INVESTIGATIONS OF THE USE OF NEAR INFRARED (NIR) SPECTROSCOPY FOR ON-LINE JUICE ANALYSES

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INTRODUCTION

Recently, Vaccari¹ and others^{2,3,4} have reported very good results determining sucrose and solids concentration in factory juices using near infrared (NIR) techniques. All analytical work appears to have been conducted using equipment supplied by Bran Luebbe. In this study, samples of diffusion juice and thin juice collected at the Betteravia factory of Holly Sugar Corporation were distributed to three major suppliers of NIR equipment for analysis. A requirement made of each supplier was that equipment used for analysis be capable of on-line installation.

Each sample was analyzed at the Betteravia factory for percent solids by refractometry and vacuum oven (sample dried on sand) and for percent sugar by polarimetry and gas chromatography. Each supplier was asked to fit calibration curves to each analytical method for diffusion juice and thin juice.

EQUIPMENT AND PROCEDURES

Suppliers Instrumentation

NIRSystems: Model 6500 On-line Spectrophotometer with

Transmission Pair fiber-optic probes.

Bran+Luebbe: InfraAnalyzer 500 with EDAPT 2 transmission

bench option.

LT Industries: Quantum 1200 NIR analyzer.

Betteravia Instrumentation and Procedures

Refractometry: A Reichert Auto Abbe, maintained at 20 C., was used to estimate refractometric solids. No sample preparation was necessary.

Vacuum Oven: Approximately 1 g of sample was quickly weighed into a tared container half filled with acid washed sand. The container was placed in a vacuum oven (VWR Model 1410) maintained at 70 C and 27 in. Hg until achieving constant weight (about 18 hours). Diffusion juice samples were sieved through a 150 micron screen before weighing.

Polarization: Measurements were made on a Rudolph Autopol IIS. Diffusion juice samples were clarified by adding 2 g of calcium hydroxide and 4 g of aluminum chloride to 200 ml of sample, mixing for one minute and filtering through a RA 226 paper. Thin juice was clarified by adding 2 g of Ecosorb S801 resin (Graver Chemical), dried to 25% moisture, mixing for one minute and filtering through a RA 226 paper.

Gas Chromatography: Samples were analyzed on a Perkin-Elmer Model Sigma 3 using a 6 ft. glass column containing OV-17. Samples were derivatized with TMSI and trehalose was used as an internal standard.

Sample Collection: Test samples were obtained at the Betteravia factory throughout the Fall, 1989 campaign (42 days). To minimize microbiological degradation, 2 ml of 30% formaldehyde per liter was added and samples were stored at 5 C. At the end of the campaign, samples were selected to cover as wide a range of values as possible.

RESULTS AND DISCUSSION

Table 1 lists correlation coefficients and standard errors calculated for each analytical data set from each supplier. A summary of results from each supplier is also given.

TABLE 1
NIR EQUIPMENT; COMPARISON OF RESULTS

		THIN JUICE			JUICE
		Corr	Std	Corr	Std
<u>Analy</u>	Supplier	Coef	Err	Coef	Err
rds	NIRSys	.951	.45	.854	.78
	B+L	.977			
	LT Ind	.987	.25	.933	.22
Vac	NIRSys	.841	. 68	.842	.78
	B+L	.970	.19		
	LT Ind	.981	.29	.996	.16
Pol	NIRSys	.945		.988	.25
	B+L	.955			
	LT Ind	.976	.29	.996	.14
GC	NIRSys	.969	.34	.970	.36
	B+L	.948	.25		
	LT Ind	.968	.36	.994	.18
	AVERAGE RES	ULTS FOR CO	MBINED ANALYSES		
	NIRSys	.927	.48	.914	.54
	B+L	.963	.22		
	LT Ind	.978	.30	.995	.18

Graphs depicting regression analyses and normal histograms for the data used to generate Table 1 are given in Appendixes I and II.

From the combined analyses section, thin juice results from Bran+Luebbe and LT Industries appear to be similar for correlation coefficients and fairly close for standard errors. NIRSystem results are worse for both statistics, and the standard error is probably unacceptable.

Unfortunately, Bran+Luebbe did not have the analytical staffing available to analyze the diffusion juice samples. Between the two suppliers who did perform the analyses, NIRSystem results again are worse and unacceptable. Surprisingly, the LT Industries results for diffusion juice were considerably better than for thin juice.

Table 2 uses the results from Table 1 to compare the average results for the two solids analysis, rds and vacuum oven, and the results for both sugar analysis, polarization and GC. The NIRSystem results were not included because of their relatively poorer values.

TABLE 2
COMPARISON OF ANALYTICAL METHODS

COMPARISON OF AVERAGE RDS AND VACUUM OVEN RESULTS

	RAW JUICE		THIN JUICE		
	Corr Coef	Std Err	Corr Coef	Std Err	
rde	982	22	993	22	

rds		.982	.22		.993	. 22
Vacu	ıum	.976	.24		.996	.16
	COMPARIS	ON OF	AVERAGE	POL AND	GC RESULTS	
Pol		.966	.28		.996	.14
GC		.958	.31		.994	.18

Very little difference was found between either of the solids methods or between the sugar methods. From these results, there apears to be no reason to believe tests more exacting than polarization or rds would be beneficial for establishing calibration curves.

SUMMARY

Ideally, for a comparison like this, all three instruments would have been collected in a single location where factors such as analyst skill level, environmental conditions and sample handling would have been similar. Unfortunately, there is no way to determine if a significant repositioning of suppliers would have occurred had this been possible. Also, had instrumentation been available at the Betteravia factory, additional calibration sets could have been run and process samples evaluated by factory calibrated instruments.

Besides judging how well these instruments have performed with the samples supplied, the intended on-line use suggests other factors such as ruggedness of construction, sample cell design and remote sampling capability need to be explored. All three of the suppliers participating on this study have on-line equipment available with fiber optic sensors. A brief technical specification list for each instrument is given in Appendix III.

How well NIR equipment designed for multi-component analysis performs depends on appropriate software as much as on hardware. And since software can frequently be improved relatively quickly, instrumentation ought to be resurveyed at regular intervals before a decision is made. Additionally, each of these suppliers have auxiliary software packages which could influence a purchasing decision.

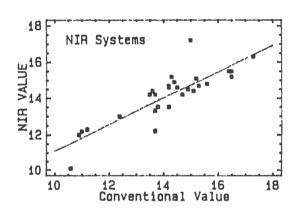
Another factor of possible importance is spectral range. Two suppliers, NIRSystems and LT Industries, supply instruments which cover both the visible (to 400 nm) and near infrared regions. This ability could enable juice color at 420 nm to be included as part of the analysis.

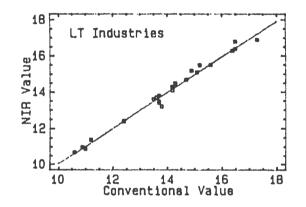
The information gathered during this study suggests that two of the three participating suppliers could be considered for further investigation involving purchase or lease of appropriate equipment for a bench trial at a factory location.

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- G. Vaccari, G. Mantovani, G. Sgualdino, P. Goberti, "Near Infrared Spectroscopy Utilization for Sugar Products Analytical Control", Zuckerindustrie, Sept., 1987, pp 800-807.
- 2. G. Marchetti, "Application of a NIR On-Line Automatic Analyzer System in a Sugar Factory", Int. Sugar Journal, 1990, Vol 92, No. 1102.
- 3. R.V. Ames, S.W. Norton, H.H. Nguyen, "Control of Sugar Polarization and Moisture Using Near Infrared Reflectance Analysis", Sugar Journal, Sept., 1989, pp 7-10.
- 4. E. Burzawa, M. Melle, "Caracterisation Des Produits De Sucrerie Par Reflectance Dans Le Proche Infrarouge", Ind. Aliment. Agr., 1988, Vol 105 [7-8], pp 629-634.

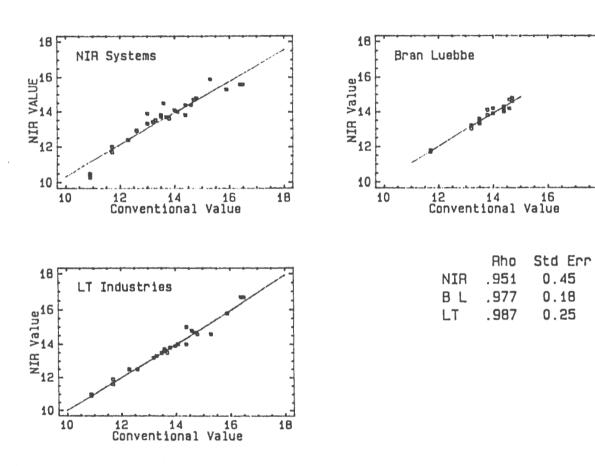
Refractometric Solids in Diffusion Juice



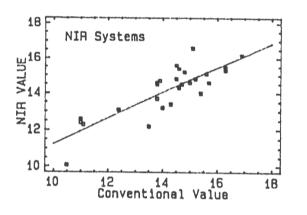


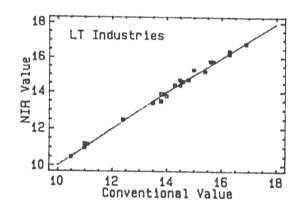
Aho Std Err NIR .854 0.78 LT .993 0.22

Refractometric Solids in Thin Juice



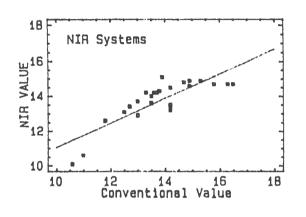
Vacuum Dried Solids in Diffusion Juice

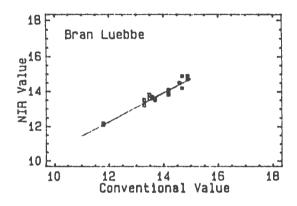


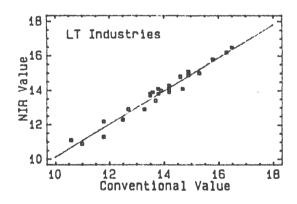


Rho Std Err NIA .842 0.78 LT .996 0.16

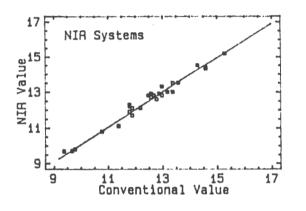
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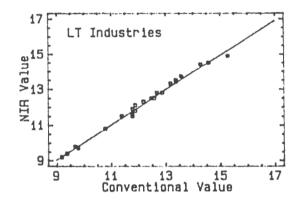






Rho Std Err NIR .841 0.68 B L .970 0.19 LT .981 0.29

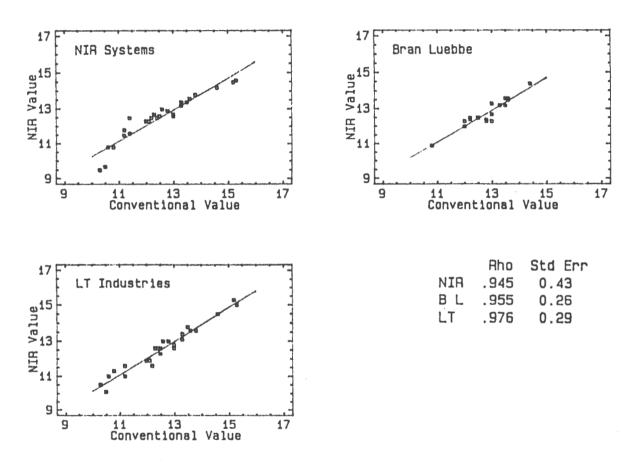




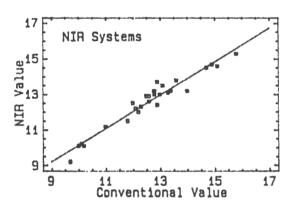
Rho Std Err NIR .988 0.25 LT .996 0.14

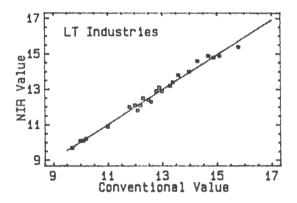
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Polarimetric Sugar in Thin Juice



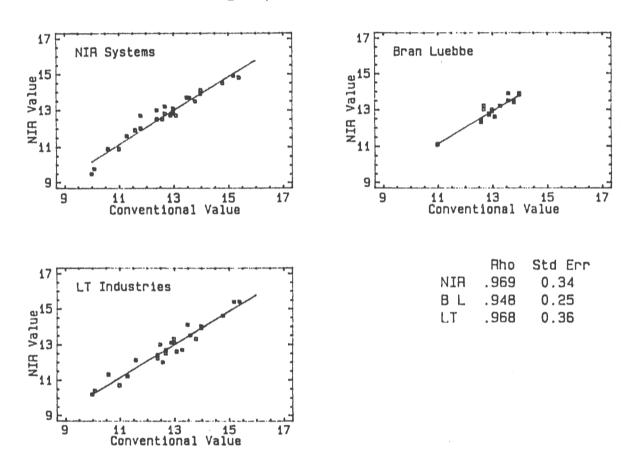
Gas Chromatographic Sucrose in Diffusion Juice



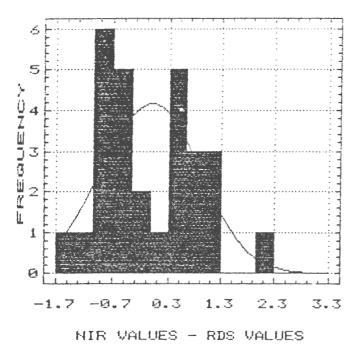


Rho Std Err NIR .970 0.36 LT .994 0.18

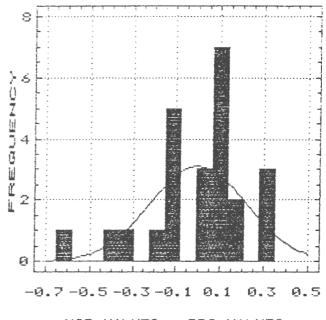
Gas Chromatographic Sucrose in Thin Juice



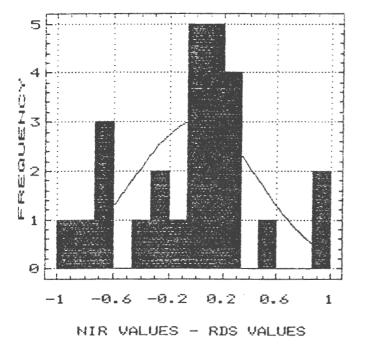
NIRSystems Refract. Est. of Solids in Raw Juice Refract. Est. of Solids in Raw Juice



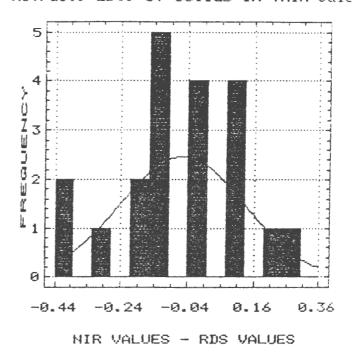
LT Industries



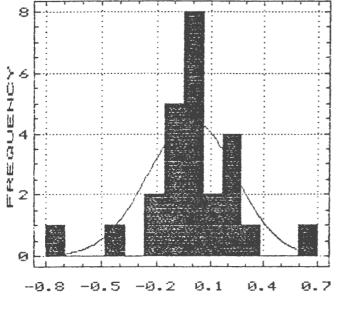
NIRSystems Refract. Est. of Solids in Thin Juice Refract. Est. of Solids in Thin Juice



BRAN+LUEBBE

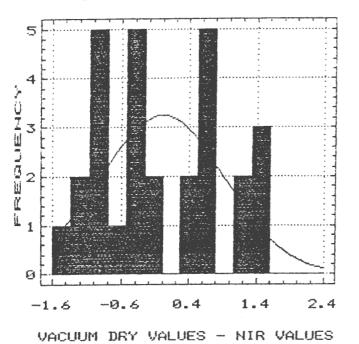


LT Industries Refract. Est. of Solids in Thin Juice

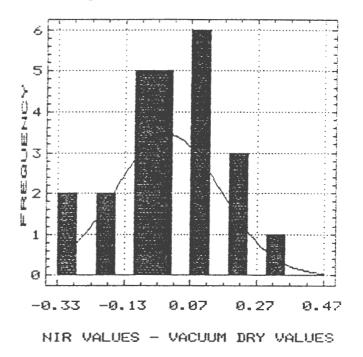


NIR VALUES - RDS VALUES

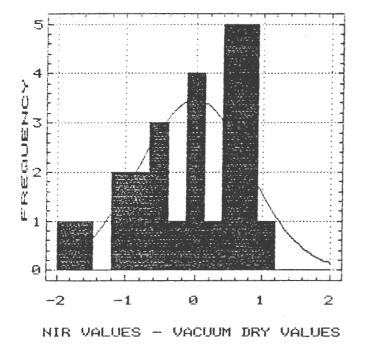
NIRSystems Wac. Dry Est. of Solids in Raw Juice Vac. Dry Est. of Solids in Raw Juice



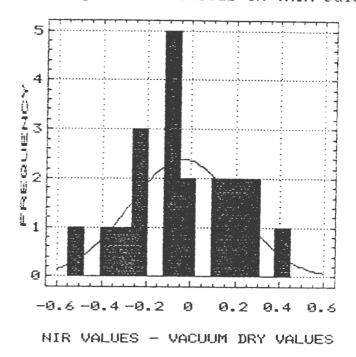
LT Industries



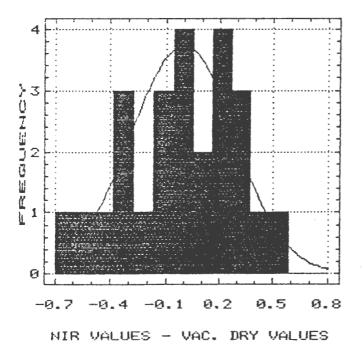
NIRSystems Vac. Dry Est. of Solids in Thin Juice Vac. Dry Est. of Solids in Thin Juice



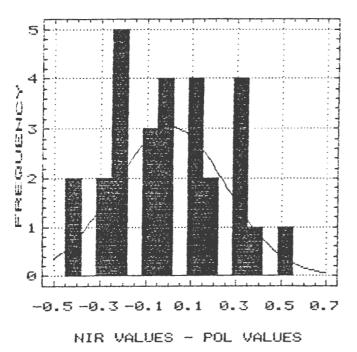
BRAN+LUEBBE



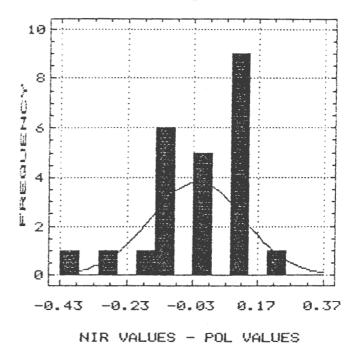
LT Industries Vac. Dry Est. of Solids in Thin Juice



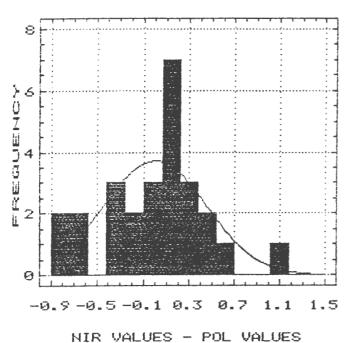
NIRSystems Pol. Est. of Sugar in Raw Juice



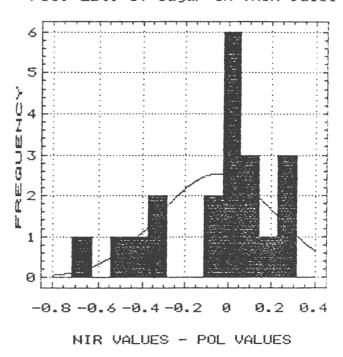
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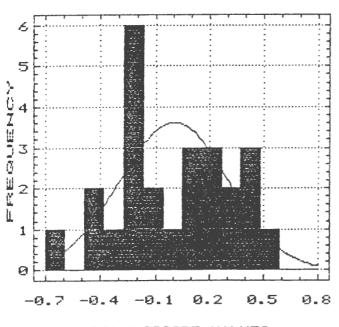
NIRSystems Pol. Est. of Sugar in Thin Juice Pol. Est. of Sugar in Thin Juice



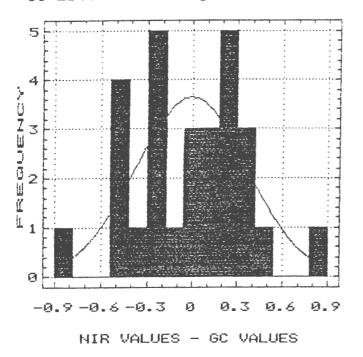
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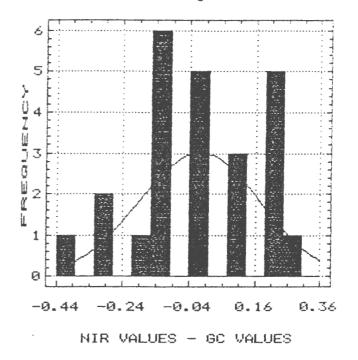
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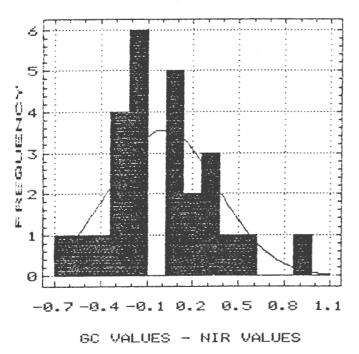
NIRSystems GC Estimate of Sugar in Raw Juice GC Estimate of Sugar in Raw Juice



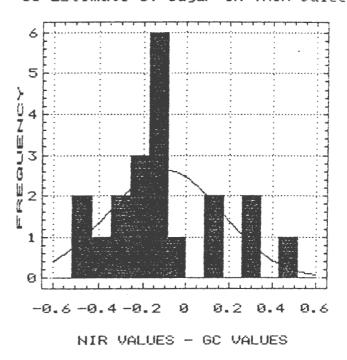
LT Industries



NIRSystems GC Est. of Sugar in Thin Juice GC Estimate of Sugar in Thin Juice



BRAN+LUEBBE



LT Industries GC Estimate of Sugar in Thin Juice

