



# Audubon Sugar Institute

Annual Report

2001 – 2002



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**Louisiana State University Agricultural Center**  
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L. J. Guedry, Executive Vice Chancellor  
**Louisiana Agricultural Experiment Station**  
William H. Brown, Vice Chancellor and Director  
**Louisiana Cooperative Extension Service**  
Paul D. Coreil, Vice Chancellor and Director

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## Vice Chancellor's Foreword

*Sugar is important to Louisiana's economy. The farm-gate value of sugar was nearly \$378 million in 2001. The value added by processing was nearly \$242 million, for a total economic impact of \$620 million, more than any other Louisiana agricultural commodity except forestry and poultry. The vitality and profitability of the sugar industry are obviously important to our state.*

*Efficient, well-managed sugar factories are keys to the success of our sugar industry because sugarcane is perishable and must be processed into raw sugar close to production fields and quickly after it is harvested. The Audubon Sugar Institute is dedicated to ensuring that Louisiana's sugar factories have the latest in knowledge, technology and training to be able to process our annual crop quickly and efficiently.*

*The Audubon Sugar Institute, a part of the LSU Agricultural Center, is unique among land-grant universities. It is a historic part of a sugar industry that has seen many changes since sugar was first crystallized in Louisiana in the late 1700s. New and improved cane varieties have been developed over the years, each one with unique properties but each producing more sugar per acre than its predecessor. The improved varieties have caused changes in mechanization, crop protection and crop management, each of which brought new challenges to the factories as larger and larger crops were processed in fewer and fewer mills. Only by adopting new equipment and technologies have our sugar factories been able to cope successfully with these many changes.*



Dr. William Brown

*The Audubon Sugar Institute has a long and rich history of technical contributions to the modernization of Louisiana's sugar factories, but its future role is as important as ever. The economic pressures that dictate fewer and more efficient, higher-volume factories persist. The probability of higher sugar imports will contain prices and result in a highly competitive world sugar market. For Louisiana to compete successfully, our factories must continually improve their processing capabilities. Dr. Peter Rein, the ASI director, is a world-renown authority on sugar processing. His capable faculty are experts in their respective specialized areas. Along with the ASI support staff, they are dedicated to providing the research and technology to keep Louisiana factories competitive. The annual short courses and the academic specialization in sugar engineering now available will ensure that technically trained individuals are available to operate Louisiana factories.*

*The LSU AgCenter provides the base of operations for ASI, but we rely heavily on our important partners, the members of the American Sugar Cane League, to help support specific studies that are high industry priorities. The ASI and AgCenter leadership is proud of our long partnership with the League and grateful for its guidance and financial support in focusing ASI's activities on the most important challenges for Louisiana factories.*

*I am pleased to have an opportunity to comment in this ASI Annual Report that describes the Institute's current research and educational activities. With the continuing support of the sugar industry, ASI's reputation will grow domestically and internationally, and it will continue to contribute new and innovative technologies to keep the Louisiana sugar processors (and producers) efficient, profitable and sustainable.*

## 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.



*This sugar factory was built on the LSU campus in 1925 when the Audubon Sugar Institute was moved from New Orleans.*

## 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

(As of March 2002)

- Dr. William Brown – LSU AgCenter
- Ronald Guillote – St. Mary Sugar Coop Inc.
- Roddy Hulett – Honiron Corporation
- Dr. Benjamin Legendre – LSU AgCenter
- Duane Legendre – Lafourche Sugar Corporation
- Bruce Maillet – M.A. Patout & Son Ltd.
- Anthony Parris – Iberia Sugar Coop. Inc.
- Dr. Peter Rein – Audubon Sugar Institute
- Chip Savoie – Westfield Sugar Factory

## Report from Head of Audubon Sugar Institute

*In the past, the results of Audubon's research work have been reported each year in the Annual Report of the Sugar Station. However, Audubon deserves its own Annual Report to reflect properly the accomplishments and the capabilities of the Research Institute. This publication shows that Audubon Research Institute (ASI) is indeed a viable and active part of the LSU AgCenter, making an impression on Louisiana and other parts of the world sugar industry.*

*I have been at Audubon for two years, but the other faculty have served Audubon for many years and they have great depth in knowledge and experience. There have, however, been a large number of staff changes. Brian White has been active in building up an accomplished and reliable analytical capability, with help from Lee Madsen and Dorothy Wood. Joe Bell has been improving factory and pilot plant facilities and, with help from Scott Barrow, now enables us to address our instrumentation and electrical needs both in Audubon and in the mills we serve. We also have a capable and hard-working team in Liz Thompson and Lisa Lindsay (part-time), who have made great progress in getting our financial affairs in order and our administrative infrastructure, including the library and the filing system, into good shape.*

*We have received excellent support from the LSU AgCenter. The bureaucracy of a university system seems less daunting now, partially through the efforts of Liz Thompson and the AgCenter support. We are particularly grateful to Vice Chancellor Dr. Bill Brown for the positive way he contributes to helping Audubon Sugar Institute.*

*The American Sugar Cane League has been very supportive, both financially and morally. We have recognized the League to be one of our primary stakeholders, and we recognize the need to provide a return on its investment and add to value to the League and the Louisiana sugar mills. We appreciate the League's support.*

*One of the issues we have attempted to address is the identification of the priorities of the Louisiana industry. It is not always easy to get agreement on priorities from the entire Louisiana milling industry, but we have been helped enormously by Roddy Hulett and his League Research Committee. We intend to become more relevant to the Louisiana processors. Our mission statement and objectives given in this Annual Report were agreed on by ASI and the Louisiana processors, and they have helped us to keep our focus.*



Dr. Peter Rein

*One of the major developments in the past year has been the acceptance by the LSU College of Engineering of the Sugar Engineering minor, a new option open to engineers who wish to make a career in sugar. Apart from encouraging undergraduate students, part of the rationale for the introduction of two new courses in Sugar Engineering is the wish to build an active and viable school of researchers actively pursuing Sugar Engineering topics in graduate research programs. We have secured funding for graduate assistants and have some good research projects under way. We still need to get more corporate sponsorship to really take our graduate research to the levels we want.*

*I have no doubt that our research output is best improved and boosted by upgrading our graduate research programs. There are valuable spin-offs, and I am convinced that developing simultaneously the three elements of research, teaching and extension will push Audubon Sugar Institute to new heights.*

*Audubon has a great opportunity to prove itself both in Louisiana and internationally. It is the only cane sugar research institute integrated with a university and the only English-language university with these options available. I am sure that with continued support from the AgCenter and the American Sugar Cane League, and with the commitment of faculty and staff, Audubon Sugar Institute will go from strength to strength.*

## On-line NIR Measurements for Cane Analysis

FOSS Tecator offered us the use on trial of an instrument specially designed for cane analysis. This was installed at Cinclare Mill and tested during the last grinding season. One hundred fifty samples were run using the FOSS Infracana Near Infra Red System (FINIRS).

Cane was analyzed by the conventional core-press method and by direct analysis of cane (DAC) using a wet disintegrator. These data were used for developing calibration equations.

The system includes a shredder into which the core sample can be deposited, and it can turn over many more samples in the same unit of time, which could allow for multiple sampling single loads. The FINIRS can provide an analytical prediction in ~110 seconds, which improves analysis turnover. This will allow for replicates and or multi-bin analysis of the same load, thus improving the quality control parameters.

The table shows the statistical parameters developed in deriving the calibration equations, using the DAC data. Values of standard error (SEC) are excellent, indicating that NIR can give reliable measurements of cane quality. Values of SEC using the core press data were significantly inferior to these.

**Table 1. NIR calibration data derived from DAC results.**

Constituent	N	Mean	SEC	r <sup>2</sup>	SECV
Pol % Cane	177	12.90	0.212	0.970	0.311
Brix % Cane	179	15.49	0.235	0.965	0.380
Fiber % Cane	171	12.78	0.670	0.813	0.838
% Moisture	170	71.49	0.546	0.900	0.638
TRS	175	217.3	6.12	0.930	7.48

The NIR system allows for analysis of more complicated matrices than core press analysis, particularly with regard to mud-laden sugarcane samples. In many instances, the core press will fail to get juice from a sample that is mostly mud. DAC analysis can, because the matrix is modified with extraction water. NIR calibrated against DAC can yield a good result quickly, where no result was possible before. This also applies to samples that are mostly

**Figure 1. FOSS Infracana NIR system for direct analysis of cane installed at Cinclare Mill.**



“trash” (tops or other leafy material). On a preliminary basis, it appears that mud and trash may be quantitated by differential in spectral intensity (log 1/I), because mud and trash absorb the NIR light. This may prevent unnecessary payments made for mud or trash.

In conclusion, the FINIRS system provides a highly precise, accurate, fast and flexible solution to the analysis of sugarcane for purposes of cane payment and factory quality control. The FINIRS can provide results that are comparable to routine cane analyses, while discriminating between clean and dirty cane. An average of these results would provide a much more accurate assessment of cane quality than the single sample regime now in place. In terms of cost effectiveness, the unit needs only one operator (as opposed to four for the core press system), and it will benefit the factory and the farmers by providing more accurate data for cane payment and for factory control.

## Biocide Usage in Louisiana Sugar Factories

Biocides, or bactericides, are compounds designed to kill bacteria. Only two groups of antibacterial compounds are FDA-approved for biocide use in the U.S. sugar industry, namely carbamates and quaternary ammonium compounds. There are restrictions on the use of materials treated with quaternary ammonium compounds. For example, molasses containing “quat” residues cannot be used in animal feeds. This limits commercial biocides in the U.S. sugar industry to carbamate-containing formulations.

Use of a biocide for controlling dextran formation is common Louisiana industry practice. Because biocides are used to minimize biofilm buildup and reduce dextran formation in the processing system, it is important to use compounds or doses that will provide residual protection for juice in dead spots in the process. Residual activity was determined from the reduction in dextran formed after 24 hours between juice with and juice without biocide from the same mill. Samples of mixed juice were obtained from each factory before and after the point of biocide addition. These samples were held at room temperature for 24 hours, and then the dextran concentration was measured in each sample. The magnitude of the difference between dextran values with these samples was taken as an indicator of residual biocide activity. A comparison of residual activity of bleach or carbamates is shown in Table 2.

**Table 2. Biocide residual activity (Mill Tests\*).**

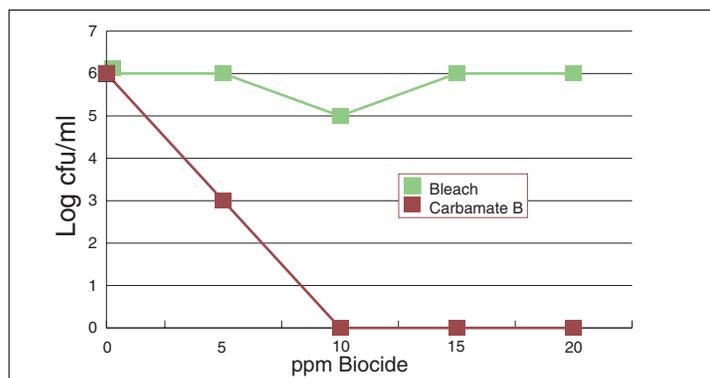
Biocide Type	# Tested (n)	Ave. Dextran difference in 24 hr (ppm/juice) Reduction from Control
Combination (Bleach, carbamate)	1	56
Bleach	6	0
Carbamates		
A	4	167
B	4	245
C	1	61

Because there was no consistency in biocide application or dose rates between the various factories, the residual reduction numbers should not be taken as absolute measures of effectiveness. The average increase in dextran in juice after 24 hours was 713 ppm without biocide addition.

Commercial biocides were found to be more effective than bleach in controlling microbial populations. This is illustrated (Figure 2) by a comparison of microbial kill in cane juice by equivalent concentrations of bleach or a carbamate biocide. Bleach dosed at

20 ppm (a level higher than commonly used in Louisiana) had no appreciable effect on microbial populations in cane juice, whereas a commercial biocide (carbamate B) was effective at the 10 ppm dose level.

**Figure 2. Effectiveness of biocides in cane juice.**



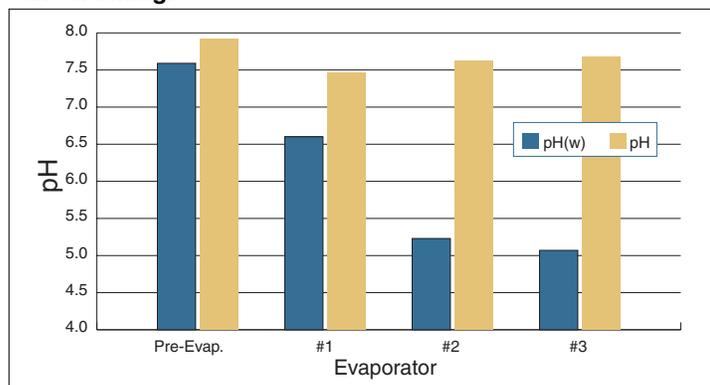
## Organic Acids and Sugar Factory Corrosion

The rapid shift in Louisiana from whole stalk harvesting to billet harvesting has resulted in unanticipated alterations of traditional processing systems in the sugar factories. Increased organic acid loading has been found in the mixed juice. This can cause processing problems that may range from increased scale formation, increased lime usage and higher sugar losses to more rapid equipment corrosion, and it may well be a factor in some of the unusual processing problems reported in some Louisiana factories this past season.

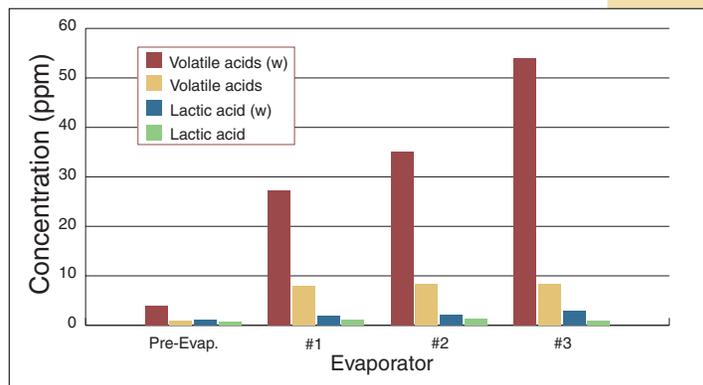
Research focused on a mill that had a history of rapid corrosion of condensate return lines. Measurements were taken of organic acid levels and pH values across the sugar processing system. Particular attention was paid to volatile (acetic, formic) and non-volatile (lactic) acids of microbial origin. Early in the study, it became apparent that a major source of the problem was related to the cane washing system. This mill has a recycle wash pond that cannot handle the BOD input. This meant that after a month of operation the cane was washed with water containing increasing levels of organic acids. Weather conditions in 2001 were such that it was possible to process the cane without washing. Cessation of washing brought an immediate improvement in operation and solved the corrosion problem in the condensate return lines that had been a problem.

Results are summarized in Figures 3 & 4. Average pH and volatile acid values in evaporator condensates are shown for periods when the mill was washing and when washing was stopped.

**Figure 3. pH of evaporator condensates, with and without cane washing.**



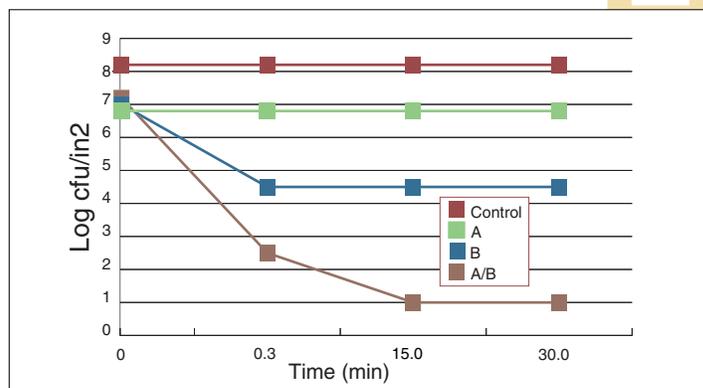
**Figure 4. Concentration of acids (ppm) in condensates.**



## A New Biocide

A bi-component contact biocide was developed that works rapidly against a wide variety of microorganisms in both the vegetative and spore form. It is effective in both a dip and a spray mode. It is non-corrosive and pH independent. It is also effective across the temperature range of 4° to 100° C. It appears to work well against organisms in biofilms. The biocide was also tested in an industrial situation against thick biofilms of *Leuconostoc mesenteroides* found in cane wash equipment at a Louisiana sugar mill. The application was by sprayer, dispensing a known dose of biocide. The results are given in Figure 5. The effect was very rapid, with kill complete in less than 2 minutes. There was a certain amount of residual activity, because biofilms did not reform for several hours under conditions favorable for development.

**Figure 5. Effect of biocides A and B and combination A + B on microorganism activity.**



Currently we are testing its effectiveness against organisms of interest that include *P. aeruginosa*, *B. cepacia*, *B. anthracis* and *Aspergillus sp.* We are also testing the soil penetration of the biocide with a view to using it for environmental sanitation. Both components are food approved, and the combination has little or no residual.

## Dextran Antibody using the Phage Display Technique

With the increase in use of antibodies in dextran analytical methods, a new technology for the production of these reagents is being developed. A phage display antibody with specificity against dextran was selected from a combinatorial antibody phage library. Antibody DNA encoding millions of various Fab fragments as protein ligands was batch cloned into a phage genome as a fusion to the gene encoding one of the phage coat proteins (pIII). Expression of the fusion product and its subsequent incorporation into the

mature phage coat results in the ligand being present on the phage surface. Phage that displays a relevant ligand for dextran was retained, while non-adherent phages were washed away. Bound phage was to be recovered, reinfected into bacteria and re-grown for further enrichment. For expression of soluble fragments, the selected phage DNA can be subcloned by PCR into an expression vector, and the vector can be transformed into E.coli TG1. Antibody fragments will be secreted into culture supernatant after overnight incubation. These antibody fragments, or phage, can be used as reagents to detect dextran antigen using immunoturbidimetric assay.

## Effect of Clarification Conditions on Composition of Evaporator Condensate and Non-sugar Elimination

A series of laboratory milling - clarification - evaporation tests was done during the 2001 season to investigate the effects of clarification conditions on the composition of evaporator condensate and non-sugar elimination in clarification. Hot liming and soda ash clarifications were tested, and clarified juice pH varied from 5.0 (no pH adjustment) to about 8.0. Anion (lactate, acetate, formate, chloride, malate, sulfate, oxalate, phosphate, citrate and transaconitate), cation (Na, K, NH<sub>4</sub>, Mg, Ca), Si and other parameters were determined in samples of raw juice, syrup and evaporator condensate. The equipment used is shown in Figure 6.

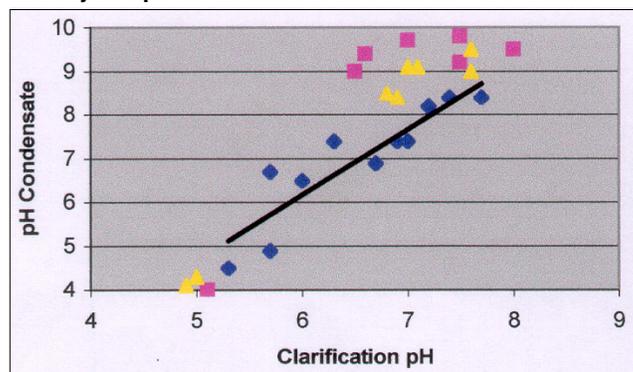
Figure 6. Laboratory equipment for clarification tests.



pH of the condensate depends mainly on the relative concentrations of ammonia and acetic acid. Both are affected, in opposite direction, by pH in clarification. Ammonia concentration is increased because of a faster rate of glutamine deamination at higher pH, and acetic acid concentration is decreased because of reduced volatility of the acid at increasing pH. The tests indicate that poor pH control in clarification, with occasional dips below 6.3 or even 6.0, is primarily responsible for the acidic condensate reported at times by the mills. It is believed that even stale cane if properly limed, and pH maintained at some 6.5 or higher, should not pro-

duce acidic condensate and potential for corrosion in the vapor and condensate space of the evaporators. A question remaining, perhaps, is whether the glutamine levels (the source of ammonia) are lower in the new than in the older varieties, which might lead to generally lower condensate pH than observed in the past. Glutamine concentration will be considered in the 2002 program. The pH dependence of volatility of acetic acid was confirmed in tests in which pH of dilute solutions of glacial acetic acid (2 g/L) in 15 Brix sucrose solutions was adjusted with milk-of-lime and evaporated under vacuum. The acetic acid concentration determined by HPIC in the condensate at pH levels above 6.5 is very low and would be offset by ammonia in condensate from cane juice.

Figure 7. pH of evaporator condensate as a function of clarified juice pH.



A significant increase in calcium during liming occurs. Increases of some 100% at pH 6.6 and over 250% at pH 7.5 were measured, while magnesium was unchanged. Trans-aconitate represents 30% to 60% of all anions and is nearly unaffected in hot liming. Of the other major anions, sulfate levels are unchanged by liming as well, while oxalate and phosphate levels are reduced by about 60% to 70%, although the oxalate removal is not enhanced by pH rise in clarification. Silicon behavior is of great interest because silicon and calcium are the major inorganic components of evaporator scale. The gravimetric analysis used does not differentiate between insoluble silica and soluble silicates, and the elimination rates that were found must include the elimination by settling of insoluble fine silica. Behavior of organic anions is complex; generation, decomposition and volatilization during clarification are likely.

Table 3. Results of clarification tests with lime and with soda ash.

	No pH Adjust	Hot Liming	Soda Ash
<b>Syrup (ppm DS)</b>			
Ca	1305	3670	966
sulfate	3012	4024	3878
oxalate	344	135	222
phosphate	1227	305	401
aconitate	7912	12252	9152
<b>Ion Elimination (%)</b>			
Ca	-4	126	-34
sulfate	-12	-9	-8
oxalate	-59	-65	-53
phosphate	-20	-85	-74
aconitate	-19	-8	-7
<b>Condensate (ppm)</b>			
pH	4.1	8.6	9.1
actate	1.3	2.1	1.8
acetate	39.3	2.5	1.3
formate	5.7	0.4	0.3

The soda ash treatment produces syrups with much lower calcium levels than hot liming, reduced with respect to raw juice by some 10% to 30% compared to the 100% to 250% increase in hot liming, although the turbidity elimination is inferior. There was some indication at the end of testing that dose and/or choice of the flocculant could bring the turbidity down to the levels seen in hot liming.

Only qualitative information could be obtained on the settling rates in the various treatments, but all were high, with settling complete within 5 to 10 minutes. It is significant that no difference was found between the pH of clarified juice and pH of syrup. The high pH drop seen routinely in the factories must come from microbial effects in filtration and chemical degradation of sugars in industrial evaporators.

### Falling Film Plate Evaporator Trials

A 10m<sup>2</sup> (131 ft<sup>2</sup>) plate pack with feed distributor was donated by Balcke-Durr (Germany). The unit was installed in a 42-inch diameter by 22 feet high mild steel column and installed at the Raceland Raw Sugar Factory.

The feed flow was designed for 12 gpm so as to be above the required wetting number [equivalent to 1.5 l/h cm] to assure proper operation and minimal fouling of the plate pack because no recirculation was used.

A man-way just above the top of the distributor and two sight glasses were installed to enable observation of the liquid-vapor separation just below the plate pack, as well as two non-condensable gas vents, a condensate measuring vessel with block valves, strainers in the feed line and an on-off level control float. Only twice did the strainers contain sizable quantities of bagacillo. Any accumulated dirt in the distributor was removed daily by opening the bypass valve for one minute, which emptied the distributor and allowed the feed to flow through the distributor to sweep away loose dirt.

The feed was "clarified juice," which was from alternating intermediate and cold limed mixed juice. The feed brix varied considerably because excess magma was returned to the clarified juice. Hourly samples were taken of the feed and the discharge to determine pH, sucrose, glucose, fructose and color.

During the first run, the unit was operated for 138 hours continuously. The feed flow rate varied continuously between 5 and 12 gpm, the feed temperature varied between 206° and 234° F, the exhaust steam temperature between 250° and 280° F and the feed brix between 14.0 and 19.4. All changes occurred continuously and independently of one another. Still, the falling film unit operated well.

The evaporative load fluctuated, because it was affected by the changing flow rate and the temperature difference between exhaust steam and feed, and varied between 7.1 and 18.5 lb/hr. ft<sup>2</sup>. The liquid residence time at 12 gpm was only 8.3 seconds. The overall heat transfer coefficient was initially around 700 Btu/hr. ft<sup>2</sup> ° F and was reduced after nearly six days to about 325.

During a factory evaporator cleaning stop, the unit was cleaned by boiling with an 8% caustic soda solution for one hour followed by a water wash. A check with a telescopic mirror and flashlight indicated a nearly clean plate pack.

When feed was available again, the unit was started and operated continuously for more than seven days until the end of the grinding season. The evaporation rate, as a result of the unsteady conditions, fluctuated between 3.8 and 24 lb/hr. ft<sup>2</sup>. The overall heat transfer coefficients reduced in just over a week from well over 600 (3.4 kW/m<sup>2</sup>C) to about 425 Btu/hr. ft<sup>2</sup>F (2.4 kW/m<sup>2</sup>C). The average feed brix was 17.1 and the discharge brix 22.3, hence it operated more like a second than a first effect.

Color measurements in ICUMSA Units (IU) of the feed varied but on average feed color was 11,950 and the average discharge 12,690 IU. For comparison, a few samples were taken from the factory first and second effects for color analyses. Color was 11,940 IU into the first effect, 13,770 IU into the second effect and 14,860 IU into the third effect. Samples from two other factories showed ~ 10,000 IU into the first effect, 11,600 IU into the second effect and 12,985 into the third effects.

In conclusion, the FFPE unit operated extremely well under difficult conditions. The evaporative load, at the same delta T, is higher than for Robert evaporators. The residence time is very short, and this minimizes color formation. Overall heat transfer coefficients in a clean unit will start at or near 700 Btu/hr. ft<sup>2</sup>F (4.0 kW/m<sup>2</sup>C) and reduce to around 350 - 400 Btu/hr. ft<sup>2</sup>F (2.0 to 2.3 kW/m<sup>2</sup>C) after more than a week of continuous operation. This is probably somewhat better under steadier conditions.

### Evaporator Scale Formation and Composition

Tubes with scale were obtained from all four effects of a factory in the Teche area, from the fourth effect of another factory in the Teche area, and from the fourth effects of factories in the Lafourche and Mississippi River areas.

Table 4 indicates the scale quantity and thickness in the tubes. In the fourth effects, an average of 16.4 grams scale per square foot of tube surface was measured. In all cases, the scale at the bottom of the tubes was thicker than at the middle and the top. Scale thickness of more than 2 mm in the fourth effects is considered to be too high.

Table 4. Louisiana scale data.

	Scale, (g/ft <sup>2</sup> )	Scale thickness at bottom (mm)	Scale thickness at top (mm)
FACTORY I			
4 <sup>th</sup> Effect	16.3	1.6	0.7
FACTORY II			
Pre-evap	15.8	0.6	0.3
Effect 1	3.9	0.8	0.6
Effect 2	5.9	1.1	0.9
Effect 3	6.8	1.6	1.1
Effect 4	14.8	2.0	1.4
FACTORY III			
Effect 4	16.1	2.2	1.1
FACTORY IV			
Effect 4	18.4	2.3	0.9

Table 5 gives the results of the scale analysis. Elemental analysis was done by ICP (Inductive Coupled Plasma). Organic matter levels in scale did not exceed 10.6% and moisture levels did not exceed 10.5%. Hence, inorganic salts make up more than 82% of the scale. ICP analysis indicated that calcium levels in fourth effect scale (the most difficult to remove) were as high as 20.1% and silicon levels as high as 17.8%. It seems that scale from the Teche area contains more silicon (or silicates) than from other areas.

X-Ray Powder Diffraction was used to identify crystalline phases because these (silicates and oxalates) are the most difficult to remove. Scanning electron microscopy and optical microscopy in polarized light were used to identify crystal morphologies and the nature of some layers of scale.

**Table 5. Louisiana scale composition (elemental analysis in % on ash).**

	Moist.%	Organic matter, %	Inorganic salts, %	Ca	Mg	Si	P	S	Fe	Al
<b>FACTORY I</b>										
Pre-evap 1 (CS)	7.1	8.3	84.6	6.1	2.6	1.9	6.5	0.8	1.4	0.1
Pre-evap 2 (SS)	6.0	10.1	83.9	16.2	3.7	3.4	6.3	0.6	1.1	0.1
Effect 1	6.8	4.3	88.9	21.2	2.2	2.8	8.9	0.6	0.6	0.1
Effect 2	8.4	3.9	87.7	9.8	1.9	5.9	10.7	0.5	0.5	0.05
Effect 3	7.4	5.1	87.5	12.4	3.0	13.1	4.0	0.4	0.8	0.03
<b>FACTORY II</b>										
Pre-evap	5.8	9.5	84.7	20.6	2.8	1.3	6.1	0.7	0.4	0.1
Effect 1	2.8	2.7	94.5	22.3	4.5	6.3	6.4	0.7	0.7	0.1
Effect 2	4.8	3.1	92.1	22.6	2.7	1.9	11.8	0.7	0.9	0.1
Effect 3	10.5	4.2	85.3	18.0	1.6	6.9	10.3	0.7	0.4	0.04
Effect 4	6.4	3.9	89.7	12.5	2.1	13.6	2.7	0.4	1.3	0.02
<b>FACTORY III</b>										
Effect 4	5.9	7.4	86.7	15.7	2.7	16.8	3.4	0.3	0.9	0.03
<b>FACTORY IV</b>										
Effect 4	7.4	10.6	82.0	20.1	2.4	17.8	3.6	0.2	1.1	0.05

Calcium in scale is, to a large extent, a function of the pH of the limed juice. For the natural pH of the juice (~ 5.4) to rise, the organic acids present must first be neutralized. With dirty wash water, additional acids are added to the expressed juice. Juices high in organic acids require more lime, and this yields more dissolved calcium salts. At a pH of 7.0, essentially 90% of the  $P_2O_5$  has been precipitated.

An easy way to assure the quantity of lime used is adequate is to add to clarified juice a few drops of clear calcium succrate solution. If the result is a precipitate or increased turbidity, insufficient lime was used. If the result is no precipitate or turbidity formed, just enough or too much lime was used. The  $P_2O_5$  levels in clarified juice should be > 10 mg/L. Otherwise overliming has taken place. This can be measured easily with the appropriate kit.

Juices high in phosphates (and silicic acid) may generally be limed to pH ~ 7.6, and juices low in phosphate should not be limed much beyond ~7.2. Calcium phosphates, sulfate, aconitate, oxalate and citrate are all relatively insoluble and will increase scale formation.

## Evaporator Chemical Cleaning

Cleaning tests were held at two factories (Teche and Lafourche areas) with EDTA in a 2% caustic solution. Based on laboratory tests, water was added to obtain a 5% EDTA solution in the evaporator bodies, and this was boiled at atmospheric pressure for 2 to 2.5 hours. Effects 1 through 3 were clean but, in one case, the fourth effect was not totally clean; in the other case, it was still somewhat dirty. It turned out that in both cases the silicon levels were high, more so in the Teche area than in the Lafourche area.

At lower silicon levels (roughly 10% or less), the EDTA removes sufficient calcium and magnesium for the scale structure to disintegrate and be removed. At higher silicon levels, that is not the case. EDTA at a pH value of at least 9 will remove 215 mg of calcium per gram of EDTA. Small amounts of ammonium bifluoride were subsequently added to the EDTA solution, so the final pH value did not go below 9 for the final cleaning of the falling film plate evaporator. After recirculating and boiling at atmospheric pressure for just over one hour, the plate pack and distributor were essentially clean. The cost of EDTA (39%) is around \$ 0.35/lb, and it is to be used at concentrations of ~ 5%. EDTA can be regenerated after use by lowering the pH with sulfuric acid to near 2, adding sufficient caustic soda to achieve a pH of 9 and possibly adding

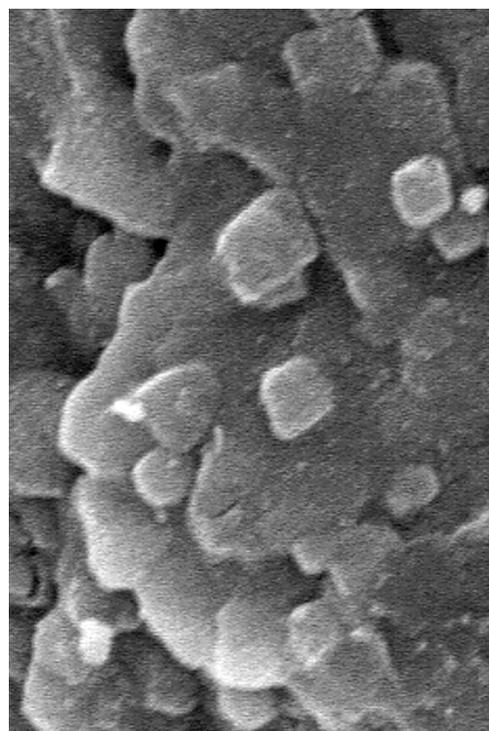
some fresh EDTA. It should be cost effective in comparison to caustic soda and HCl.

A new acid (HCl + rodine + wetting agent) from Southern Chemical was tested on juice heaters, and the same material cleaned three heaters well. In the evaporators it did not clean the fourth effect unless some ammonium bifluoride was added. Sodium acid sulfate has been tested in the laboratory. A 10% solution removed some 99% of fourth effect scale.

Corrosion tests on copper tubes have been carried out on the three cleaning agents with ammonium bifluoride added. Per hour of boiling time the rates are respectively:  $5.1 \times 10^{-7}$  inches for EDTA,  $5.8 \times 10^{-5}$  inches for the HCl and  $7.7 \times 10^{-5}$  inches for the  $NaHSO_4$ .

With some additional work, all three cleaning agents will work well in a short period. Compositions have to be fine-tuned to

**Figure 8. Scanning electron microscopy picture of calcium silicate crystals in scale at 2500X.**



reduce the cost and make them similar or lower than the present caustic and acid boils.

Ammonium bifluoride or sodium fluoride should be used to assure that the silicon is attacked properly. In solution it forms HF, which attacks silicon to form  $SiF_4$  and/or  $H_2SiF_6$ . Once sufficient silicon has been removed, the other cleaning agents such as EDTA and the acids can handle the rest.

## Vacuum Pan Automation

A survey of pan automation was done last year in Louisiana and Florida sugar mills. Some mills in Florida are running very successful automatic pan systems, but no Louisiana mills run pans under fully automatic control. A large number of pans have instrumentation fitted, but in many cases it is inappropriate or obsolete.

The two Audubon Sugar Institute (ASI) lab pans have been automated using different systems. One of the pans has a number of different measuring transducers fitted, to establish which is the most appropriate measurement for different grades of boiling. The microwave density unit seems to be the best measurement device for high grade boilings, and conductivity is the most cost-effective for low grade boilings.

ASI has been involved in the specification of a fully automatic pan control system being installed at St. James. Using the experience of automating the ASI systems, we will assist in optimizing the St. James control system, and ASI should be in a position to assist all mills in specifying the most appropriate control systems, and to assist in design and operation of the automatic systems. It is expected that the benefits of better quality, better pan exhaustion, reduced manpower and maximum use of installed pan capacity can be achieved.



Pilot plant batch centrifugal.

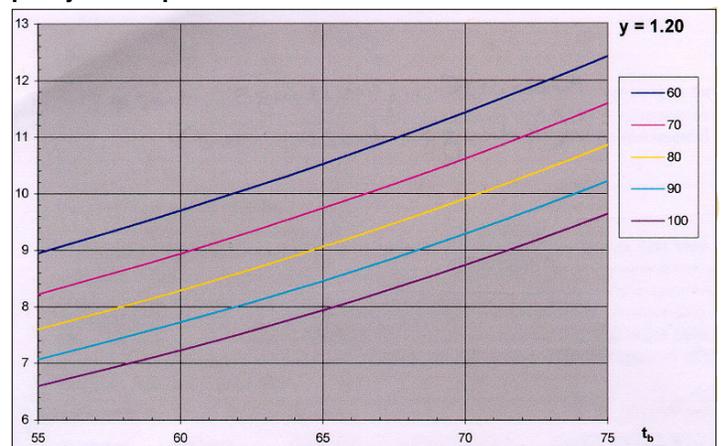
Table 6. Values of parameters to be used in equation for bpe.

Parameter	Estimate	(Hoekstra)	Approx	App.95% Std Error	Limit conf.
A	0.1660	(0.1379)	0.0556	0.0568	0.2752
B	1.1394	(0.808)	0.0172	1.1056	1.1732
C	1.9735	(2.327)	0.2750	1.4331	2.5138
D	0.1237	(-0.42)	0.0137	0.0967	0.1507

what surprising, but it must be recognized that most of the literature data are either based on measurements with sugar beet liquors or sparse historical sugar cane data. This work is still in progress, and a more thorough presentation and analysis of the new data will be forthcoming.

An example of the data presented in the form suitable for vacuum pan control (as the boiling point elevation at different massecuite temperatures and Nutsch (true) purities) is shown in Figure 10. A comparison with the frequently used nomogram from the Cane Sugar Handbook reveals differences of up to 2°C from the present data.

Figure 10. BPE (°C) as a function of temperature (°C) and purity at a supersaturation of 1.2.



## Boiling Point Elevation of Technical Sugarcane Sucrose Solutions

In the framework of a larger program on “optimization of sucrose crystallization,” a series of boiling point measurements was done in the 50 gal. ASI Pan No. 2 that is now fully instrumented and well suited for accurate measurements of various parameters involved in evaporation and crystallization of sucrose solutions.

Figure 9. Laboratory vacuum pan.



Lenn Goudeau, research associate at the Audubon Sugar Institute, is in charge of sugar boiling at the factory on the LSU campus, which includes pilot plant equipment to simulate all of the operations of a sugar mill.

An equation that was previously used at Tongaat-Hulett Sugar to fit literature data for the boiling point elevation (bpe) of sugar liquors concentration with purity  $q$ , concentration (Brix)  $W_{DS}$  at the water boiling temperature  $t_{bW}$  (°C), was used to fit the experimental data.

$$\Delta t_b = A * \left( \frac{W_{DS}}{100 - W_{DS}} \right)^B * \left( \frac{273 + t_{bW}}{100} \right)^C * \left( \frac{q}{100} \right)^D$$

The equation with the new coefficients listed in Table 6 fits the bpe data very well. The effect of purity is much less than, and even opposite to, that reported in the literature; that is, bpe was found to be slightly higher, the higher the purity of the solution. This is some-

## Storability of Mill Syrup in Louisiana Conditions

At the request of two sugar mills, a laboratory study was initiated to explore the feasibility of storing the mill syrup for processing in January and February, after the sugarcane season has ended. This option is routinely practiced in the colder climates of the beet sugar industry, and we hoped it could open the door for the much-needed extension of the processing season in Louisiana. As the grinding capacity of the mills continues to increase, diverting a part of the mill syrup to storage during the crushing season would potentially provide a lower-cost alternative to expanding the boiling house capacity.

Although the study is incomplete, the results so far — with storage of four syrups of different origins and with pH and Brix adjusted to various levels — indicate that the sucrose losses, if any, are small, as shown in Table 7. The slight drop seen in some samples of the sucrose content (as per cent Brix) indicated by HPLC (“true” purity) is not matched by the expected increase in invert levels. For syrup with initial pH higher than 6.6 and 66 Brix or more, the pol (apparent) purity appears unchanged over the four-month storage within the bounds of the analytical precision. The only syrup where the sucrose loss appears noticeable and is accompanied by both invert rise and pol purity drop is “syrup C,” which initially had a low pH; even the pH adjustment before storage did not prevent some sucrose inversion during 3 1/2 months’ storage. Based on these preliminary results, it appears prudent that syrup intended for storage have a pH and Brix above 6.6 and 66, respectively. Other precautions, such as cooling the syrup before storage and others related to the design and operation of the storage tanks that have

been exercised with success for years by the beet industry, may need to be observed as well.

Within a few weeks of storage in sealed glass jars, a small amount of fine precipitate settled in each syrup, with the amount apparently quite independent of the pH. The X-ray diffraction analysis revealed the precipitate to be composed of silica (SiO<sub>2</sub>) and calcium oxalate. Because the amount is small and appears to be merely the fine suspended solids that passed through clarification and settled over the long storage period (the supernatant syrup is distinctly less turbid than the fresh syrup), the precipitate is not expected to pose problems in post-season processing of the stored syrup.

## Evaluation of Horizontal and Vertical Crystallizers

As part of a program to test and evaluate new types of equipment installed in the Louisiana sugar industry such as vertical crystallizers, continuous vacuum pans, short retention time clarifiers, etc., work was initiated on vertical crystallizers and on pans to a limited extent.

Three types of vertical crystallizers are in operation in Louisiana, provided by Fletcher-Smith, Silver and Honiron. The horizontal types of crystallizer in use are predominantly the Blanchard type, although two Werkspoor rapid cooling crystallizers are in service.

A Nutsch device fitted with a conventional low grade C centrifugal screen and operated on 100 psig plant air was used to obtain mother liquor samples from the massecuite at various stages in the process. In particular, the mother liquor in the massecuite leaving the pan, after cooling in the crystallizers, and after reheating prior to centrifuging, was analyzed for refractometric Brix and apparent purity. Samples of the final molasses leaving the factory centrifugals were also analyzed for refractometric Brix and apparent purity.

The pan purity drops (massecuite purity minus mother liquor purity at striking) for continuous pans varied from 15 to 22 purity points and averaged 19 purity points. The purity drop for the batch pans varied from 12 to 23 purity points and averaged 16.5 purity points. The wide variation in purity drops for both types of pan appears to be caused more by operational variations rather than by the type of pan. Low massecuite Brixes were generally associated with low purity drops.

The temperature drop across the crystallizer varied from 29 to 51° F for factories with only Blanchard type of crystallizers. The temperature drop for one factory employing only vertical crystallizers was 45° F. One factory using a vertical crystallizer followed by a Werkspoor and using Blanchards for the final cooling had an average temperature drop of 52° F. Another factory using a vertical crystallizer followed by a line of Blanchards operating without cooling had an average temperature drop of 35° F. The temperature drop achieved in the crystallizers depends on the ambient temperature, use of cooling water and retention time available rather than on any inherent differences in the crystallizer type.

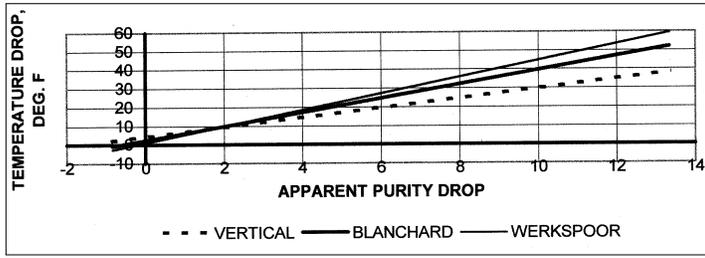
The mother liquor purity drop in the various types of crystallizers also was measured. Figure 11 summarizes the temperature drop/purity drop data for all three crystallizer types in a single plot. Note that the mother liquor purity drop per unit of temperature drop was highest for the vertical type followed by the Blanchard type, with the Werkspoor crystallizers achieving the lowest purity drop per unit of temperature drop.

The purity drop across the crystallizer station varied from 6 to 13 purity points. The purity drops achieved depended more on the degree of cooling achieved across the crystallizer station than on the type of crystallizer employed. The low grade station purity pro-

Table 7. Syrup storage trials.

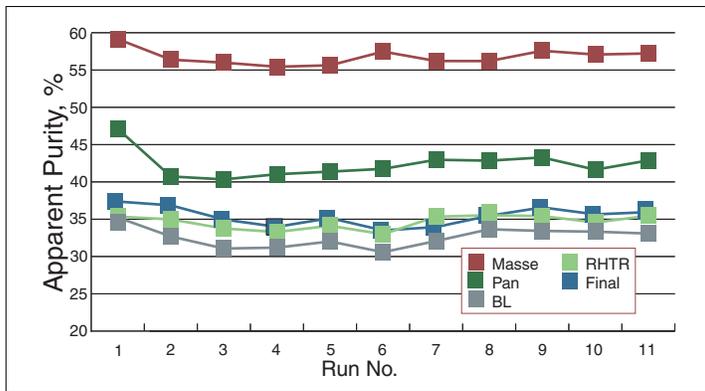
	pH	Sucrose	Invert	Pol
<b>Syrup A (66.0 Bx)</b>				
<b>Fresh</b>	6.7	89.1/87.9	6.2	83.3/83.5
<b>1-month storage</b>	6.7	87.1	6.4	83.7
(from 10/15 to 1/15)	7.0	87.9	6.5	83.6
	7.3	87.2	6.4	82.9
	7.7	88.1	6.1	83.2
<b>4-month storage</b>	6.7	87.9	6.4	84.4
(from 10/15 to 1/15)	7.0	88.0	6.5	84.4
	7.3	87.7	6.8	83.9
	7.7	87.5	6.9	83.4
<b>Syrup B (66.0 Bx)</b>				
<b>Fresh</b>	6.7	89.1/87.9	6.2	83.3/83.5
<b>3-month storage</b>	6.7	88.4	6.1	82.0
(from 10/15 to 2/19)	7.0	87.8	6.1	82.6
	7.3	87.7	6.3	83.6
	7.7	88.4	6.3	83.1
<b>4-month storage</b>	6.7	87.6	6.4	82.6
(from 10/15 to 2/19)	7.0	87.7	6.5	83.0
	7.3	87.5	6.8	83.7
	7.7	87.5	6.3	83.9
<b>Syrup C (65.6 Bx)</b>				
<b>Fresh</b>	6.0	91.3/90.7	5.3	86.1/86.1
<b>2 ½ month storage</b>	6.0	90.6	5.5	85.7
(from 11/5 to 1/15)	7.1	89.8	6.0	85.7
	7.7	88.8	6.2	84.8
<b>3 ½ month storage</b>	6.0	89.2	6.6	84.7
(from 11/5 to 1/15)	7.1	88.4	6.9	83.6
	7.7	88.3	6.5	84.2
<b>Syrup D (70.1 Bx)</b>				
<b>Fresh</b>	6.9	91.3/90.8	5.3	85.8/85.5
<b>2 ½ month storage</b>	6.9	90.4	4.9	86.2
(from 11/5 to 2/19)	7.8	88.5	5.1	85.6
<b>3 ½ month storage</b>	6.9	90.9	5.2	85.8
(from 11/5 to 2/19)	7.8	90.3	4.6	86.2

**Figure 11. Crystallizer temperature drop versus apparent purity drop.**



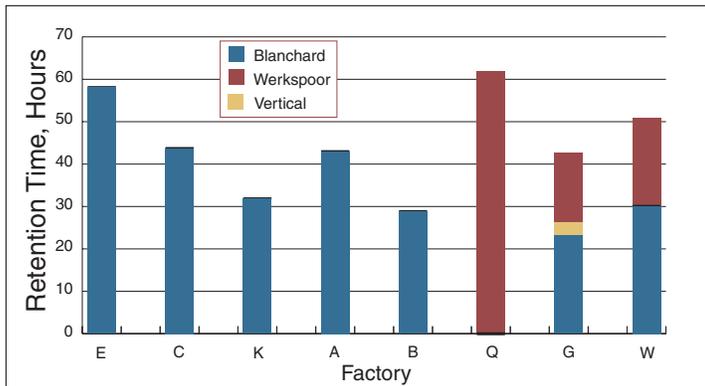
file for 11 days at one factory is shown in Figure 12. The plot includes the C massecuite purity, the mother liquor purity at striking, the mother liquor purity leaving the last crystallizer, the mother liquor purity after reheating and the final molasses purity leaving the factory centrifugals.

**Figure 12. C massecuite purity profile.**



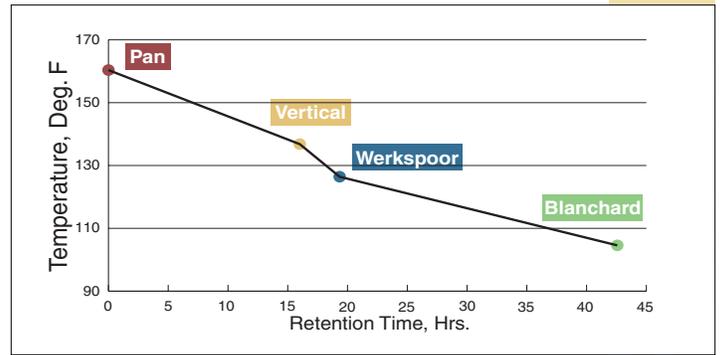
The average massecuite retention time in the crystallizers for the eight factories monitored is shown in Figure 13. Massecuite retention times in the crystallizers varied from 29 to 61 hours. Most factories had a retention time of more than 40 hours.

**Figure 13. C massecuite retention time in crystallizers.**



The temperature versus retention time profile for a factory with a vertical, a Werkspoor and Blanchard crystallizers operating in series is shown in Figure 14. Of note is the high temperature of the massecuite leaving the pan (160° F) and the rapid cooling achieved by the Werkspoor crystallizer. The lower cooling rate of Blanchard crystallizers in comparison with the vertical is caused mainly by the lower massecuite temperature in the Blanchard crystallizers.

**Figure 14. C massecuite crystallizer temperature profile.**



ASI Milling Tandem.

## Routine Final Molasses Analyses for Louisiana Mills

For the past two seasons, ASI has analyzed weekly molasses samples from Louisiana mills, using HPLC methods for sucrose and monosaccharides. Purities have been compared with the South African target purity equation predictions. The target purity differences generated provide the mills with the best and most reliable measure of the efficiency of their molasses exhaustion.

The total solids in the molasses were measured by vacuum oven drying, and a new relationship between refractometric brix and total solids was developed. In addition, molasses samples were analyzed by NIR, and calibration equations developed. These calibrations are most satisfactory, with excellent standard errors as shown in Table 8. NIR will be used for future analysis of molasses in the coming season.

**Table 8. Correlation information from NIR calibrations on molasses analysis.**

Constituents	2000 Season			2001 Season			Combined		
	SEC	n	R <sup>2</sup>	SEC	n	R <sup>2</sup>	SEC	n	R <sup>2</sup>
RDS	.12	189	.99	.10	226	.99	.12	416	.99
Pol	.25	190	.93	.31	223	.95	.28	409	.95
Sucrose	.27	190	.89	.22	224	.97	.24	414	.95
Glucose	.13	189	.93	.15	225	.97	.11	412	.98
Fructose	.10	189	.92	.12	229	.97	.12	415	.97
Ash	.15	192	.95	.11	226	.97	.13	421	.97
DS	.16	187	.97	.27	230	.93	.24	413	.95

A limited number of composite mixed juice samples were analyzed by HPLC. Surprisingly, the pol/sucrose ratios of about 0.95 were registered, significantly lower than values of about 0.99 recorded in South Africa. The mixed juice analysis program will be extended considerably next season.

## Comparison of Analytical Methods for Determination of Dextran in Juice and Raw Sugar

Measuring dextran in sugar-containing streams has always been a problem of detecting a small amount of a specific carbohydrate against the background of a large excess of other carbohydrates. Most methods have focused on reducing the background by selectively removing dextran by alcohol precipitation. To date, instrumental methods either have not been selective enough or rapid enough for dextran determinations to be of value as a process control aid to the sugar manufacturer.

Two recent procedures, the Midland SucroTest™ and the Optical Activity Ltd. DASA methods, were reported to hold promise. Comparison with two established procedures, Roberts' and Haze, was undertaken in collaboration between ASI and SPRI. Twenty raw sugar samples were chosen, and three samples of fresh raw juice prepared by milling billeted cane at ASI. Each of the three juice samples was analyzed fresh within 1/2 hour after milling, and again after up to 24 hours of standing at ambient temperature, to monitor the rate of dextran generation as well as to widen the dextran range. The DASA and SucroTest™ procedures were done simultaneously at ASI as the juice sample was withdrawn; the Roberts' analysis was done at SPRI after the juice was kept frozen for

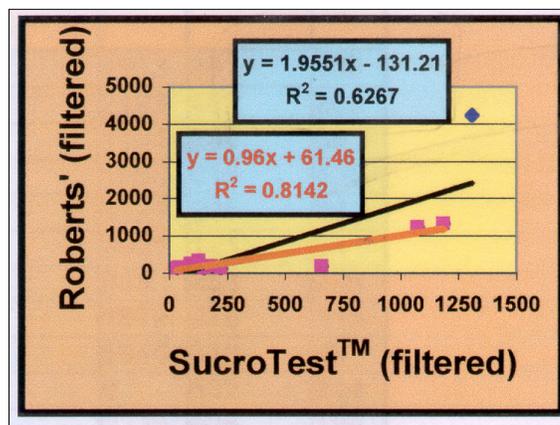
a week or so. Leftover solutions (juices and sugar liquors) after sample preparation for DASA analysis (filtration and dextranase treatment) were quickly frozen and analyzed again later.

Table 9. Comparison of different methods of starch analysis.

Raw Sugar	SucroTest™	DASA	Roberts'	Haze
1	243	1913	361	139
2	289	2192	462	209
3	101	1134	280	69
4	562	1968	618	439
5	93	1724	225	64
6	101	2021	125	54
7	322	668	453	239
8	154	1879	278	114
9	36	1945	318	49
10	263	0	532	214
11	217	2339	526	194
12	331	2255	249	199
13	86	1413	204	64
14	239	408	380	119
15	254	1266	390	114
16	104	1237	308	134
17	431	1424	585	304
18	90	1111	252	59
19	65	1221	234	49
20	431	1363	503	264

With the exception of the DASA method that was found to give erratic results for raw sugars, all others display some degree of correlation, the best being between the Haze and SucroTest™ procedures with a  $r^2$  of 0.872. To study in more detail the characteristics of the DASA procedure, the raw sugar liquors after filtration and after the DASA dextranase treatment also were analyzed together with the Roberts' and SucroTest™ procedures. Both methods indicate some residual dextran after dextranase hydrolysis, and Roberts' indicated some dextran removal in filtration. This is not likely since no DE was used, only a coarse filter paper, and it is possibly caused by changes in dextran conformation, solubility characteristics or other physico-chemical properties of the dextran molecules. Reduction of turbidity as a consequence of freezing and melting sugar juices and liquors has been noted before and may be related to the present observation regarding dextran analysis.

Figure 15. Comparison of Midland Sucro test and Roberts dextran methods on filtered samples.



Distillation columns used in research at the sugar factory.

## Modeling of Ion Exchange Adsorption with Gel Permeation Chromatography

This research focuses on the White Sugar Mill (WSM) process developed by Tongaat-Hulett Sugar, AECI Bioproducts (now SA Bioproducts) and Calgon Carbon Corporation. The process uses ultrafiltration and three ion exchange steps in series to produce white sugar directly from sugarcane. Little is known about what material is removed at each step, making optimizing the process difficult.

Gel Permeation Chromatography (GPC) is a separation technique based on molecular size. A 100 $\mu$ L sample is injected into an eluent stream. This flows into a column containing a gel of precisely controlled pore size. Small molecules are able to diffuse into the gel and take longer to elute from the column than larger molecules that cannot enter the pores. The concentration of material exiting the column is measured with a refractive index (RI) detector and the color of the material exiting with an absorbance detector set to 420nm. A sample chromatogram is shown in Figure 16. By calculating areas beneath the chromatogram, the molecular weight makeup of the sample can be quantified.

Figure 16. Sample GPC chromatogram – ultra filtered syrup.

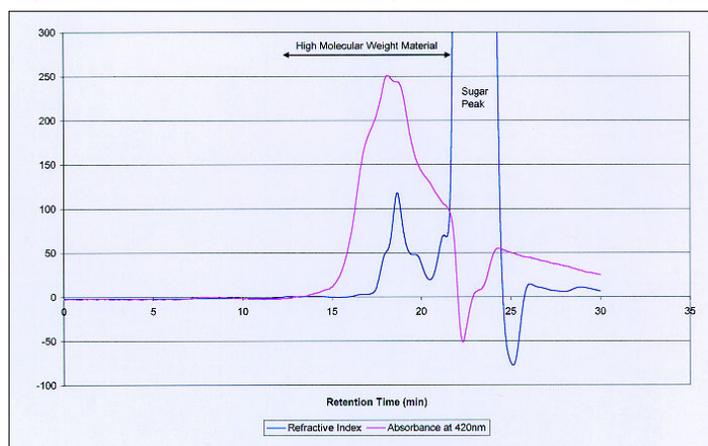


Figure 17. Color loading on a column during liquor decolorization.



Column testing is performed on each of the three resins: strong acid cation, weak base anion and strong base anion (decolorizing resin). By analyzing the samples with GPC, an accurate picture of the process can be obtained. These data may then be applied to adsorption models to develop a tool to optimize the process. Figure 17 shows the decolorizing resin during column loading.

A regression tool has been developed to correctly interpret the GPC chromatograms. A MATLAB computer program was created that fits the chromatograms with a number of Normal distributions. The areas under these distributions are then used as components in the adsorption model.



Dr. Donal F. Day, ASI faculty, with 30-liter fermenter with pH, temperature, DO, rpm and other controls.

## Glucooligosaccharides, A New Product from Sucrose

Current technology for the production of oligosaccharides is limited to extraction from plant sources, acid or enzymatic hydrolysis of polysaccharides or synthesis from starch by transglycosylation reactions. These procedures are costly, limiting use of oligosaccharides to high-value products. Conventional fermentations are the most practical means for industrial manufacture of carbohydrate polymers. Use of a chain shortening acceptor and a *Leuconostoc* strain that primarily produce highly branched polymers in a dextran fermentation resulted in production of selected glucooligosaccharides. These oligosaccharides are branched polymers between DP 2 and 8 in size.

The degree of polymerization (DP) for most prebiotic oligosaccharides falls in the range of 2 to 8. Those oligosaccharides larger than DP 3 produced in this fermentation were branched. Oligosaccharides synthesized by dextransucrase from this bacterium had  $\alpha$ -1,6 backbone with  $\alpha$ -1,3, and/or  $\alpha$ -1,4-branched side chains when maltose was used as acceptor.

Growth of *S. typhimurium* or *E. coli* on these oligosaccharides was less than 40% of the growth on an equivalent amount of glucose and similar to growth on commercial fructooligosaccharides. *Lactobacillus johnsonii* and *B. longum* showed no difference in growth rate on glucose or the oligosaccharide preparations. When *L. johnsonii* and *S. typhimurium* were grown together on the oligosaccharide preparations, the oligomers stimulated the growth of the *Lactobacillus* but were not readily used by the pathogenic organism. It appears that these oligosaccharides are used preferentially by probiotic strains.

Use of these oligosaccharides as prebiotics should lead to the production of intestinal lactic acid, increases in short-chain fatty acid production and lower pH's in large intestines. With appropriate application, they may be as a useful food additive to help prevent establishment of pathogenic organisms. Similar effects have been seen in studies on the effect of fructooligosaccharides in feed trials with broilers. Oligosaccharides reduced the susceptibility to *Salmonella* colonization of the intestine of chickens, increased *Bifidobacterium* levels and reduction in the level of *Salmonella* present in the caecum. These studies do not allow direct prediction of "in vivo" effects but indicate that this type of oligomers can be a prebiotic for intestinal microflora.

## Extension Work

### Washed and Unwashed Cane

The ash % cane for both washed and unwashed cane was performed on numerous samples for a factory that was processing mainly unwashed cane during the 2001 crop. The mill's pol extraction was determined for both the washed and unwashed cane. The quantity of field soil in the cane had little effect on the mill's pol extraction. The preparation index achieved when processing unwashed cane was not appreciably different from that for washed cane.

### Core Lab Procedures

Several factories requested help in standardizing core lab procedures. A few observed that dull cutters on the core tube resulted in juice extraction during the coring operation.

### Sugar Losses in Factory Effluents

Three factories requested assistance in determining the magnitude of sugar losses in the effluent water streams, both from cane washing and from the condenser and floor drains.

### Preparation Index

A few factories requested preparation index determinations on cane preparation equipment. Direct calculation of the mill's pol extraction also was performed at these mills.

### Boiler Efficiency and Compliance Tests

Assistance was provided to one factory undergoing compliance tests for boiler emissions. Boiler efficiency testing was performed for other factories trying to improve their boiler efficiency to reduce gas usage.

### Raceland Plate Evaporator

Assistance was given to Raceland in the integration of the Alfa Laval rising filter plate evaporator into the evaporator system and in the evaluation of the results.

### Flash Tank Design

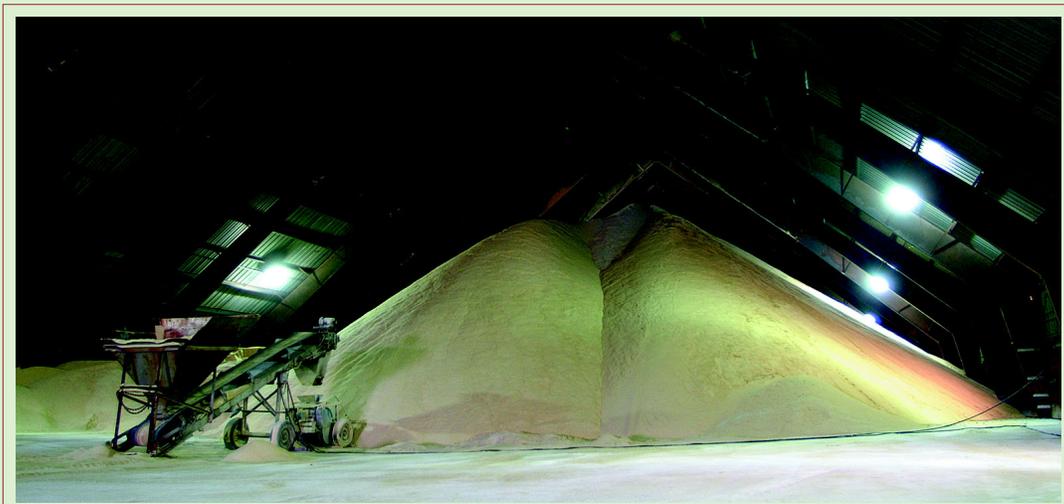
New juice flash tanks were designed for two Louisiana mills.

### Cane Quality Payment System

Audubon has been involved in various industry and other group discussions on options for a new cane payment system that will encourage greater productivity in the Louisiana sugar industry.

### Automatic Pan Controls

Options for automatic control of a C seed pan at St. James were discussed. Audubon participated in the specification of a new system to be commissioned in the next season.



*Raw sugar is stored in warehouses before being transported to refineries.*



*ASI analytical laboratory.*

## **Audubon Sugar Institute Analytical Capabilities**

In the past year, Audubon Sugar Institute (ASI) has invested considerable money and time in increasing and improving its analytical capability. New and pre-owned equipment has been purchased or acquired as gifts. Grants have been applied for and some received and used. Personnel have attended seminars on analytical equipment and software use.

ASI has also invested in the upgrade and automation of existing analytical instruments. Through these investments, ASI has improved both the accuracy and the capacity of its analytical laboratories. There remains room for further improvement.

ASI has six operational HPLC systems. Three units are ion chromatography units, one set up for cation analyses and the other two for anion analyses including organic acids. One unit is a GPC unit for dextran analyses with both a UV-Visible and a Refractive Index detector. The GPC unit has been used mostly for a color project, but it has been used occasionally for other applications. ASI also has an ion exchange chromatography unit used for alcohol and sugar analyses. The other HPLC unit is used for routine sugar analyses on juice, syrup and molasses and other process samples.

Other instrumentation includes a refractometer with 0.01 Brix resolution and temperature compensation capabilities, a polarimeter with 0.01°Z resolution for sucrose by Pol and a conductivity meter with temperature compensation for conductivity ash. A new gas chromatograph (GC) has been ordered for use in alcohol, sugar and organic acid analyses.

ASI has also had a demonstration Near-Infrared (NIR) Transmittance Spectrophotometer in house for more than a year. This unit has scanned all of the Final Molasses Survey samples from the past two seasons, and a calibration for Final Molasses has been generated. This NIR unit is being purchased and will be used for routine analyses of final molasses for the next season. The NIR has given accurate results very rapidly. ASI will continue to analyze a number of select samples by conventional methods to validate and improve the calibration of the NIR.

ICUMSA methods have been adopted as the standard procedures for ASI. Methods and calculations unique to the sugar industry in Louisiana have been replaced by ICUMSA methods where possible and/or tested to verify that they are accurate with samples from Louisiana. Work will continue in an effort to have the best and most universal methods and the best calculations for the sugar industry in Louisiana.

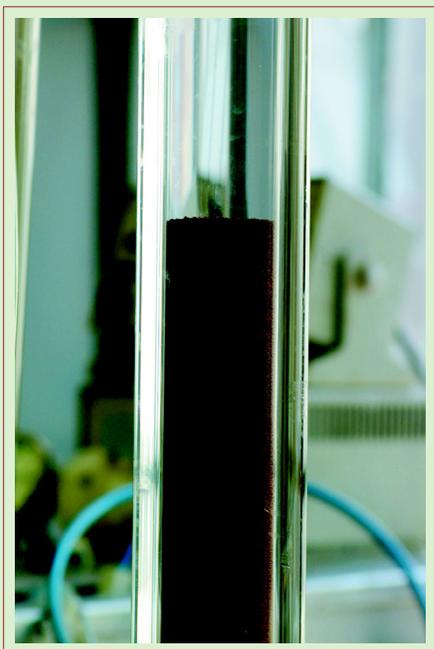
Overall significant improvements have been made to increase the capability and accuracy of the analyses that ASI conducts. ASI is embracing new technologies to improve the efficiency in all areas, including its analytical capabilities.

## LSU AgCenter, Audubon Sugar Institute Short Courses

In 2001, Audubon Sugar Institute (ASI) presented eight short courses aimed at increasing knowledge in the sugar industry. The range of courses offered was modified and expanded, with three new courses offered in 2001. Classes were attended by Louisiana sugar mill personnel as well as private industry and international delegates. The classes qualified for Continuing Professional Development for registered professional engineers.

### Technology of the Raw Sugar Factory – Three days

This course gave a comprehensive outline of the important aspects of sugar technology aimed at managing a raw sugar mill. It covered aspects of the cane crop, harvesting, milling and diffusion, liming, clarification, mud filtration, evaporation, sugar boiling, crystallization, centrifugation, molasses exhaustion, raw sugar quality, steam and power generation and waste treatment. Demonstrations using ASI equipment were provided.



*Research is under way on removing inorganic impurities and direct production of white sugar in one step, which will be a tremendous economic boost for the Louisiana sugar industry.*

### Polysaccharides and Dextran – One day

This was an introduction to the subject of polysaccharides, a range of carbohydrates such as dextran, starch and gums that affect sugar processing. This course focused on giving a manager or operator the background to understand and cope with the effects of polysaccharides.

### Bagasse Boiler Operation – Two days

This class provided an understanding of the role that the boiler plant plays in the raw sugar factory and the factors that affect boiler capacity, efficiency and air emissions. Bagasse boilers and natural gas boilers were discussed.

### Introductory Sugar Boiling – Three days

This class was aimed at individuals who had no (or limited) sugar boiling experience and who wanted to gain an understanding of the principles. Focus was on providing a good, practical grasp of sugar boiling.

### Vacuum Pan Instrumentation and Control – One day

This class focused on the practical principles of automatic control of vacuum pans and how to maximize the performance and capacity of the pan station. Students were able to get firsthand exposure on ASI's pilot plant pans.

### Continuous Vacuum Pan Boiling – One day

This class focused on providing an understanding of continuous pan boiling and how it compared with batch processing. It was aimed at workers involved in factory operations and those involved in designing and planning factory modifications and expansions.

### Bench Chemist – Four days

This class focused on individuals with little or no experience working in a lab. It provided an overview of the process involved in manufacturing raw sugar as well as how to perform the routine daily analyses needed by the sugar factory. Actual analyses were performed, and care and calibration of laboratory instruments were discussed.

### Chief Chemist – Four days

This class focused on helping the chemist, process engineer or manager understand how factory manufacturing reports are calculated, interpreted and checked against theoretical considerations. Hands-on calculation of a manufacturing report was provided as well as using computers to prepare, correct and check manufacturing reports. How to use the data to analyze factory operations was covered.

## LSU Sugar Engineering Courses

The Audubon Sugar School has a long history in education and training. The first sugar engineering course was established when the sugar school started in New Orleans in 1891. In 1925 it was moved to the Baton Rouge campus of Louisiana State University and the Audubon sugar factory was built. The school was an integral part of the chemical engineering department, but in 1977 it became a department on its own and the name changed to the Audubon Sugar Institute.

A degree in sugar engineering continued to be offered, but in the 1980s it became clear that the students preferred a degree in chemical engineering as a more broadly applicable qualification. Formal courses covering sugar processing have recently started again, with 11 students registered for BE4342 Sugar Process Engineering.

Some sugar companies around the world have formalized training programs for young engineers entering the sugar industry. The ideal graduate engineer for the sugar industry has a strong grounding in the basic science and engineering disciplines, with a specialization in sugar processing as part of an engineering degree. This can provide everything that the sugar company wants and overcomes the aversion of students to an industry-oriented degree that leaves them less marketable. It is possible to do this by introducing a “minor” in sugar engineering.

### Sugar Engineering Courses

ASI is not an academic department, so new courses have been introduced through the Department of Biological and Agricultural Engineering. The two sugar engineering courses offered in the College of Engineering are a course in sugar process engineering, which teaches all the background to sugar processing, and sugar factory design, which goes into the greater depth and detail required in equipment design in a factory.

Pre-requisites for these courses, apart from the basic sciences, are credits in thermodynamics, fluid mechanics, heat transfer and basic electrical engineering.



*Graduate assistant determining the zeta potential of colloids in clarified juice.*

### 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 would be encouraged to work as a part-time student at ASI and would be given 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 be able to 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 to be overseen by the staff of the Audubon Sugar Institute.

Another option involves spending the fall semester at an operating sugar mill, using this period as an internship, yielding 3 credit hours. This could substitute for one of the courses prescribed for the minor, but it would probably require a slightly longer time in which to complete the degree.

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

Students are recruited to earn a master's degree in chemical, mechanical or biological engineering. This is targeted at those who have a first degree and wish to gain some 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. Depending on the first degree of the individual, he or she would be encouraged to choose electives appropriate to providing a strong sugar expertise.

A thesis is required, and this should be related to a relevant sugar-processing topic. In all cases, the student would be encouraged to take on a research assistantship in ASI for the duration of the course.

There are perceived to be major benefits to this education and training initiative to the companies who employ the graduates, to the graduates pursuing a career in the sugar industry and to ASI in terms of its research output.

## Library

The Audubon Sugar Institute Library, under the direction of Dr. Peter Rein, has 716 books, which are cataloged in the office. There are 312 titles, some of which are part of a series such as the Sugar Journal, the International Sugar Journal, Sugar Y Azucar and Zuckerindustrie; they have been bound for easy access. The library is a great reference library for faculty, staff and students using the library for research. A wealth of sugar information can be found in the many theses that are part of the library. The collection not only represents the sugar industry in Louisiana and the United States but has an international flair with many journals from around the world.



## Publications

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- Day, D.F., and Yoo, S.K. (2001). Bacterial Metabolism of X- and B-Pinene and Related Monoterpenes by *Pseudomonas* sp. Strain PIN. *Process Biochemistry*, in press.
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- Rein, P.W. (2001). How Do We Arrive at the Optimum Cane Quality Payment System? *Sugar Bulletin*, 80:3, 9-12.
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- Rein, P.W. (2001). The ISSCT Congress in Australia. *Sugar Bulletin*. 80:2, 10-12.
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- Saska, M., Goudeau, S.L., and Godshall, M.A. (2001). Preliminary Results of Pilot Plant Boiling Tests. ASSCT Meeting, Baton Rouge.
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- Saska, M. (2002). Effect of Clarification Conditions on Non-sugar Removal and Condensate Composition. ASSCT Meeting, Baton Rouge.
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## Faculty and Staff

### Administrative Staff

**Dr. Peter Rein**, Professor and Head – BSc. and MSc. Chemical Engineering (University of Cape Town, South Africa), PhD. Chemical Engineering (University of Natal, South Africa)

**Liz Thompson**, University Administrative Specialist – B.S. Business Administration (Indiana Institute of Technology)

**Lisa Lindsay**, Secretary – B.A. Spanish (Louisiana State University)

### Analytical Lab

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

**Dr. Dorothy Wood**, Post-Doctoral Researcher – B.S. Chemistry (University of Florida), PhD. Chemistry (Louisiana State University)

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

### Factory Staff

**Joe Bell**, 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

**Chris Cavanaugh**, Student Worker

### Faculty and Staff

**Dr. Donal Day**, Professor – BSc. Biochemistry (University of New Hampshire), PhD. 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), PhD. Chemical Engineering (Georgia Institute of Technology)

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

**Dr. Willem H. Kampen**, Associate Professor – B.S. (College of Sugar Tech Amsterdam, Holland), M.S. Chemical Engineering (Louisiana State University), PhD. Food Science (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)

**Lifeng Cui**, Graduate Assistant – B.S. Forestry (Beijing Forestry University, China)

**Hugh Broadhurst**, Graduate Assistant – BSc. Chemical Engineering (University of Natal, South Africa)

**Chang-Ho Chung**, Graduate Assistant – B.S. Food Science (Sejong University, Korea), M.S. Food Science (Sejong University, Korea)

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

**Duwoon Kim**, Graduate Assistant - B.A. Food Science, (Chonnam National University, Korea)

### Adjunct Faculty

**Mary An 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), PhD. Agricultural Engineering (University of Tennessee)

## Representation on Technical Societies and Research Institutes

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

Chairman: P.W. Rein

Co-Products Section Committee: D.F. Day

### SPRI (Sugar Processing Research Institute)

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

### International Sugar Journal

M. Saska (Referee)

### American Society for Microbiology

D.F. Day

### American Society for Advancement of Science

D.F. Day

### Society for Industrial Microbiology

D.F. Day

### Southern Regional Development Committee - USDA

D.F. Day

## Meetings, Conferences, and Workshops Attended

### February 2001

ASCL Annual meeting (H. Birkett, P. Rein)

ASSCT (La. Division), Baton Rouge (H. Birkett, D. Day, W. Kampen, P. Rein, and M. Saska)

### April 2001

Audubon Sugar Institute Conference (all staff and faculty)

### May 2001

SIT, Taiwan (M. Saska)

South China University of Technology, Guangzhou, China

### June 2001

ASCL Contact Committee Meeting (H. Birkett, P. Rein)

ASSCT, New Orleans (H. Birkett, D. Day, W. Kampen, P. Rein and M. Saska and B. White)

### September 2001

ISSCT, Australia (D. Day, W. Kampen, and P. Rein)

### February 2002

ASSCT (La. Division), Baton Rouge (H. Birkett, D. Day, W. Kampen, P. Rein, and M. Saska and B. White)

Forum on the Future of the Sugar Cane Industry in Mexico, Cordoba, Mexico (P. Rein)

### March 2002

SPRI meeting, New Orleans (W. Kampen, P. Rein, B. White, D. Wood, and M. Saska)



## Foreign Visitors to the Institute

### January 2001

Peter Mason (Canada)

Mary Lou Cunningham and Julian M. Cooper (Great Britain)

Martine Decloux (France)

### February 2001

Mullaperdi Narendranath and Lavu Nagesh Kumar (India)

### March 2001

Tim Diringer (Germany)

### June 2001

Young Yun (Korea)

### August 2001

Jose E. Cueto Rodriguez (Mexico); Abdulla Abdel-Gawud (Egypt)

### November 2001

Jose Coronez (Argentina)

### January - March 2002

Joshua Jaddoo (Jamaica); Francois Langrenay (France); Jesus E. Larrahondo (Colombia);

Marc-Andre Theoleyre (France)

Pam Morel du Boil (South Africa)

Mike Inkson (United Kingdom)



*St. James sugar mill, Louisiana.*



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