Direct Spectroscopic Sucrose Determination of Raw Sugar Cane Juices

Frédéric Cadet* and Bernard Offmann

Laboratoire de Biochimie, Faculté des Sciences, Université de la Réunion, 15 Avenue René Cassin, B.P. 7151, 97715 Saint-Denis Messag Cedex 9, Réunion, France

A more accurate, less time-consuming, and nonpolluting spectroscopic method than the currently used HPLC or polarimetric methods is proposed for the routine quantitative determination of sucrose in complex biological samples. Opaque raw sugar cane juices representative of a sugar cane harvest are analyzed by Fourier transformed mid-infrared attenuated total reflectance, and the spectral data are processed by principal component analysis (PCA) and principal component regression (PCR). The most suitable region for the measurement of sucrose was found to be the 1250−800 cm^{-1} region. The spectroscopic representation of the first axis as assessed by PCA in this spectral region featured characteristic absorption bands of sucrose. By PCR on the spectral data from a calibration set, a prediction equation was established to predict sucrose content in unknown samples. Good overall predictions were obtained. The values of the predicted sucrose concentration were more accurate (bias = 0.041 g/100 mL) than those obtained by direct polarimetry (bias = −0.163 g/100 mL). The method is validated on a panel of 1267 samples representative of a sugar cane harvest.

Keywords: *Raw sugar cane juices; mid-FTIR; PCR; sucrose*

INTRODUCTION

Sucrose is the principal product of the photosynthesis process and is the most widespread sugar in the plant kingdom. Sucrose is a renewable chemical resource and has considerable commercial value in the chemical, microbiological, pharmaceutical, and food industries.

One of the most essential concerns in the sugar cane industry is to determine the price of the sugar canes that are delivered and sold to the miller. The price of sugar cane is estimated by its sucrose content. Not only is the efficiency of the method used to determine sucrose content in sugar cane critical, but the delay and costs of the analyses are also important.

The officially adopted methods for the routine analysis of sucrose content in sugar cane juice vary from country to country. In some countries the classical and physical refractrometric and polarimetric methods are used, while other countries use chromatographic methods such as HPLC or GLC (Wong Sak Hoi, 1982; Honda, 1984; Meade and Chen, 1985). In all cases, routine analysis can only be done on processed juices. As for example, for polarimetric measures, the raw juices that are obtained from pressed sugar cane at high pressure contain fibers and impurities that make the solution opaque. These juices need to be processed with lead acetate, which precipitates impurities, and then filtered on a cellulose filter so as to obtain clarified juices ready for analysis. About 20 min is necessary to conduct these operations.

The most used methods currently are polarimetry (Schneider, 1985) and HPLC (Clarke, 1985). It has been shown that polarimetric measurements underestimate sugar content (Meade and Chen, 1985; Brokensha *et al.*, 1978; Cadet *et al.*, 1991). Measuments by HPLC are not precise enough, and the results are not easily reproducible for routine analysis (Rouch *et al.*, 1995).

* Author to whom correspondence should be addressed [telephone Intl (262) 93 82 02; fax Intl (262) 93 81 66].

Spectroscopic methods such as near-infrared or mid-infrared can be easily adapted for routine use for industrial control of food products.

Rapid analytical methods have developed considerably since the advent of near-infrared reflectance spectroscopy (NIR) between 1960 and 1970 (Norris, 1978). More and more constituents and products are now being analyzed according to this method (Osborne, 1981; Williams and Norris, 1987). The spectroscopic method is most widely used in the food industry for the quantitative measurement of major biochemical products.

Few studies have focused on the use of the MIR range for analysis of food products since mid-infrared reflectance (MIR) spectra are complex and since water contained in biological products is a strong IR absorber. However, with the advent of Fourier transform infrared spectroscopy in parallel with the use of powerful microcomputers and with the advent of new spectroscopic techniques such as attenuated total reflectance (ATR), MIR spectroscopy has developed considerably. ATR is an analytical technique that has great potential with regard to analysis of food products (Depecker *et al.*, 1985; Crocombe *et al.*, 1987; Van de Voort and Ismail, 1991; Cadet *et al.*, 1991).

There are different mathematical methods that can be used for the processing of complex infrared spectral data. Only recently has principal component analysis (PCA) been used for the study of transmission MIR spectra (Antoon *et al.*, 1979; Gillette and Koenig, 1982). PCA was used for the first time for the study of diffused reflection near-IR spectra by Bertrand *et al.* (1984) and Cowe and McNicol (1985). The aim of this work was to evaluate PCA and principal component regression (PCR) mathematical treatments for the description and processing of mid-infrared spectral data of raw sugar cane juices. This method has been tested on 1267 samples obtained throughout a sugar cane harvest time (4 months).

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MATERIALS AND METHODS

Raw Sugar Cane Juices. Sampling of sugar was done by coring. The average core was about 7 kg. After pulverization, a subsample of approximately 1 kg was removed. A hydraulic press was used to extract juice from the samples obtained from the coring and from the disintegrator. The sample was pressed for 2.5 min at 250 bar.

Mid-infrared ATR Spectra. Mid-Fourier transform infrared (mid-FTIR) spectra were collected on a Michelson 100 Fourier transform spectrophotometer. ATR spectra were obtained with a Specac Overhead ATR system. The crystal of the reflectance element is made from zinc selenide, a material that is quite inert to water; it is quite rapidly cleaned between samples by spraying with water and then dried with filter paper.

The data were recorded from 700 to 5000 cm^{-1} in 4 cm^{-1} increments at log(1/*R*), in which *R* is the ratio of the reflected intensity for the background to that of the sample. Although the ATR experiment does involve the reflection of the radiation within a crystal, the interaction of the radiation with the sample is the transmittance of radiation through the sample; this depth of penetration is wavelength dependent, but it is passing through a finite layer of the sample. For this reason, plots can be read according to absorbance (or transmittance). The combination of four scans resulted in an average spectrum. The intensity the spectra was low; the highest peaks had log(1/*R*) values <0.6 on baseline spectra.

Mathematical Treatment. Mathematical treatments were performed on a Compaq 486 personal computer with software written in C language and developed in our laboratory. Multidimensional statistical analyses, such as PCA, describe variation in multidimensional data by few synthetic variables. These synthetic variables are a linear combination of all the original variables and have the advantage of having no correlation with each other. Simpler descriptions of data sets are thus obtained with minimal loss of information. These treatments were used for morphological analysis of spectra (Le Nouvel, 1981) and for graphical representation of spectra similarity (Devaux *et al.*, 1988).

PCA was applied to the spectra from 800 to 1250 cm^{-1} (with 235 data points used as principal variables). Spectra were centered prior to PCA according to

$$X_{ij} = A_{ij} - A_j - A_i + A \quad (1)$$

where X_{ij} = centered data, A_{ij} = spectral data (log 1/*R*) of spectrum *i* and wavelength *j*, A_j = mean value of spectral data at wavelength *j* for every spectrum, A_i = mean value of spectral data of spectrum *i* for every wavelength, and A = average mean of all spectral data in the collection.

PCR was used to establish a prediction equation. PCR is basically a multilinear regression applied to scores assessed by PCA (Lefebvre, 1983; Lebart *et al.*, 1977). Interest in the introduction of scores according to their predictive ability had already been shown (Dagnelie, 1975; Bertrand *et al.*, 1987).

Concentrations are predicted according to

$$\mathbf{C}_{nl} = \mathbf{X}_{nk} \cdot \mathbf{V}_{kp} \cdot \mathbf{R}_{pl} \quad (2)$$

where **C** is the column vector of predicted concentrations, **X** is the centered matrix of spectral data, **V** is the matrix of latent vectors of PCA, and **R** is the column vector of the regression coefficients of the prediction equations. *n*, *k*, and *p* are, respectively, the number of samples, the number of wavelengthss, and the number of significant principal components. The dot product **V·R** is a vector, the components of which may be interpreted in terms of absorption bands. Plotting the components against the corresponding wavelengths gives a spectral pattern. Peaks correspond to absorption bands which are characteristic of the measured chemical constituents. Hollows indicate that when the concentration increases, the corresponding absorption bands will decrease (Bertrand *et al.*, 1988).

Calibration and Verification Sets. The calibration set is constituted of 107 collected spectra of raw sugar cane juices that are representative of a sugar harvest. The verification set is constituted of 1267 samples distributed in 33 families of about 39 sample spectra that cover the entire sugar harvest and different geographical regions.

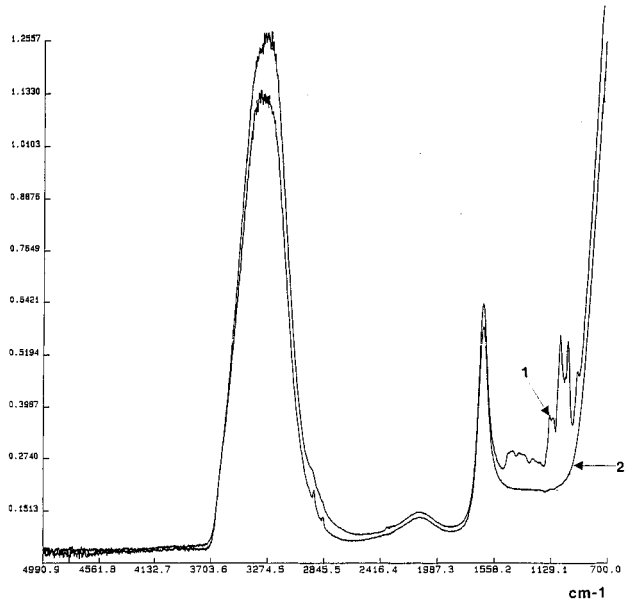

Figure 1. Mid-FTIR ATR spectra of a sample of raw sugar cane juice (1) and of water (2).

Reference Values. The reference sucrose content in raw sugar cane juices was measured using the corrected polarimetric method as described previously (Cadet *et al.*, 1992; Rouch *et al.*, 1995).

RESULTS AND DISCUSSION

Analytical application traditionally requires four steps: (i) the search for and analysis by a standard reference method of representative samples of the products; (ii) recording of the infrared spectra of these samples; (iii) setting up a prediction equation that links the chemical data to the spectral data; and (iv) application of a prediction equation to samples of unknown composition.

The various problems linked to sampling methods can be overcome by ATR. This technique can be used to study samples that are difficult to analyze according to traditional spectroscopic methods. This is the case for raw sugar cane juice, which is a completely opaque solution.

Selection of Spectral Range and Spectra. The spectra of raw sugar cane juice that is observed between 5000 and 700 cm^{-1} show three major absorption zones: 3700−2800, 1800−1470, and 1250−800 cm^{-1} (Figure 1).

When the spectrum of water is analyzed and compared to the sugar cane solution spectra, the first two zones can be assigned to water. The 1250−800 cm^{-1} zone corresponds to the absorption zones of the three major constituents of sugar cane juice: sucrose, glucose, and fructose.

Measurements on raw sugar cane juices should be carried out rapidly since fermentation reactions that alter sugar content rapidly take place (Rouch *et al.*, 1995). Raw juices need to be filtered before analysis; Figure 2 shows changes in raw sugar cane juice spectra. There is an increase in absorbance owing to the precipitation of impurities and fibers. Hence, filtration is necessary before the juice is analyzed by infrared spectroscopy. This is instantaneously done by an adequate porosity plastic filter when the ATR cell is filled.

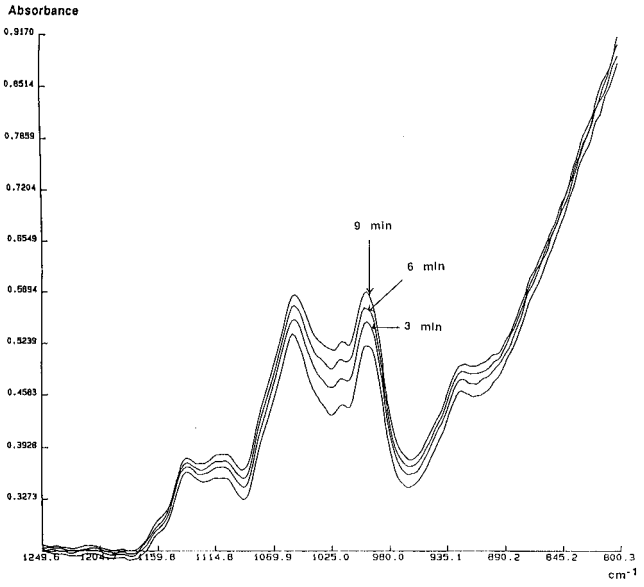

Figure 2. Effect of precipitation of impurities on mid-FTIR spectra of raw sugar cane juices.

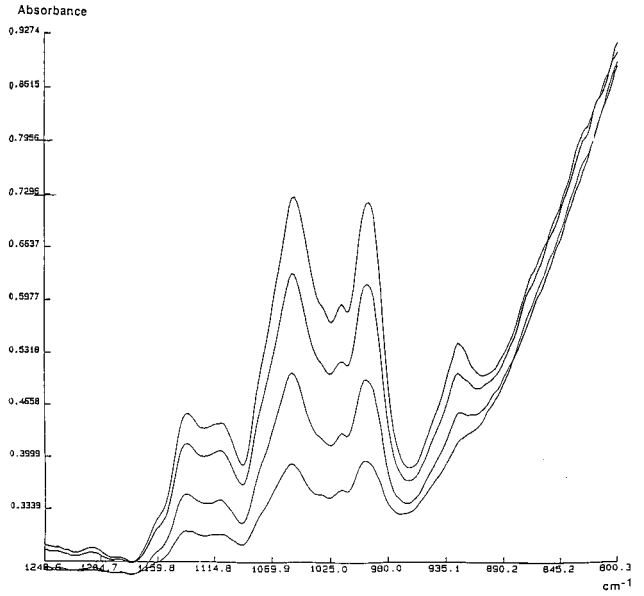

Figure 3. Mid-FTIR ATR spectra of pure sucrose solutions at different concentrations (10, 20, 30, and 40 g/100 mL).

The spectra of a pure sucrose solution at different concentrations (Figure 3) show that the intensities of absorbance vary significantly with concentration. This suggests that sucrose content could be quantified. However, the sucrose content in sugar cane juice cannot be measured directly from the height of a peak since the resulting spectra include three sugars (sucrose, glucose, and fructose) in various concentrations. The influence of fructose and glucose is not negligible. Hence, it is necessary to extract, from the spectral data, information concerning sucrose only. This can be done through PCA. From prediction equations established by PCR sucrose content can then be predicted.

The spectral region that is the most representative of sucrose has been determined. Several zones have been tested: 880−1175 cm^{-1} (153 points), 800−1250 cm^{-1} (235 points), 900−1175 cm^{-1} (143 points), 900−1250 cm^{-1} (182 points), 900−1500 cm^{-1} (311 points), and 975−1015 cm^{-1} (21 points). Three criteria were used for evaluating the different spectral regions: the percentage of inertia, the first correlation coefficient between the chemical data and the first axis of PCA, and, finally, the standard deviation of the difference between the predicted values and the reference values. As shown in Table 1, the percentage of inertia did vary with the spectral regions but none could be selected without ambiguity since no linear difference in function of the spectral region was found between the values; whereas the best inertia percentages were obtained for 800−1175 cm^{-1} with the first two families (97.01% and 96.54%, respectively), for the third family, it was the 975−1015 cm^{-1} region for which the best inertia percentage was obtained (93.73%) .

Table 1. Influence of the Spectral Region on the Inertia Percentage of Axis 1 As Assessed by PCA on the Calibration Set (107 Samples)

spectral region (cm^{-1})	family 1[a]	family 2[a]	family 3[a]
800−1175	97.01	96.54	89.01
800−1250	96.91	95.71	87.06
900−1175	96.37	95.64	86.74
900−1250	96.38	95.06	83.12
900−1500	79.22	67.11	64.96
975−1015	94.48	94.48	93.73

[a] Each family is constituted of 39 raw sugar cane juice spectra.

Table 2. Influence of the Spectral Region on the First Correlation Coefficient between Spectral Data and Chemical Data As Assessed by PCA on the Calibration Set (107 Samples)

spectral region (cm^{-1})	family 1[a]	family 2[a]	family 3[a]
800−1175	0.98672	0.98083	0.98725
800−1250	0.98614	0.97741	0.9465
900−1175	0.98645	0.98253	0.98601
900−1250	0.98459	0.98514	0.94593
900−1500	0.88907	0.94699	0.66984
975−1015	0.98201	0.98914	0.93916

[a] Each family is constituted of 39 raw sugar cane juice spectra.

Table 3. Standard Deviation Values of the Difference between Predicted Values and Reference Sucrose Content Values According to the Spectral Region

	spectra region (cm^{-1})		
family[a]	880−1175	800−1250	900−1500
1	0.1576	0.1144	0.1484
2	0.1671	0.1006	0.1458
3	0.2793	0.181	0.3036
4	0.2382	0.1551	0.2696
5	0.2615	0.1831	0.2347
6	0.2052	0.146	0.1701
7	0.1713	0.1605	0.1799
8	0.4985	0.4329	0.4742

[a] Each family is constituted of 39 raw sugar cane juice spectra.

The value of the first correlation coefficient between chemical data and spectral data cannot be used for similar reasons (Table 2); whereas the best correlation coefficient values were obtained in the 800−1175 cm^{-1} range with the first and third families (0.9872 and 0.9873, respectively), for family 2, the best correlation coefficent value was obtained in the 975−1015 cm^{-1} region. On the other hand, it appeared from the standard deviation values of the differences between predicted and reference values that the best results are obtained in the 800−1250 cm^{-1} spectral region when compared to the 800−1175 and 900−1500 cm^{-1} regions (Table 3). With other spectral regions, identical conclusions were reached (data not shown). The 800−1250 cm^{-1} region had hence been selected for the determination of sucrose content in raw sugar cane juice.

Multidimensional Analysis and Predictions. Multidimensional analyses give a simple way to have a

Table 4. Predicted and Reference Values of Sucrose Content for One Family

sample	ref	pred	sample	ref	pred
1	22.865	23.007	21	19.762	19.78
2	19.513	19.884	22	19.711	19.708
3	16.706	16.83	23	19.801	19.988
4	19.462	19.513	24	18.716	18.926
5	20.881	21.05	25	19.458	19.146
6	19.967	20.05	26	21.85	21.601
7	20.751	20.926	27	22.635	21.998
8	21.041	20.838	28	23.417	22.877
9	19.621	19.856	29	20.579	20.476
10	19.961	20.127	30	19.986	19.828
11	18.,801	19.014	31	19.729	19.395
12	18.977	19.158	32	20.128	19.738
13	19.937	20.079	33	20.231	19.65
14	19.441	19.478	34	20.051	19.875
15	19.545	19.545	35	18.539	18.184
16	21.031	21.172	36	19.513	19.165
17	19.169	19.423	37	21.226	29.744
18	19.87	20.066	38	19.973	19.739
19	15.354	15.729	39	21.894	21.16
20	17.385	17.458			
diff between pred and ref values				bias	−0.059
				SD	0.293

[a] The predicted equation was established by PCR from the calibration set spectral data.

global description of a set of variables. They have been applied in a wide range of different fields (psychology, economy, marketing, biology, ...) to give pictorial representation of data. MIR spectral data can be treated almost in the same way as chemical data. Multidimensional analyses work on a rectangular table, where the rows are called "observation" and the columns "variables". They lie in the extension of the usual three-dimensional space to multidimensional space. An analysis is simply an attempt to find a new system of axes better adapted to the description of data than the old one. This creation of axes can be done with no assumption of the nature of the differences between samples; this descriptive method corresponds to PCA.

A sample of 107 collected spectra of raw sugar cane juice representative of all the families from the harvest was grouped into a single calibration set and entered into a PCA. Figure 4 illustrates the PCA factorial map of axis 1 against axis 2 obtained from a calibration set (107 individuals). These first two axes of the PCA of the MIR spectral data represented 78% and 11%, respectively, of the total inertia. The distribution of these individuals with regard to the two axes was a function of the concentration of the juices. The samples were distributed following a sucrose concentration gradient.

The collection of spectra is modeled by PCA into a sum of characteristic signals which form a spectral pattern (Le Nouvel, 1981; Robert *et al.*, 1987). This spectral representation of the principal component of PCA features characteristic absorption bands of biochemical constituents in a sample.

Figure 5 shows the spectral pattern of the principal component (axis 1) of PCA obtained from this calibration set. The spectral representation of the principal component is similar to that obtained with clarified sugar cane juice (Cadet *et al.*, 1991); no shifts in the absorption bands were noticed, thus indicating that the influence of impurities in the raw sugar cane juice is negligible with regard to the quality of the spectral information.

In the present case the spectral pattern that is obtained can be easily interpreted. The absorption bands at 1134, 1048, 992, and 924 cm^{-1} are characteristic of sucrose.

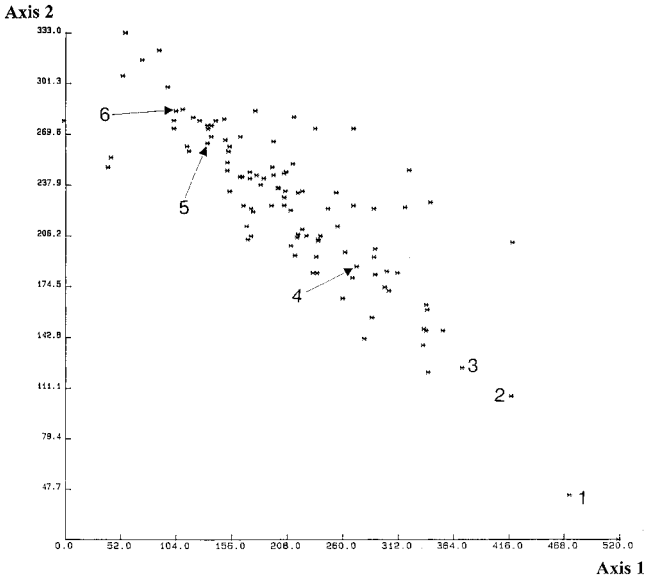

Figure 4. Factorial map of axis 1 against axis 2 of the calibration set as assessed by PCA. The samples are distibuted along a concentration gradient: 12.930% (1), 13.966% (2), 15.916% (3), 17.643% (4), 21.544% (5), 22.021% (6).

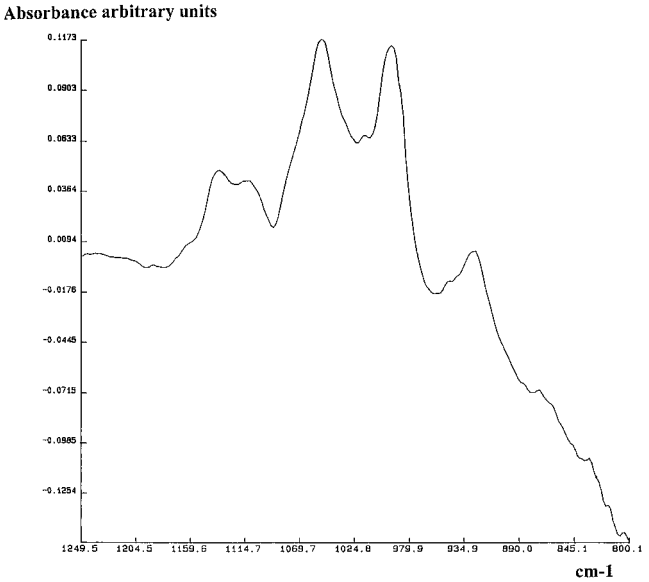

Figure 5. Spectral representation (pattern) of the first axis of PCA of the calibration set.

The sucrose content chemical values for all of the raw sugar cane juices throughout a sugar cane harvest (400 000 analyses) were distributed according to the normal distribution law (data not shown). From these, the spectra of 1267 samples that came from four different geographical regions and that were collected on the 4 months covering the sugar cane harvest were measured and divided into 33 families. The reference sucrose content values obtained for the 33 families were between 7.24% and 23.625% (Table 5) with mean and standard deviation (SD) values of 18.910 and 1.410, respectively. For the calibration set (107 spectra), the chemical values obtained were between 11.842% and 23.154% (mean = 19.656%, SD = 2.168), and the individuals were again distributed according to the normal distribution law (data not shown).

By PCR of the principal components of the calibration set a prediction equation that links spectral data to sucrose content was established. On the 106 axes issued from PCA, the first introduced component gave

Table 5. Mean and SD Values of the Difference between Predicted Values and Reference Sucrose Content Values (Grams/100 mL) for All 1267 Samples (33 Families)[a]

family	minimum sucrose content	maximum sucrose content	SD	mean
1	12.93	21.535	0.262	0.285
2	17.736	22.831	0.166	−0.426
3	19.621	22.394	0.119	−0.009
4	13.682	22.916	0.290	−0.073
5	15.35	23.417	0.293	−0.059
6	13.673	21.383	0.587	−0.005
7	15.976	21.646	0.215	0.125
8	15.715	19.691	0.455	−0.136
9	11.63	20.729	0.363	0.050
10	17.728	20.884	0.396	−0.636
11	14.23	19.657	0.336	0.191
12	10.702	20.39	0.589	0.556
13	14.072	20.303	0.318	−0.353
14	16.592	20.778	0.152	0.500
15	11.074	21.093	0.199	0.471
16	14.782	21.399	0.262	0.319
17	19.441	22.981	0.234	0.003
18	16.234	23.175	9.296	0.049
19	11.623	19.827	0.194	0.242
20	14.004	22.621	0.174	0.153
21	17.704	22.449	0.282	0.199
22	17.224	19.182	0.145	−0.053
23	13.751	20.755	0.425	−0.145
24	20.508	22.452	0.199	−0.134
25	12.906	23.625	0.198	−0.015
26	7.248	21.532	0.485	0.340
27	18.345	19.595	0.204	0.105
28	13.342	22.403	0.179	0.071
29	13.953	24.307	0.568	0.159
30	18.668	21.955	0.164	−0.113
31	17.893	20.11	0.202	0.240
32	9.776	22.4	0.184	−0.036
33	12.824	21.327	0.495	−0.503
mean			0.289	0.041

[a] Prediction equation was established by PCR on principal component assessed from the calibration set.

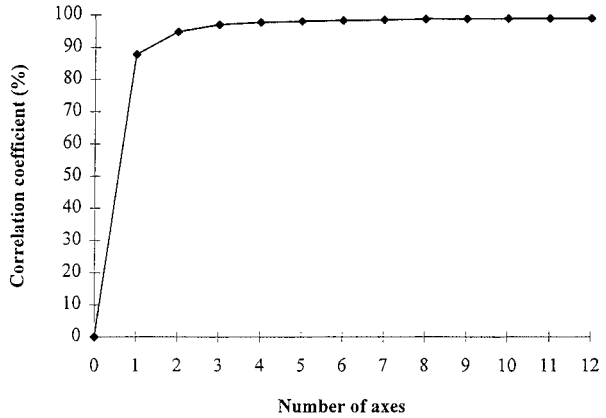

Figure 6. Influence of the number of axes on the correlation coefficient between chemical data and the spectral data.

a correlation coefficient equal to 0.878 and reached 0.948 with the second introduced component. Other PCA axes improved the prediction equation (Figure 6). A correlation coefficient value of 0.981 was reached when five axes were entered in PCR. The other axes did not significantly improve the results, so the prediction equation was established on the first five axes. The sucrose concentrations of the 1267 samples from the verification set were predicted from the prediction equation hence established. All 33 families were tested. The predicted and reference sucrose content values for one family (composed of 39 individuals) are given in Table 4 as an example. The mean (bias) of the difference between predicted and reference values (as determined by corrected polarimetric measurements) of the raw sugar cane juices for all families (1267 individuals) are given in Table 5.

For all 33 families, the bias and the standard deviation values were 0.041 and 0.289 g/100 mL, respectively. The mean and standard deviation of the difference between direct polarimetric measurements and corrected polarimetric reference values are −0.163 and 0.087 g/100 mL, respectively (Cadet *et al.*, 1991, 1992). The polarimetric method (presently used in industrial processing) hence underestimated sucrose content by 0.163 g/100 mL with a negligible error equal to 0.087 g/100 mL. The method developed and reported in this paper gives sucrose content values that are very close to reference values (very weak overestimation of 0.041 g/100 mL) with a SD of 0.289 g/100 mL.

In fact, the difference between the predicted sucrose content values and the reference values (corrected polarimetric measurements) is much lower than that between direct polarimetric estimation and the reference values. The accuracy of the proposed method is therefore much better than that of the current direct polarimetric method.

We have also previously shown that the HPLC method was not reproductible enough for routine quantification of sucrose in an industrial environment (Rouch *et al.*, 1995).

The method described in this paper hence appears to be more convenient than the HPLC and polarimetric methods, particularly for routine analysis of sucrose content in the sugar cane process.

Conclusion. In this paper we have established the possibilty of using MIR spectroscopy for analysis of raw sugar cane juice. When one is working with raw juices, pollutant lead acetate consumption costs and release are prevented and the preliminary filtration processing (20 min long) is no longer necessary. This method is shown to be more accurate than the commonly used polarimetric technique and more convenient than HPLC. It can be easily adapted to routine analysis in an industrial process. The entire procedure, from the analysis of raw sugar cane juice to its predicted sucrose content value, needs no more than 25 s (mean time) per sample with our nonautomated process. This time could be considerably reduced if the whole procedure is automated. This MIR coupled to multidimensional statistical analysis technique is validated on a sample that is representative of a whole sugar cane harvest campaign and could be substituted for polarimetric and chromatographic methods that are traditionally used. The precision of such a method could be improved by correcting the spectral information (baseline and subsequent distortion corrections), and these have been the subject of other publications.

LITERATURE CITED

Antoon, M. K.; D'esposito, L.; Koenig, J. L. Factor analysis applied to Fourier transform infrared spectra. *Appl. Spectrosc.* **1979**, *33*, 351−357.

Bertrand, D.; Robert, P.; Tran, V. Traitement mathematiques des spectres NIR de melanges. Presented at the 11ème Congrès de l'Association Internationale de Chimie Ceréalière, Vienna Austria, June 6, 1984.

Bertrand, D.; Lila, M.; Furtoss, V.; Robert, P.; Downey, G. Application of principal component analysis to the prediction of lucerne forage protein content and in vitro dry matter digestibility by NIR spectroscopy. *J. Sci. Food Agric.* **1987**, *41*, 299−307.

Bertrand, D.; Robert, P.; Devaux, M. F.; Abecassis, J. Assignment of near infrared Absorption bands by multidimensional analyses of spectral data. In *Analytical Applications of Spectroscopy*; Creaser, C. S., Davies, A. M. C., Eds.; Royal Society of Chemistry: London, 1988; pp 450−455.

Brokensa, M. A.; Niemeyer, R. H.; Schaffler, K. J. A comparison of the estimation of sucrose in sugar cane juice by polarimetric and gas liquid chromatography methods. *Proceedings of the 52nd Congress of the South African Sugar Technology Association*; 1978; p 54.

Cadet, F.; Bertrand, D.; Robert, P.; Maillot, J.; Dieudonné, J.; Rouch, C. Quantitative determination of sugar cane sucrose by multidimensional statistical analysis of thier mid-infrared attenuated total reflectance spectra. *Appl. Spectrosc.* **1991**, *45* (2), 166−172.

Cadet, F.; Maillot, J.; Rouch, C.; Conan, J. Y. Analyse de jus de cannes à sucre. Comparaison de differentes methodes de determination du saccharose. *J. Nat.* **1992**, *4* (1), 10−18.

Clarke, M. A. HPLC in sugar industry. An overview. *Sugar Azucar* **1985**, *80*, 8−14.

Cowe, I. A.; McNicol, J. The use of principal component in the analysis of near infrared spectra. *Appl. Spectrosc.* **1985**, *39*, 257−265.

Crocombe, R. A.; Olson, N. L.; Hills, S. L. *Quantitative Fourier Transform Infrared Methods for Real Complex Samples*; American Society for Testing and Materials: Philadelphia, 1987; pp 95−130.

Dagnelie, P. *Analyse Statistique à Plusieurs Variables*; Les Presses Agronomiques: Gembloux, Belgium, 1975; pp 185−190.

Depecker, C.; Legrand, P.; Merlin, J. C.; Sombret, B. Contribution de la reflexion diffuse à l'étude de composés biologiques. *Spectrosc. Biol. Mol.* **1985**, 69−75.

Devaux, M. F.; Bertrand, D.; Robert, P.; Qannari, V. Application of multidimensional analysis to the extraction of discriminant spectral patterns from NIR spectra. *Appl. Spectrosc.* **1988**, *42* (6), 1015−1020.

Gillette, P. C.; Koenig, J. L. Noise reduction via factor analysis in FT-IR spectra. *Appl. Spectrosc.* **1982**, *36* (5), 535−539.

Honda, S. High performance liquid chromatography of mono and oligosaccharides. *Anal. Biochem.* **1984**, *140*, 1−8.

Lebart, L.; Morineau, A.; Tabard, N. *Techniques de la Description Stastitiques*; Dunod: Paris, 1977; pp 7−46.

Lefebvre, J. *Introduction aux Analyses Statistiques Multidimensionnelles*, 3rd ed.; Masson: Paris, 1983; pp 137−148.

Le Nouvel, J. Etude d'une famille de courbes par des méthodes d'analyse des données. Application à l'analyse morphologique de courbes provenant de données médicales. These de 3ème cycle, Université de Rennes I, France, 1981.

Meade, G. P.; Chen, J. C. P. *Sugar Cane Handbook*, 11th ed.; Wiley: New York, 1985.

Norris, K. H. Near infrared reflectance spectroscopy. The present and future. In *Cereal 78: Better Nutrition for the World's Millions*; Sixth International Cereal and Bread Congress; American Association of Cereal Chemists: St. Paul, MN, 1978.

Osborne, B. G. Principles and practice of near infrared (NIR) reflectance analysis. *J. Food Technol.* **1981**, *16*, 13−19.

Robert, P.; Bertrand, D.; Devaux, M. F.; Grappin, R. Multivariate analysis applied to near infrared spectra of milk. *Anal. Chem.* **1987**, *59* (17), 2187−2191.

Rouch, C.; Cadet, F.; Maillot, J. Storage and analysis of cane juice samples. *Int. Sugar. J.* **1995**, *97* (1156), 154−159.

Schneider, F. *Sugar Analysis*; ICUMSA: Dublin, Ireland, 1982.

Van de voort, F. R.; Ismail, A. A. A rapid FTIR quality control method for fat and moisture determination in butter. *Trends Food Sci. Technol.* **1991**, 13−17.

Williams, P.; Norris, K. *Near-Infrared Technology in the Agricultural and Food Industry*; American Association of Cereal Chemists: St. Paul, MN, 1987; 330 pp.

Wong Sak Hoi, Y. L. Gas-liquid chromatographic determination of fructose, glucose and sucrose in cane sugar products. *Int. Sugar J.* **1982**, *84* (999), 68−74.

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