Industrial Validation of Fourier Transform Infrared *trans* and Iodine Value Analyses of Fats And Oils

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ABSTRACT: A Fourier transform infrared (FTIR) edible oil analysis package designed to simultaneously analyze for *trans* content, *cis* content, iodine value (IV), and saponification number (SN) of neat fats and oils by using calibrations based on pure triglycerides and derived by application of partial-least-squares (PLS) regression was assessed and validated. More than 100 hydrogenated rapeseed and soybean samples were analyzed by using the edible oil analysis package as well as the newly proposed modification of the AOCS IR *trans* method with trielaidin in a *trans*-free oil as a basis for calibration. In addition, ~1/3 of the samples were subsequently reanalyzed by gas chromatography (GC) for IV and *trans* content. The PLS approach predicted somewhat higher *trans* values than the modified AOCS IR method, which was traced to a combination of the inclusion of trilinolelaidin in the calibration set and the effects of baseline fluctuations. Eliminating trilinolelaidin from the triglyceride standards and the use of second-derivative spectra to remove baseline fluctuations produced excellent concurrence between the PLS and modified AOCS IR methods (mean difference of 0.61% *trans*). Excellent internal consistency was obtained between the IV and *cis* and *trans* data provided by the edible oil analysis package, and the relationship was close to that theoretically expected [IV = 0.86 (*cis* + *trans*)]. The IV data calculated for the GC-analyzed samples matched the PLS IV predictions within 1 IV unit. The *trans* results obtained by both IR methods were linearly related to the GC data; however, as is commonly observed, the GC values were significantly lower than the IR values, the GC and IR data being related by a slope factor of ~0.88, with an SD of ~0.80. The concurrence between the *trans* data obtained by the two FTIR methods, and between the FTIR and GC–IV data, as well as the internal consistency of the IV, *cis* and *trans* FTIR predictions, provides strong experimental evidence that the edible oil analytical package measures all three variables accurately.

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KEY WORDS: *Cis,* FTIR spectroscopy, fats and oils, Fourier transform infrared spectroscopy, iodine value (IV), lipid analysis, oil analysis, *trans, trans* analysis, triglycerides.

Edible fats and oils vary in their triglyceride makeup, relative degree and forms of unsaturation (*cis* or *trans*), weight-average molecular weight, and overall fatty acid composition/distribution, and these complex determinants define the physicochemical properties of the lipid system. For stability and functionality reasons, oils are often hydrogenated and converted into fats. Hydrogenation reduces the overall degree of unsaturation, but also leads to increased levels of *trans* fatty acids, now of increasing concern to health professionals due to their association with heart disease (1). *Trans* fatty acid levels can reach values of 40% or more in hardened fats, and consideration is being given to legislation to require the labeling of the *trans* content in fat-based products, such as margarines, spreads and frying fats (2). For oil processors, iodine value (IV) and *trans* content are important process parameters, which require monitoring during hydrogenation to ensure that consistent products with desired physical properties are produced. As such, the availability of a simple, rapid and routine means of determining IV and *trans* directly on neat fats and oils would be of substantial benefit.

The McGill FTIR Group has been carrying out research on the development of simple, rapid, and accurate methods of edible oil analysis, based on Fourier transform infrared (FTIR) spectroscopy (3–10). One of the practical developments of this research has been an FTIR-based edible oil analysis package, which is capable of simultaneously determining IV, saponification number, and *cis* and *trans* content (IV/SN/*cis*/*trans*) in a single analysis on a neat fat or oil in less than 2 min. The system is preprogrammed and precalibrated and has a heated sample accessory (8) to allow both fats and oils to be analyzed in their liquid state at 80°C; a commercial version is marketed by Nicolet Instrument (Madison, WI). This system is in use by a number of edible oil processors who are satisfied with its performance; however, it has been noted, through our own as well as the users' experience with the system, that, although the *trans* and IV predictions are linearly related to their own reference analyses, the *trans* values tend to be overestimated. This is readily corrected for by linear regression; however, because the original concept for the development of the method is to be independent of recalibrations or adjustment, we felt that this problem should be addressed by a structured validation study. This study, carried out in cooperation with a major European edi-

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ble oil processor, compares the *trans* and IV data obtained from the FTIR edible oil analysis package with the *trans* data obtained from a proposed modification of the standard AOCS IR method (11) as well as those obtained by gas chromatography (GC) analyses (12).

MATERIALS AND METHODS

Analytical system. The instrument used was a Nicolet Magna 510 FTIR spectrometer, equipped with a heated sample-handling accessory, manufactured by Dwight Analytical (Toronto, Ontario, Canada), and a 25-μm KCl flow cell heated to 80°C. The instrument was controlled by a 486 PC, running under Omnic 2.1 software and preprogrammed with Microsoft Visual Basic to carry out IV/SN/*cis*/*trans* analyses by using previously developed calibrations based on pure triglyceride standards and derived by application of the partial-least-squares (PLS) regression technique (3,8). A calibration equation for the determination of *trans* content from a modified version of the AOCS IR method (Cd14-61) (12) was derived and appended to the PLS analytical package to produce analytical values concurrently from the same spectra. For calibration of the modified AOCS method, five calibration standards (0–50% trielaidin) were prepared by addition of trielaidin (Sigma Chemical Co., St. Louis, MO) to an unhydrogenated soybean oil with a *trans* content of <0.1% (as determined by GC). Calibration was performed by recording the spectra of these standards in the same sample-handling accessory, flow cell, and temperature conditions as described above, and ratioing the spectrum of each standard against that of the base oil. The concentration of trielaidin in these standards was related to the peak height measured in the differential spectra at 966 cm^{-1}, corresponding to the position of the peak maximum of trielaidin in the spectra of the calibration standards, relative to a baseline drawn between 995 and 937 cm^{-1}. The spectra of all samples analyzed by the modified AOCS method were ratioed against the same base oil spectrum as the calibration standards.

Samples/analyses. One hundred and eight samples of hydrogenated low-erucic rapeseed ($n = 36$) and soybean ($n = 71$) oils were obtained from Vandemoortele (Izegem, Belgium) for analysis by FTIR spectroscopy. For all FTIR analyses, the samples were prewarmed in a microwave oven to ~80°C and aspirated through the IR cell. All samples supplied were analyzed for IV and *cis* and *trans* content by the FTIR edible oil analysis package (6) as well as for *trans* content by the modified AOCS peak height method. The data output was stored in a spreadsheet format to a text file for subsequent statistical analysis. Twenty of the samples were run in duplicate to determine repeatability (back-to-back duplicates) and were also reanalyzed 4 wk later to assess week-to-week reproducibility of the FTIR method. *Cis* and *trans* data were expressed as % triolein and % trielaidin, respectively, and internal analytical consistency was determined by relating total unsaturation [$\sum$(*cis* + *trans*)] to IV. Approximately one-third of the samples supplied were independently analyzed by P. Maes at the Vandemoortele laboratory in Belgium for *trans* content by the AOCS gas-chromatographic procedure (Method Ce 1c-89) (12), and IV values were determined from the GC data by the Calculated Iodine Value method (Cd 1c-85) (12). A Hewlett-Packard GC (Palo Alto, CA), equipped with a flame-ionization detector and capillary injection system, was operated isothermally (192°C) with a 20 meter SP-2340 (Supelco Inc., Bellefont, PA) column and the injection port and detector set at 250°C. Helium was used as the carrier gas at a flow rate of 15 cm/s and a column head pressure of 125 kPa. Methyl esters of the oil samples were prepared by the AOCS (Ce 2-66) boron trifluoride method, and ~0.5–1 μL injected for quantitative analysis; the percentage *trans* content was determined by relating the integrated *trans* fatty acid peak areas to that of the total area obtained for all fatty acids eluted. The IV and *trans* GC validation data, obtained at the Vandemoortele laboratory, were compared to the corresponding FTIR results by linear regression and Z-linear regression (data forced through the origin), as well as in terms of mean difference (MD) and standard deviation of the differences (SDD) (13).

RESULTS AND DISCUSSION

IR determination of trans *content.* IR spectroscopy for the determination of the *trans* content of fats and oils is a well-established AOCS method (12). This method is based on the measurement of the peak height of a characteristic absorption band of isolated *trans* bonds at 10.3 μ (970 cm^{-1}). However, the procedure has a number of well-known drawbacks, such as the use of volatile and noxious/toxic CS_2, and suffers from interferences of overlapping triglyceride absorptions, which cause the *trans* values to be overestimated (19). To circumvent the latter problem, the AOCS method requires that samples with <15% *trans* be saponified and the fatty acids converted to their methyl esters prior to analysis. In recent years, as a result of the development of FTIR instrumentation and computerized data analysis techniques, various modifications to the AOCS method for the IR determination of *trans* content have been investigated. Several of these (11,14,15) measure the *trans* content from the IR spectrum of neat oil (or melted fat) or methyl esters recorded in a short-pathlength (~0.1 mm) transmission cell or by the attenuated total reflectance (ATR) sampling technique, thereby eliminating the use of CS_2. Because of the inconvenience of injecting samples into short-pathlength cells and the drawbacks of the ATR sampling method, particularly the difficulty of cleaning the ATR crystal and the extreme sensitivity of ATR/FTIR measurements to changes in the alignment of the crystal and temperature fluctuations, the McGill IR Group developed a *trans* method based on the use of a heated flow-through transmission cell sample-handling accessory, designed specifically for the analysis of neat fats and oils (8). Another modification to the AOCS method recently suggested (11,16,17,20) is to remove the spectral contributions of the overlapping absorptions by ratioing the single-beam FTIR spectrum of the fat or oil being analyzed against the single-beam spectrum of a sim-

ilar reference oil that is free of *trans* groups. This approach allows the isolated *trans* content to be obtained unambiguously as per the AOCS peak height calculation and avoids the use of CS_2 as well as the saponification and methylation procedures required for low *trans* samples, leading to a substantial simplification of the method. This procedure, applicable to quantitation of isolated *trans* isomers at levels equal to or greater than 1%, has been proposed for adoption by the AOCS as Recommended Practice CD14b-95 (16).

An alternative approach to the problem of underlying absorptions was employed in our previous development of a *trans* method (8). By employing PLS, a form of factor analysis, we were able to develop a calibration model for the prediction of *trans* content that, in principle, accounts for the overlapping absorptions that contribute to the intensity of the *trans* absorption band. The PLS approach has the additional benefit of allowing the determination of IV and *cis* content in addition to *trans* content, as well as SN, and has been developed as an integrated analytical package. PLS is effectively a "whole-spectrum" approach, which relates spectral information from broad regions of the spectrum to the compositional variables of interest, as opposed to being based on a single-frequency measurement. Thus, it is a powerful multivariate analysis technique that is capable of accurately measuring components in the presence of interfering absorptions while providing greater accuracy than can be achieved with single-frequency measurements (18). The modified AOCS method described above and the PLS approach both attempt to compensate for overlapping absorptions but in distinctly different ways, and accordingly, it was of interest to examine their relative concurrence by comparing the predictions obtained from these two methods for a common set of samples.

Figure 1A shows the *trans* absorption region (995–937 cm^{-1}) in the overlaid spectra of the standards employed to derive the calibration equation for the modified AOCS method, prepared by adding varying amounts of trielaidin to a *trans*-free base oil. The corresponding spectra obtained by ratioing the single-beam spectra of these calibration standards against the single-beam spectrum of the base oil are presented in Figure 1B, which illustrates the horizontal baseline produced by the ratioing procedure, which eliminates some of the uncertainty in the measurement of the *trans* peak height. The standard curves derived from the spectra shown in Figures 1A and B are presented in Equations 1 and 2, respectively:

$$\%\ trans = -3.917 + 131.276\ A(966) \quad [1]$$
$$R = 0.999 \qquad SE = 0.430$$

$$\%\ trans = -0.230 + 130.973\ A(966) \quad [2]$$
$$R = 0.999 \qquad SE = 0.398$$

where % *trans* = *trans* content expressed as % trielaidin, and A(966) = absorbance @ 966 cm^{-1} relative to a baseline drawn between 995 and 937 cm^{-1}.

Equation 1 shows that the underlying triglyceride absorption in the spectrum of the *trans*-free oil corresponds to an intercept value of 4% *trans,* whereas the intercept is eliminated from the calibration equation by the ratioing procedure. Equation 2 was employed to predict the *trans* contents of 108 samples of hydrogenated rapeseed and soybean oils after ratioing their spectra against that of the same base oil used in preparing the calibration standards. The *trans* values of the samples were also predicted with the PLS calibration model incorporated in the FTIR edible oil analysis software package. Figure 2 presents a comparison of the % *trans* values predicted by the modified AOCS method and the PLS method for the 108 samples. The linear regression equation obtained for Figure 2 was:

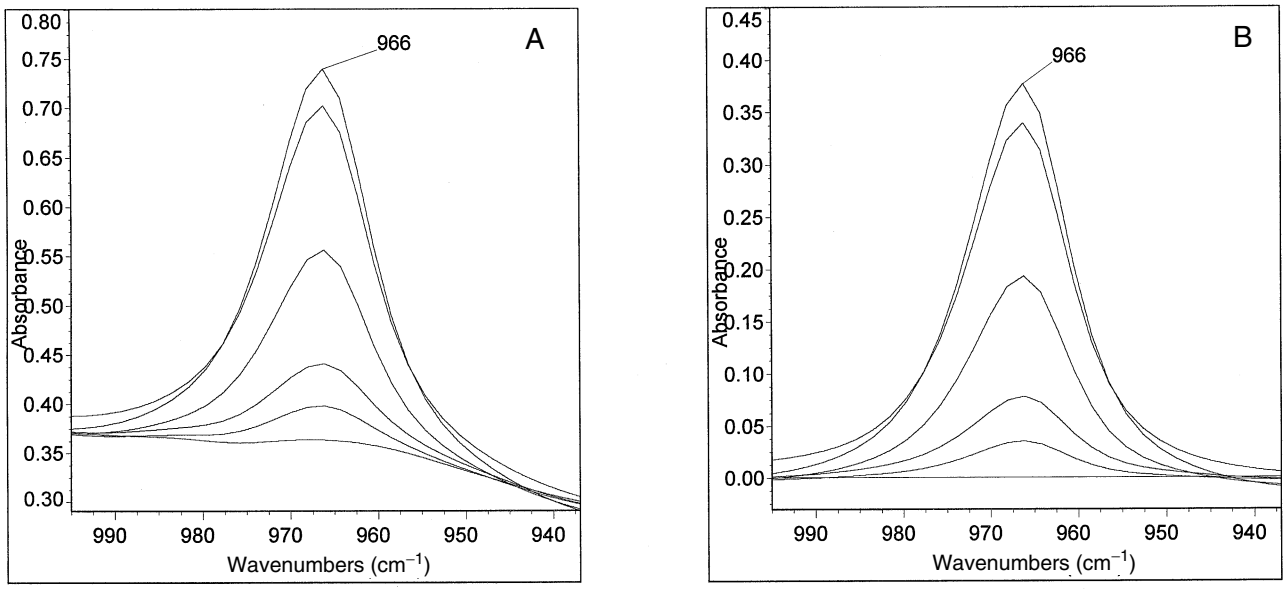

FIG. 1. (A) Overlaid spectra in the *trans* absorption region of calibration standards prepared by addition of various amounts of trielaidin to a *trans*-free base oil; (B) same spectra as in (A) after ratioing out the spectrum of the base oil.

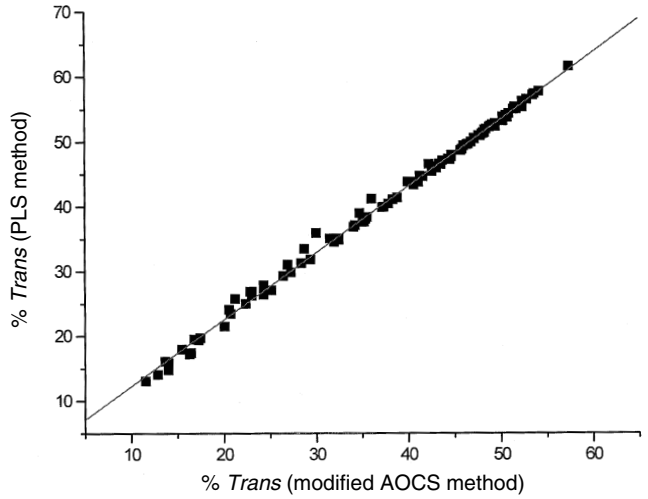

FIG. 2. Plot of % *trans* values predicted by the partial-least-squares (PLS) calibration for hydrogenated rapeseed and soybean oils vs. the % *trans* values obtained by the modified AOCS method.

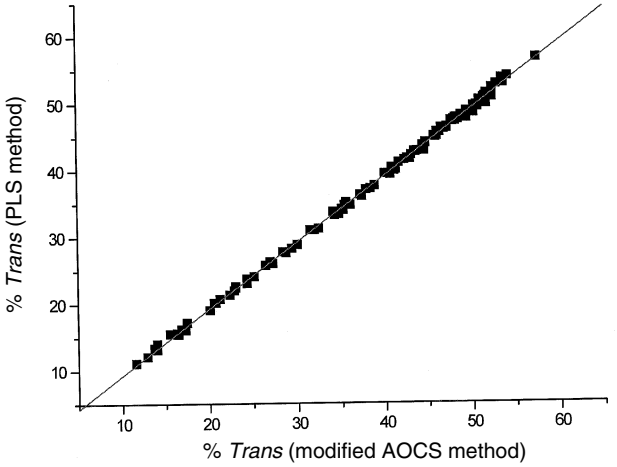

FIG. 3. Plot of % *trans* values predicted by the refined PLS calibration for hydrogenated rapeseed and soybean oils vs. the % *trans* values obtained by the modified AOCS method. See Figure 2 for abbreviation.

$$\%\ trans\ (\text{PLS}) = 1.930 + 1.029\ \%\ trans\ (\text{AOCS}) \qquad [3]$$
$$\text{SE} = 0.692 \qquad \text{R} = 0.998$$

This equation indicates that the % *trans* values are overestimated by the PLS method as compared to the modified AOCS method. The mean difference (MD) between the two sets of predictions revealed a bias of 3.05 *trans* units, with a standard deviation of the differences (SDD) of 0.78. Furthermore, examination of the plot in Figure 2 indicates that several of the PLS-derived % *trans* values are substantially higher (5–6% *trans*) than the values obtained from the modified AOCS method. Because the predictions being compared are based on a common set of spectra, experimental sources of error, such as instrumental noise or sampling and temperature variations, cannot account for the observed discrepancies. The general overestimation of *trans* content by the PLS method was subsequently confirmed by analyzing the gravimetrically prepared trielaidin standards used to calibrate the modified AOCS method. The results obtained confirmed our hypothesis that the PLS method tends to predict somewhat higher *trans* values and led to a detailed reexamination of the PLS calibration model. We found that, by excluding trilinolelaidin ($C_{18:2tt}$) from the calibration set and basing the calibration on second-derivative spectra to decrease the sensitivity of the calibration model to baseline fluctuations, the PLS predictions could be reconciled with both the known concentrations of the gravimetrically prepared standards and the modified AOCS predictions for the samples; the latter results are shown in Figure 3, which yields the following linear regression equation:

$$\%\ trans\ (\text{PLS}) = -0.151 + 0.988\ \%\ trans\ (\text{AOCS}) \qquad [4]$$
$$\text{SE} = 0.353 \qquad \text{R} = 0.999$$

Comparison of Equations 3 and 4 and the corresponding SE reveals that the refined PLS calibration model provides much better concurrence with the modified AOCS method, as also demonstrated by reduction of the MD between the two sets of predicted values to –0.61 *trans* units. In addition, the fact that good agreement could be obtained between the PLS and modified AOCS methods indicates that both methods successfully compensate for the overlapping absorptions that cause interferences in the traditional AOCS method.

Internal consistency of the data. A key element in assessing the FTIR–PLS approach is to ensure that the *cis* and *trans* data are internally consistent with the IV data because the two measures are related. Because the *cis* and *trans* data provided by the FTIR edible oil analysis package are expressed in terms of % triolein and % trielaidin, the IV is related to the sum of the *cis* and *trans* double-bond contributions in the following manner:

$$\text{IV} = 3[(\text{C} + \text{T})\ (\text{MW}\ I_2/\text{MW TG})] \qquad [5]$$

$$\text{IV} = 0.8601(\text{C} + \text{T}) \qquad [6]$$

where IV = iodine value, C = % *cis* (as triolein), T = % *trans* (as trielaidin), MW TG = molecular weight of triolein or trielaidin (885.40), and MW I_2 = molecular weight of molecular iodine (253.81).

Based on this relationship, one would expect the FTIR-predicted *cis* + *trans* to be linearly related to the FTIR-predicted IV, and a plot of 0.8601(*cis* + *trans*) vs. IV to produce a slope of 1.0 and an intercept of 0.0. Figure 4 presents a plot of predicted FTIR IV for the 108 rapeseed and soybean samples vs. the sum of predicted *cis* and predicted *trans,* multiplied by the theoretical slope factor of 0.8601. The linear regression equation obtained for Figure 4 was:

$$\text{IV} = 0.0119 + 1.0002\ \text{CT} \qquad [7]$$
$$\text{SE} = 0.069 \qquad \text{R} = 1.0000$$

where IV = iodine value, and CT = 0.8601(*cis* + *trans*).

Elimination of the intercept by means of a regression that forces the data through the origin produced a slope of 0.998,

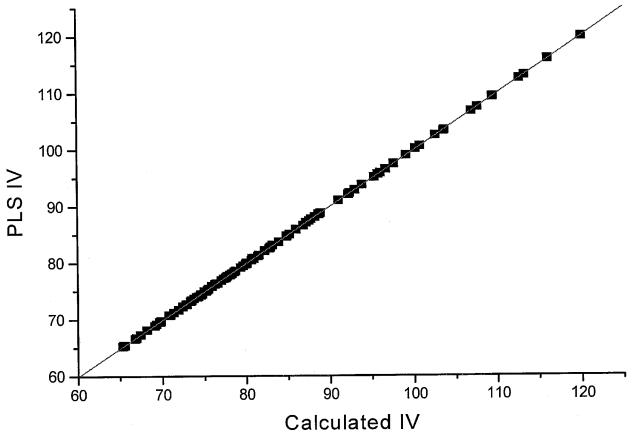

FIG. 4. Plot of Fourier transform infrared (FTIR)-PLS-predicted iodine value (IV) for hydrogenated rapeseed and soybean oils vs. the IV calculated from the FTIR-PLS-predicted *cis* and *trans* values, multiplied by the slope factor of 0.8601, determined from Equation 6. See Figure 2 for other abbreviation.

within 0.2% of the theoretical slope of unity. Multiple regression (forced through the origin) of the IV data vs. the *% cis* and *% trans* data produced coefficients of 0.858 and 0.859, respectively, close to the ideal value of 0.8601 expected from Equation 6. These results provide strong evidence that the IV and *cis*/*trans* predictions obtained with the FTIR analytical package are internally consistent, in that they reflect the theoretical interrelationships one expects to observe among the three parameters. Furthermore, the relationship between IV and *cis*/*trans* predictions was consistent for both rapeseed and soy samples, demonstrating that the FTIR–PLS results are oil-independent.

Repeatability and reproducibility of FTIR data. The repeatability of the FTIR data was assessed by comparing the IV, SN, *cis* and *trans* predictions obtained from the edible oil analysis package for 20 samples from duplicate spectra of each sample, recorded consecutively. The results for each of the four measures are presented in Table 1, in terms of the mean difference for repeatability (MD_r) and the standard deviation of the differences for repeatability (SDD_r), the standard deviation obtained from linear regression of the duplicate predictions, and the corresponding coefficient of variation. The repeatability data indicate no significant biases between duplicates, the ideal value for the MD_r being zero. The SDD_r, which represents the relative variability around the mean difference, is quite similar to the SD obtained by linear regression. The coefficient of variation (CV), which expresses overall variability in percent terms relative to the overall mean value of the samples, is less than 0.3% for all components.

After 4 wk, over which the instrument was configured for other analyses which required removal of the cell, the FTIR system was restandardized, and 20 randomly selected samples were run to determine the reproducibility of the predictions. Table 2 presents the MD_r and SDD_r that were calculated by comparing the predictions originally obtained to those obtained 4 wk later. The reproducibility results indicate that there is no bias per se in the data; however, as expected, there is a small increase in the variability around the MD, reflected by a general increase in the CV. However, the reproducibility is well within 1% for all parameters measured.

Validation data. Of the global set of samples analyzed by FTIR, about one-third were selected for validation analysis by capillary GC (IV and *trans*). The GC data obtained are compared in Table 3 to the corresponding FTIR-predicted values for these samples (i.e., IV and *% trans* from the PLS-based FTIR edible oil analysis package and *% trans* from the modified AOCS method). Figures 5 and 6 present plots of the FTIR–PLS IV vs. the GC IV data and the FTIR–PLS *trans* vs. the GC *trans* data, respectively. Table 4 presents a summary of the linear and Z-linear regression equations derived from these plots and the associated statistics as well as equations that relate the modified AOCS IR *trans* data to the GC *trans* data.

The conventional linear regression equation for IV, given in Table 4, indicates that there is a combined bias and slope contribution that relates the two variables; however, the Z-regression results demonstrate that, in fact, the agreement is excellent, because the intercept can be incorporated into the slope without any appreciable increase in the regression error. This is confirmed by the MD of 0.004 IV between the two sets of data. On the other hand, the GC *trans* data, when related to the FTIR *trans* data, whether obtained by the PLS or the modified AOCS method, produced a slope factor of 0.88 rather than a 1:1 correspondence. Accordingly, there is an increasing discrepancy between the IR and GC data as the *trans* content of the samples increases. Similar discrepancies between IR and GC *trans* determinations have been observed by others (19) and have been attributed to unsatisfactory separation of 18:1*t* and 18:1*c* isomers on the currently available GC sta-

TABLE 1
FTIR Predictions for IV, SN, and *cis* and *trans* Content: Repeatability for Duplicate Samples (*n* = 20) Run Consecutively[a]

Measure	IV	SN	% *Cis*	% *Trans*
Mean	87.74	193.18	61.7	40.15
MD_r	0.048	0.012	0.068	–0.011
SDD_r	0.108	0.109	0.140	0.058
Lin. Reg. SD	0.110	0.104	0.139	0.056
CV	0.13%	0.05%	0.23%	0.14%

[a]FTIR, Fourier transform infrared; IV, iodine value; SN, saponification number; MD_r, mean difference for repeatability; SDD_r, standard deviation of the differences for repeatability; Lin. Reg. SD, linear regression standard deviation; CV, coefficient of variation.

TABLE 2
FTIR Predictions for IV, SN, and *cis* and *trans* Content: Reproducibility Data for Samples Run Four Weeks Apart[a]

Measure	IV	SN	% *Cis*	% *Trans*
MD_r	–0.179	0.437	–0.057	–0.074
SDD_r	0.502	0.269	0.484	0.221
CV	0.57%	0.14%	0.78%	0.55%

[a]See Table 1 for abbreviations.

TABLE 3
Validation *trans* and IV Data Compared to the Corresponding FTIR Results for 31 Randomly Selected Samples

	GC		FTIR		
Sample	GC IV	GC *trans*	PLS IV	PLS *trans*	AOCS *trans*[a]
R2	80.4	38.06	79.76	42.70	43.34
R3	92.2	20.73	92.06	22.58	22.73
R6	86	27.83	84.62	30.97	31.40
R8	100.3	13.06	100.63	13.43	13.54
R10	81.4	41.44	80.74	45.60	45.97
R11	89.5	21.61	88.58	23.73	24.04
R12	89.9	22.03	88.53	23.32	24.04
R13	74.1	48.02	72.27	53.58	53.32
R14	91.24	20.79	91.02	22.38	22.86
R20	98.69	14.13	98.99	15.53	15.25
R28	92.2	18.2	92.32	20.13	20.37
R31	75.8	45.64	76.27	50.72	51.35
R33	96.6	15.56	97.50	17.16	17.09
R34	78.4	45.05	78.14	49.50	50.17
S2	113.9	11.15	115.93	12.15	12.49
S5	96.8	25.49	95.86	27.61	28.11
S6	117.4	10.4	119.89	11.15	11.18
S8	95.4	21.95	96.51	24.06	24.70
S19	65.5	40.16	65.41	47.22	47.67
S25	94.4	21.29	95.11	23.08	24.04
S27	82.2	29.22	80.87	33.20	34.02
S29	103.5	19.99	103.43	21.99	22.21
S35	79.6	36.6	79.50	41.57	42.55
S36	85.1	27.41	84.72	30.95	31.66
S44	72.6	42.84	72.32	48.73	49.38
S47	68.7	39.8	68.14	46.03	46.76
S50	66.8	41.31	66.78	47.68	48.07
S57	76.5	37.32	76.92	42.75	43.47
S59	89.7	28.35	88.79	31.19	32.05
S68	87.5	31.49	87.09	34.71	35.33
S71	111	15.14	112.50	16.24	16.30
MD[b]	—	—	0.004	–3.212	–3.659

[a]Modified AOCS method.
[b]Mean difference with respect to the corresponding gas chromatography (GC) data. See Table 1 for other abbreviations.

tionary phases. Given the relative complexity and well-known limitations associated with the GC method, the fact that independently obtained IR and GC *trans* data do track each other well provides additional, indirect evidence for the efficacy of the IR *trans* approaches.

The initial results obtained in this validation study confirmed that the *trans* predictions obtained from the edible oil analysis package were somewhat higher than those produced by peak height-based IR methods. Refinement of the PLS calibration model corrected this problem and produced good agreement with the results obtained from a modified AOCS method assessed concurrently, the latter having the benefit of a simple and accurate gravimetric preparation procedure. This agreement between the two methods indicates that both methods allow for the accurate determination of *trans* content from neat fats and oils without the need for their conversion to methyl esters, with the PLS method having the advantage that it does not require the availability of a *trans*-free base oil. In addition, the PLS method provides IV results that match GC IV data as well as *cis* and SN data. The agreement of total unsaturation (*cis* + *trans*) obtained by GC with that obtained by IR lends credence to the efficacy of the GC procedure in general, its limitation being the accurate partition of the *cis* and *trans* components due to peak overlap. The concurrence of the *trans* and IV data obtained from the PLS-based edible oil analysis package with the *trans* values obtained from the modified AOCS method, and with GC IV data, as well as the internal consistency of its IV, *cis* and *trans* predictions, provide strong experimental evidence that it measures all three variables accurately. This precalibrated and preprogrammed analytical package, carrying out four separate analyses simultaneously (IV/SN/*trans*/*cis*) in a total analysis time of less than 2 min, should be useful for general quality control use in the edible oil industry.

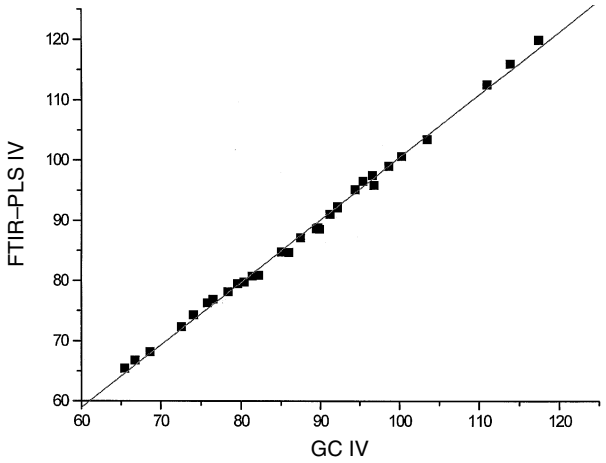

FIG. 5. Plot of FTIR–PLS-predicted IV for 31 hydrogenated rapeseed and soybean oils vs. IV calculated from gas chromatography (GC) data. See Figure 4 for other abbreviations.

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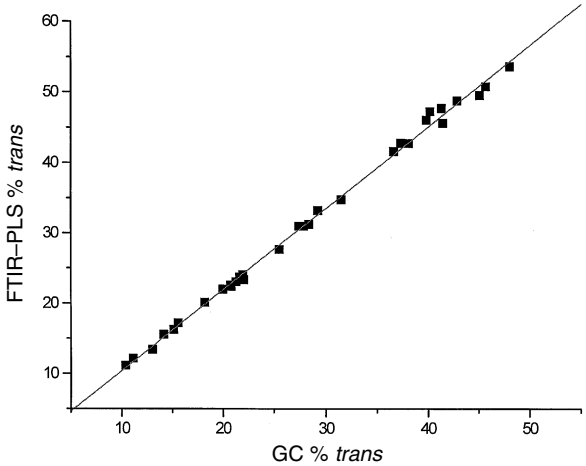

FIG. 6. Plot of FTIR–PLS-predicted % *trans* for 31 hydrogenated rapeseed and soybean oils vs. % *trans* obtained by GC. See Figures 4 and 5 for abbreviations.

TABLE 4
Regression Analysis Summary for Figures 5 and 6 Plus the Regression of the Modified AOCS IR *trans* Data vs. the GC *trans* Data[a]

Regression	Abscissa	Ordinate	Intercept	Slope	SD	R
Normal	GC IV	PLS IV	–3.405	1.0386	±0.783	0.998
Z-Regression	GC IV	PLS IV	0.0000	1.0008	±0.921	0.999
Normal	GC *trans*	PLS *trans*	–1.146	1.1552	±0.736	0.998
Z-Regression	GC *trans*	PLS *trans*	0.0000	1.1199	±0.843	0.999
Normal	GC *trans*	AOCS *trans*	–1.019	1.1663	±0.805	0.998
Z-Regression	GC *trans*	AOCS *trans*	0.0000	1.1350	±0.880	0.999

[a]IR, infrared; PLS, partial least squares. See Figures 1 and 3 for other abbreviations.

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