, Chemical Engineering (University of Porto), Ph.D. Chemical Engineering (Minho University, Portugal)

**Dr. Harold Birkett**, Associate Professor, B.S. Chemical Engineering (Louisiana State University), M.S. Chemical Engineering (Louisiana State University), Ph.D. Chemical Engineering (Louisiana State University)

**Jeanie Stein**, Research Associate, B.S. Plant Science (Nicholls State University)

**Stuart 'Lenn' Goudeau**, Research Associate, B.S. Industrial Technology (Louisiana State University)

**Niconor Reece**, Graduate Assistant, B.S. Biological and Agricultural Engineering (Louisiana State University)

**Dr. Chang-Ho Chung**, Post-doctoral Researcher, B.S. Food Science (Sejong University, Korea), M.S. Food Science (Sejong University), Ph.D. Food Science (Louisiana State University)

**Giovanna Dequeiroz**, Graduate Assistant, B.S. Food Science (Clemson University), M.S. Food Science (Clemson University)

**Duwoon Kim**, Graduate Assistant – B.S. Food Science (Chonnam National University, Korea), M.S. Food Science (Louisiana State University)

**Luis Echeverri**, Graduate Assistant, B.S. Mechanical Engineering (Universidad del Valle, Colombia)

**Bruce Ellis**, Graduate Assistant, B.S. Chemical Engineering (University of Natal, South Africa)

**David Solberg**, Graduate Assistant, B.S. Chemical Engineering (University of Natal, South Africa)

**Nicolas Gil Zapata**, Graduate Assistant, B.S. Chemical Engineering (Universidad Industrial de Santander)

**Lee Yong-Jae**, Graduate Assistant, B.S. Food Science (Chonnam National University, Korea)

**Pedro Martins**, Visiting Research Associate, Licenciante in Engineering, Chemical Engineering (University of Porto, Portugal)

**Petr Horecky**, Visiting Research Associate, B.S. Chemical Engineering (Institute of Chemical Technology, Czech Republic)

### Adjunct Faculty

**Mary Godshall**, SPRI, B.S. Biological Science (Louisiana State University, New Orleans), M.S. Biochemistry (University of New Orleans)

**Dr. Terry Walker**, Department of Biological Engineering, B.S. Engineering Science & Mechanics (University of Tennessee), M.S. Agricultural Engineering (University of Tennessee), Ph.D. Agricultural Engineering (University of Tennessee)

### Staff Changes

**Liz Thompson**, University Administrative Specialist, transferred to LSU AgCenter Human Resources Department October 2003.

**Lisa Lindsay**, Secretary, transferred to LSU Theatre Department October 2003.



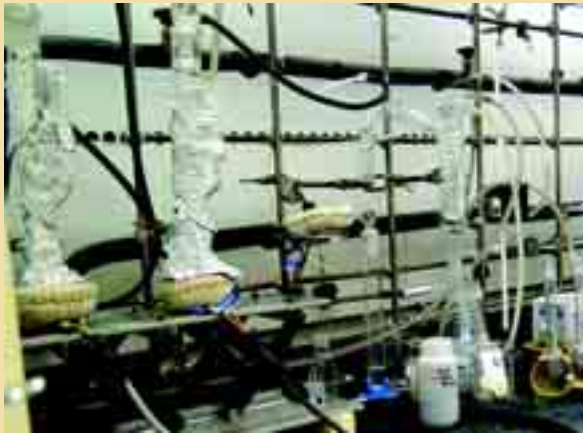
*William Jenkins, president of the LSU System (seated, left), and Robert Slaven, plant manager with Syngenta Crop Protection, Inc. (seated, right), signed the agreement giving a \$5.4 million facility at St. Gabriel to the LSU AgCenter for Audubon Sugar Institute. Also present at the signing were Dr. Bill Brown, former vice chancellor and director of the Louisiana Agricultural Experiment Station (standing, left), Dr. Peter Rein, head of Audubon Sugar Institute (center), and LSU AgCenter Chancellor Bill Richardson (right).*

## New Facility

Nestled in 4.4 acres of land on Highway 75 in Saint Gabriel, Louisiana, Audubon Sugar Institute's new facility was originally built to house Syngenta's former Ciba Geigy Research and Development department. After a merger to become Syngenta Crop Protection, Inc. this building was donated to the LSU AgCenter in 2003.

Audubon Sugar is now located only a mile south of the St. Gabriel Sugar Research Station and 13 miles south of the LSU campus, location of the old factory building. The new site will provide ASI with several advantages. Among these are modern laboratory space (26,885 square feet), ample room for the pilot scale sugar mill, space for an on-site library, conference rooms, offices for faculty members and staff, and area for expansion.

A new cooling tower was commissioned and the ventilation system improved to implement a more efficient way to climate-control the building. A solvent shed, east of the building, was converted to a welding shop. Installation of pilot scale equipment brought from the old factory is underway in the pilot plant area. A covered slab area was also built adjacent to





the facility where the mill equipment is placed out of the weather before its final installation. It is hoped that the sugar factory will resume operation once the mill equipment is reassembled.

ASI is also given the privilege to use Syngenta's "Pavillion" adjacent to the ASI laboratory for special occasions such as hosting larger groups for educational purposes. ASI's 2004 *Factory Operations Seminar* was held in this building. ASI's close proximity to the LSU AgCenter's sugarcane variety development and production research being conducted at the St. Gabriel Research Station should facilitate closer collaboration among the production and processing research faculty.





*The 3-roll mills, the last remaining equipment from the Audubon Sugar Factory on the Baton Rouge campus, being removed for transport to St. Gabriel.*



## **Audubon Sugar Institute**

LSU AgCenter  
3845 Highway 75  
Saint Gabriel, LA 70776  
USA