

Trans Determination of Edible Oils by Fourier Transform Near-Infrared Spectroscopy

Hui Li^a, F.R. van de Voort^{a,*}, A.A. Ismail^a, J. Sedman^a, and R. Cox^b

^aMcGill IR Group, Department of Food Science and Agricultural Chemistry, Macdonald Campus of McGill University, Saint Anne de Bellevue, Québec, Canada H9X 3V9, and ^bBomem Inc., Québec, Canada

ABSTRACT: A generalized partial-least-squares calibration for determination of the *trans* content of edible fats and oils by Fourier transform near-infrared (FT-NIR) spectroscopy using 8-mm disposable glass vials for sample handling and measurement was developed. The *trans* contents of a broad range of oils were determined using the American Oil Chemists' Society single-bounce horizontal attenuated total reflectance (SB-HATR) mid-infrared spectroscopic procedure, these *trans* reference data were used in the development of the generalized FT-NIR calibration. Additional refined and product-specific calibrations were also developed, and all the calibrations were assessed for their predictive capabilities using two sets of validation samples, one comprising a broad range of oil types and the other restricted to oils with specific characteristics. The FT-NIR *trans* predictions obtained using the generalized calibration were in good agreement with the SB-HATR results; the values were accurate and reproducible to within ± 1.1 and $\pm 0.5\%$ *trans*, respectively, compared to a reproducibility of $\pm 0.40\%$ *trans* obtained for the SB-HATR method. The accuracy of the predictions obtained from the generalized FT-NIR calibration for particular oil types was not significantly improved by supplementing the base training set with samples of these specific types. Calibrating only these oil types did, however, produce a substantial improvement in predictive accuracy, approaching that of the SB-HATR method. These product specific calibrations produced serious predictive errors when nonrepresentative samples were analyzed. The incorporation of a supplementary discriminate analysis routine was found to be a powerful safeguard in flagging nonrepresentative samples as outliers and could also be used to select the calibration most appropriate for the characteristics of the sample being analyzed. Overall, it was concluded that FT-NIR spectroscopy provides a viable alternative to the SB-HATR/mid-Fourier transform infrared method for *trans* determination, making use of more industrially robust instrumentation and equipped with a simpler sample handling system.

Paper no. J9518 in *JAOCs* 77, 1061–1067 (October 2000).

KEY WORDS: Edible oil analysis, Fourier transform near-infrared spectroscopy, FT-NIR, partial least squares, PLS, *trans* content.

*To whom correspondence should be addressed at McGill IR Group, Department of Food Science and Agricultural Chemistry, Macdonald Campus of McGill University, Box 187, Saint Anne de Bellevue, Quebec, Canada, H9X 3V9. E-mail: vandervoort@macdonald.mcgill.ca

Trans fatty acids (TFA) in edible fats and oils have been implicated in the etiology of arteriosclerosis and heart disease (1,2), and the U.S. Food and Drug Administration (FDA) has recently published a proposed amendment of their regulations on nutritional labeling (3). This will require that the amount of TFA in foods, as well as dietary supplements be included in the amount and percent daily value declared for saturated fatty acids. TFA are commonly produced from unsaturated fatty acids during hydrogenation in a concurrent side reaction, in which some of the *cis* double bonds are incompletely hydrogenated and converted to the *trans* configuration instead. The degree of hydrogenation, as well as the levels of TFA formation, are key determinants of the quality and functionality of fats and oils, and monitoring TFA formation is a useful way of tracking and controlling the hydrogenation process.

Mid-infrared (IR) spectroscopy has been the predominant means of determining the *trans* content of fats and oils since the 1940s (4). The advent of Fourier transform infrared (FT-IR) spectroscopy has led to substantive improvements in compensating for interfering triglyceride absorptions, eliminating the need to saponify and methylate the oils to produce fatty acid methyl esters (5). A related infrared spectroscopic technique, near-infrared (NIR) spectroscopy, is widely used in the food industry for quality control applications, being a rapid, reliable, and nondestructive means of analyzing various materials without reagents or solvents. NIR instruments are more rugged and less energy-limited than mid-IR spectrometers, and offer more flexible sample handling options, which make it possible to analyze samples in low-cost, convenient glass vials. These advantages are also inherent to FT-NIR spectrometers, with the added benefit of highly repeatable measurements owing to the use of a helium-neon reference laser, which ensures wavelength accuracy. Wavelength accuracy is the key to obtaining robust calibrations and facilitates the transfer of calibrations from one instrument to another, generally a problematic process with traditional grating or filter-based instruments.

The McGill IR Group has worked extensively on the development of rapid FT-IR methods for the analysis of edible oils, with a focus on mid-FT-IR spectroscopy. One of the first *trans* analysis methods developed was part of a mid-FT-IR edible oil analysis package that simultaneously allowed the

determination of *cis* and *trans* content as well as iodine value (IV) and saponification number (SN), using partial-least-squares (PLS) calibrations based on pure triglyceride standards (6). Subsequently, it was shown that an FT-NIR spectrometer, equipped with a 5-mm quartz transmission cell could be used to accurately determine *cis/trans*/IV/SN when calibrated using mid-FT-IR spectroscopic data (7). Bomem, an FT-IR instrument manufacturer, developed a "global" FT-NIR IV calibration for 8-mm glass vials based on a broad variety of oil samples; the reference values for this calibration are provided by gas chromatography (GC) (8). Subsequently, more accurate, oil-specific IV calibrations were developed and employed in conjunction with discriminate analysis to differentiate between closely related oil types using discrimination criteria based on various types of information provided by these calibrations (9). This work led to the conclusion that a generalized, instrument-transferable, FT-NIR IV calibration would provide sound IV analytical data, and that the calibration could be further refined with processor-specific oils if additional accuracy was needed. An FT-NIR IV method, based on the use of a global calibration, is being considered by the American Oil Chemists' Society (AOCS) for official method status.

This paper expands on the concepts developed as a result of the IV work. The objective of this research was to develop a generalized *trans* calibration for an FT-NIR spectrometer, equipped with a heated sample-handling accessory that uses 8-mm disposable vials. The overall goal is to provide processors with a rapid and simple means of determining *trans* content with instrumentation more suited to the industrial quality control environment.

MATERIALS AND METHODS

Oil samples, calibration and validation standards. Oil samples representing a broad range of oil types and degrees of hydrogenation were obtained from California Oils Co. (Richmond, CA), Bunge Foods (Bradley, IL) and Lipton (Baltimore, MD). The reference *trans* values (expressed as % trielaidin) for all the California/Bunge group (CBG) oils were obtained using the AOCS single-bounce horizontal attenuated total reflection mid-IR method (5). For the calibration of the reference method, triolein and trielaidin were obtained from Nu-Chek-Prep, Inc. (Elysian, MN) and an ultra-degummed, bleached, expelled soybean reference oil was obtained from Owensboro Grain Co. (Owensboro, KY). In the case of the Lipton group (LG) samples, *trans* data obtained by the new AOCS GC method (5) were provided by the processor.

The training set for the generalized *trans* calibration consisted of 44 CBG oil samples, which included, among others, undefined commercial shortenings and palm, corn, almond, soybean, canola, and walnut oils, both hydrogenated and non-hydrogenated. Figure 1 provides a frequency histogram of the *trans* content distribution in the training set, which ranged from ~0 to 50% *trans*. The LG samples consisted of four sets of 40 preanalyzed but undeclared oil types, only identified by

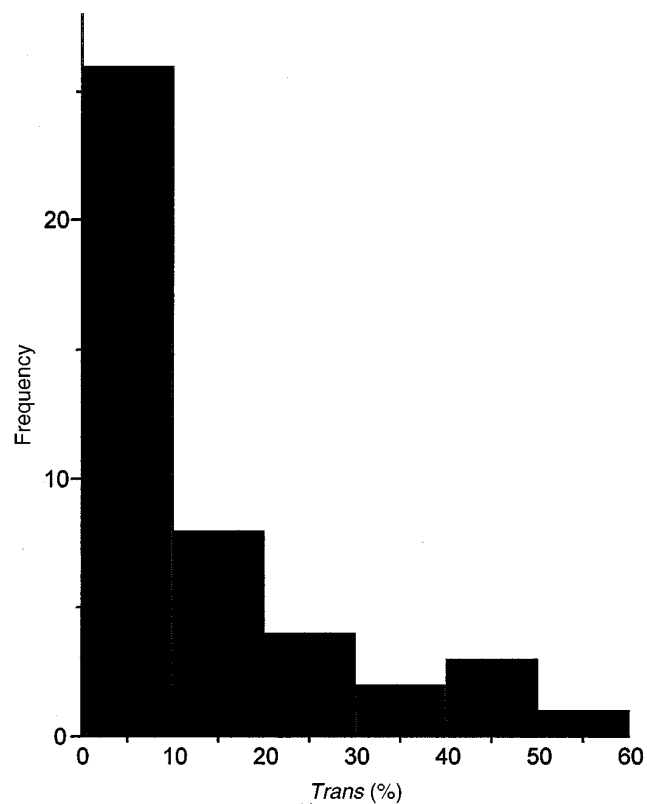


FIG. 1. A frequency distribution plot illustrating the *trans* values of the samples in the training set used to derive the generalized *trans* calibration (Cal-1).

being grouped as A–D (Table 1). A second *trans* calibration set was prepared by adding 20 LG samples, five of each type, to the 44-sample base training set used for the general calibration. Additional calibrations were developed for each specific LG oil type (A–D) by using 20 samples of each group to develop individual group calibration training sets, as well as a combined LG calibration training set consisting of five samples from each oil type. The makeup of the various calibration training sets is summarized in Table 2. Two sets of validation samples were used to evaluate all the calibrations. None of these validation samples were used in any of the calibration training sets. The first validation set (Val-1) consisted of 21 samples selected from the CBG oil group, while the second set (Val-2) consisted of 20 LG samples obtained from oil groups A–D (five from each).

TABLE 1
Mean *trans* Values, Standard Deviations and Ranges
for Oil Samples Within Lipton Oil Groups A–D

Oil type	<i>Trans</i> (%)		
	Mean	SD	Range
A	0.567	0.099	0.33–0.71
B	26.921	1.207	25.20–28.81
C	8.893	0.714	7.80–10.17
D	8.898	0.656	7.95–10.12

TABLE 2
Summary of the Sample Makeup of the Training Sets
Used for the Various *trans* Calibrations

Calibration	CBG ^a oils	LG oils
1	44 samples	0 samples
2	44 samples	20 samples (5 each of oil types A, B, C, and D)
3	0 samples	20 samples (5 each of oil types A, B, C, and D)
4a	0 samples	20 samples of oil type A
4b	0 samples	20 samples of oil type B
4c	0 samples	20 samples of oil type C
4d	0 samples	20 samples of oil type D

^aCBG, California/Bunge Group; LG, Lipton Group.

Instrumentation and sample handling. A Bomem (Bomem Inc., Québec, Canada) MB155S FT-IR spectrometer, configured for NIR operation and equipped with a deuterated triglycine sulfate (DTGS) detector, was used for this study. The spectrometer was controlled by an IBM-compatible 486 DX 66-MHz PC running under Windows-based Bomem-Grams/386 software (Galactic Industries Co., Salem, NH) and AIRS, a specific quality assurance software package (DHC Analysis, Cleveland, OH). A Nicolet (Madison, WI) Magna FT-IR spectrometer, operating under Nicolet OMNIC E.S.P. software and equipped with a heated ZnSe SB-HATR accessory (Graseby-Specac, Smyrna, GA), was employed for the determination of the reference *trans* values by AOCS Official Method Cd 14d-97 (5).

For acquisition of FT-NIR spectra, samples were transferred to 8-mm o.d. transparent glass vials (Kimble Glass Inc., Vineland, NJ) having a nominal volume of 1 mL. The vials were placed in a temperature-controlled, multivial sample holding block (Bomem Inc.) maintained at $68 \pm 1^\circ\text{C}$. All sample and background spectra were recorded by co-adding 128 scans at a resolution of 16 cm^{-1} over the spectral range of $10,000\text{--}4,000\text{ cm}^{-1}$. Open-beam background spectra were collected every 30 min with the vial holder in the IR beam, and each sample spectrum was ratioed against the most recent background spectrum collected. To compensate for inherent variations in vial pathlength, the ratioed spectra were normalized to unit area over the region of $9,100\text{--}7,560\text{ cm}^{-1}$ by using a normalization routine provided with the AIRS software.

NIR calibration and validation. The spectra of the *trans* calibration standards along with their respective reference *trans* values were input into the Galactic PLSplus chemometrics program (Galactic Industries Co.) to develop PLS calibrations. Correlation and variance spectra were generated to identify spectral regions that contain information related to variations in *trans* content, and these regions were then explored for calibration development and refinement. Each calibration was assessed by using the leave-one-out cross-validation procedure and optimized in terms of the appropriate number of factors by minimizing the predicted residual error sum of squares (PRESS). The performance of the calibrations, as well as their respective cross-validations, was assessed using linear regression, with accuracy (a) and repro-

ducibility (r) calculated as mean differences (MD) and standard deviations of the differences (SDD) for r or a according to the method of Youden (10).

RESULTS AND DISCUSSION

Single-bounce horizontal attenuated total reflectance (SB-HATR) results. The calibration of any secondary method, such as an FT-NIR *trans* method, is strongly dependent on the a and r of the reference method upon which it is based. The new AOCS SB-HATR mid-FT-IR *trans* method (5) was used to obtain the reference *trans* values for the CBG oil samples. The SB-HATR method itself requires calibration based on the use of two-component gravimetrically prepared mixtures of trielaidin and triolein. This method entails ratioing the spectra of these standards, as well as those of the samples subsequently analyzed, against the spectrum of a zero-*trans* reference oil, with quantitation being based on measurement of the area under the isolated *trans* absorption band between 990 and 945 cm^{-1} in the ratioed spectra. The calibration equation obtained by linear regression of the mean area from duplicate spectra of the calibration standards vs. the gravimetric *trans* content was:

$$\text{Area} = 0.37008 + 0.38121(\% \text{ trans}) \quad r^2 = 0.997 \quad \text{SD} = 0.58 \quad [1]$$

Subsequently, the 44 oil samples constituting the training set for the FT-NIR calibration were analyzed in duplicate by the SB-HATR method and their *trans* contents were predicted using Equation 1. The duplicate predictions are plotted against each other in Figure 2; the equation for the line is:

$$\% \text{ trans}_1 = 0.0181 + (1.0086 * \% \text{ trans}_2) \quad r^2 = 0.999 \quad \text{SD} = 0.40 \quad [2]$$

The SD associated with Equations 1 and 2 indicate that the reference method can be reproducible to within $\pm 0.40\%$ *trans* and accurate to $\pm 0.60\%$ *trans*. For comparative purposes, selected LG samples, analyzed by GC, were also analyzed by the SB-HATR method, and the GC *trans* data were regressed against the FT-IR-predicted *trans* contents to obtain an estimate of their relative degree of concurrence, which is summarized by Equation 3.

$$\text{GC } \% \text{ trans} = 0.24 + 0.975 \text{ SB-HATR } \% \text{ trans} \quad r^2 = 0.998 \quad \text{SD} = 0.92 \quad [3]$$

Equation 3 indicates that the LG GC data, obtained using the new AOCS GC method (5), are a remarkably good match to the SB-HATR data, corroborating the observations of Adam *et al.* (11) that general concurrence between GC and IR *trans* data is finally being achieved.

Spectroscopy and calibration development/assessment. Figure 3 illustrates the spectral characteristics of a low-*trans* and a high-*trans* edible oil across the NIR and mid-IR regions of the spectrum. Owing to the low intensity of NIR absorption in comparison with mid-IR absorption, the absorbance in the NIR region ($10,000\text{--}4,000\text{ cm}^{-1}$) has been magnified 10-fold to allow detail to be seen. The bands in the NIR region are broad and nebulous in comparison with the detailed structure observed in the mid-IR region. What is particularly note-

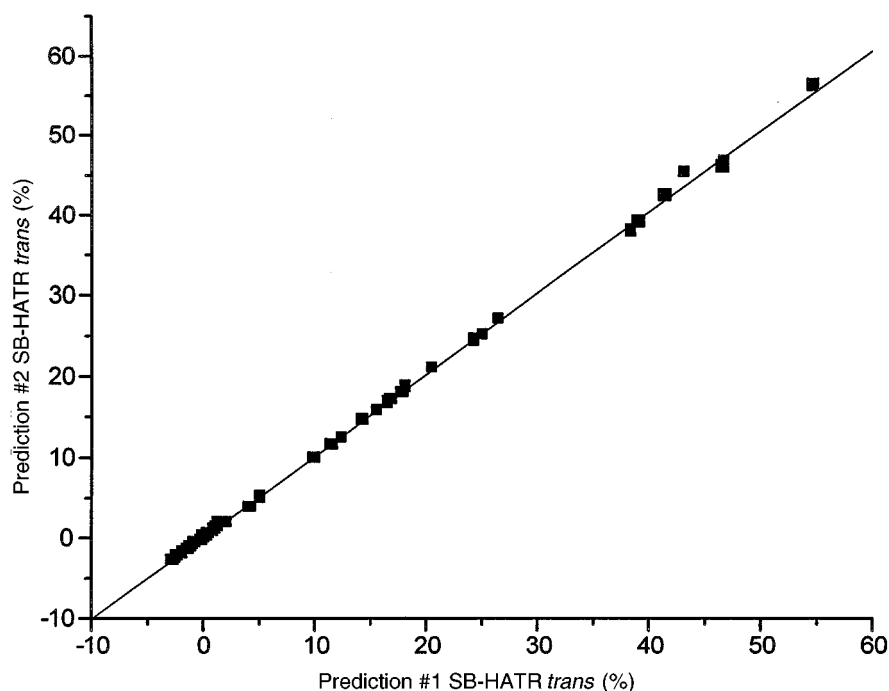


FIG. 2. Duplicate predictions of California/Bunge group calibration standards by single-bounce horizontal attenuated reflectance (SB-HATR) method.

worthy about these spectra, in relation to the quantitation of *trans* content, is the lack of any discernible difference between the spectra of the low- and high-*trans* oils in the NIR region, whereas the mid-IR portion of the spectrum shows a clear-cut, strong *trans* signal at 967 cm^{-1} . Although the NIR region contains much of the same information as the mid-IR

region, extensive band overlap makes it more difficult to extract this information since the NIR bands correspond to overtones and combinations of the fundamental vibrations that give rise to mid-IR absorption bands. As a result, in most cases, the only means of carrying out quantitative work in the NIR region is to make use of more sophisticated chemomet-

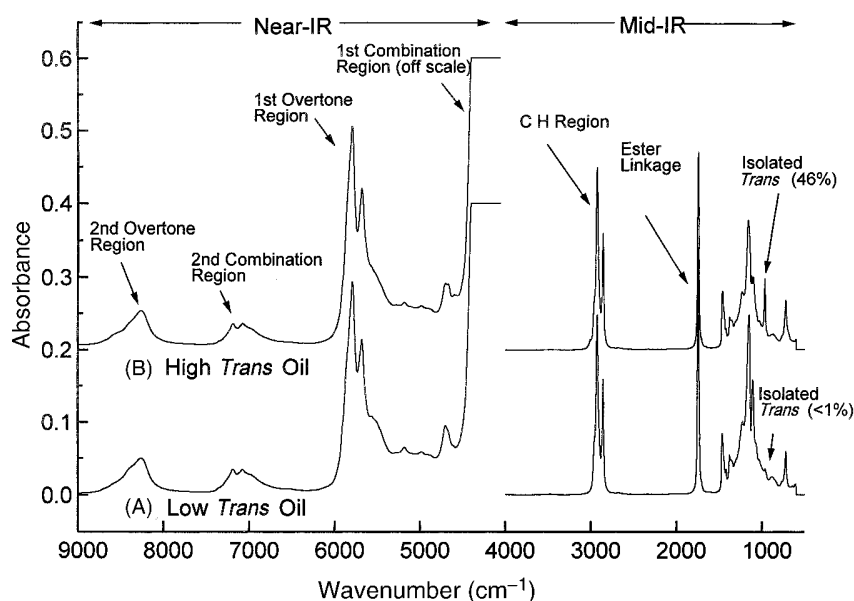


FIG. 3. Collated near/mid-infrared (IR) spectra of (A) a low-*trans* oil and (B) a high-*trans* oil, illustrating the key spectral features associated with the two IR spectral regions. The fundamental *trans* absorption at 967 cm^{-1} is readily discernible in the mid-IR region. The absorbance values in the region from $10,000$ to $4,000\text{ cm}^{-1}$ have been magnified by a factor of 10 to provide greater detail.

ric techniques, such as PLS regression, which provide a means of developing a calibration by mathematically correlating spectral changes to changes in the measure of interest. In the present work, PLS was used to derive a generalized FT-NIR calibration for the prediction of *trans* content.

The FT-NIR spectra of the samples in the Cal-1 training set (Table 2) were input, along with their SB-HATR reference *trans* values into a chemometrics program to develop a generalized PLS calibration. Correlation and pure-component spectra as well as PRESS and cross-validation results indicated that the regions selected in our initial study (7) were optimal for the determination of *trans* content. The optimal spectral regions were found to be 8,975–7,189 cm^{-1} with a single-point baseline at 7,575 cm^{-1} , 5,238–5,056 cm^{-1} with a single-point baseline at 5,247 cm^{-1} , and 4,779–4,564 cm^{-1} with a single-point baseline at 4,800 cm^{-1} . The Cal-1 PLS model used five factors and Figure 4 illustrates the cross-validation plot of FT-NIR predicted *trans* vs. SB-ATR *trans* content, expressed as % trielaidin with its linear regression equation being:

$$\text{FT-NIR } trans = 0.17 + 0.99 * \text{SB-HATR } trans \quad r^2 = 0.995 \quad \text{SD} = 1.51 \quad [4]$$

The MD_a and SDD_a for the predictions from the Cal-1 calibration relative to the ATR *trans* values were -0.05 and 1.51 , respectively, quite similar to the results obtained in our first FT-NIR *trans* study (7). In that study, as in the present work, the reference values were obtained by mid-FT-IR spectroscopy; however, a PLS-based method using a 25- μm transmission flow cell was employed instead of the SB-HATR method. The comparable accuracy obtained in the two studies indicates that the performance of the two mid-FT-IR *trans* methods is essentially equivalent, such that either can serve as a reference method in the development of an FT-NIR cali-

bration. It also indicates that the use of glass vials for sample handling, with the use of a spectral normalization procedure to compensate for their inherent pathlength variability (9), does not appear to detract from the general performance of the FT-NIR predictive capability relative to the use of a constant pathlength transmission cell.

Although the FT-NIR predictions obtained clearly track the reference values (Fig. 4), the overall cross-validation SD is $\pm 1.5\%$ *trans*, about three times that of the r of the SB-HATR reference method. Cal-1 was subsequently validated using two sets of validation oils, Val-1 (CBG samples) and Val-2 (LG samples). Table 3 illustrates the MD and SDD obtained for both a and r of the FT-NIR predictions for these two validation sets relative to their respective reference methods. Overall, the SDD_r for duplicate FT-NIR *trans* predictions is very similar to that of the reference SB-HATR method. The SDD_a reflects a somewhat better accuracy than indicated by the cross-validation results (± 1.1 vs. ± 1.5), most likely because the calibration training set spanned a wider range of *trans* values than the validation samples. The similarity of the SDD_a values for the two validation sets provides further evidence of the general consistency between the AOCS SB-HATR and AOCS-GC reference methods. The SDD_a values are about twice the SD for the SB-HATR method in Equation 1. This somewhat poorer accuracy is not surprising given the need to extract the *trans* spectral information from weak overtone and harmonic bands rather than the sharp, fundamental band present in the mid-IR region.

In this context, we also evaluated whether Cal-1 could be further refined to better predict LG type samples by the inclusion of 20 additional representative samples in the training set (Cal-2). The MD and SDD statistics for this calibration

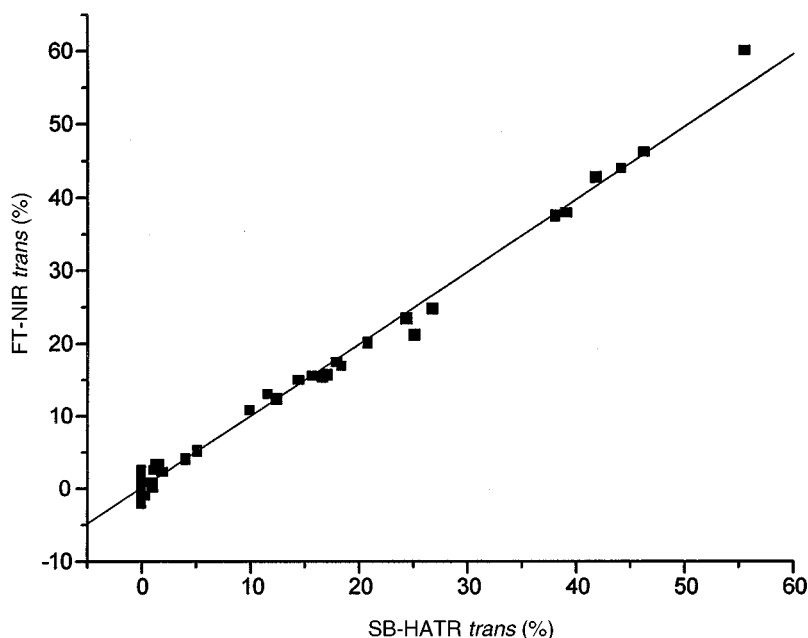


FIG. 4. A cross-validation plot of FT-NIR *trans* predictions obtained using the Cal-1 vs. the SB-HATR *trans* reference values. See Figure 2 for abbreviation.

TABLE 3
MD and SDD for Reproducibility (*r*) and Accuracy (*a*) Obtained for Samples Analyzed with the General (Cal-1) and the Refined (Cal-2) *trans* Calibrations as Assessed Using the Two Validation Sets (Val-1 and Val-2)^a

Validation set	Reference method	Cal-1 statistics		Cal-2 statistics	
Val-1 <i>n</i> = 21 CBG oils	AOCS	MD _{<i>r</i>} = -0.08	SDD _{<i>r</i>} = 0.55	MD _{<i>r</i>} = -0.15	SDD _{<i>r</i>} = 0.42
	SB-HATR	MD _{<i>a</i>} = -0.06	SDD _{<i>a</i>} = 1.13	MD _{<i>a</i>} = 0.02	SDD _{<i>a</i>} = 1.12
Val-2 <i>n</i> = 20 LG oils	AOCS	MD _{<i>r</i>} = -0.04	SDD _{<i>r</i>} = 0.38	MD _{<i>r</i>} = -0.05	SDD _{<i>r</i>} = 0.38
	GC	MD _{<i>a</i>} = -0.15	SDD _{<i>a</i>} = 1.11	MD _{<i>a</i>} = -0.27	SDD _{<i>a</i>} = 1.01

^aAOCS, American Oil Chemists' Society; SB-HATR, single-bounce horizontal attenuated total reflectance; MD, mean differences; SDD, standard deviations of the differences; GC, gas chromatography.

are presented together with those for Cal-1 in Table 3. The accuracy of the predictions for the Val-2 set did not improve appreciably with the inclusion of LG samples in the training set, implying that the training set for Cal-1 modeled the LG samples adequately. Subsequently, a calibration was developed specifically for the LG oils; five samples of each oil type, a total of 20 samples, were used to develop the calibration (Cal-3). The reference values for this calibration were provided by GC analyses, and the optimized Cal-1 spectral regions were employed. Cal-3 was used to predict Val-1 and Val-2, and the prediction statistics, in terms of MD and SDD for *r* and *a*, are given in Table 4. As expected, since Cal-3 is specific to LG samples, it is useless for predicting Val-1 oils. However, Cal-3 does a very good job of predicting the *trans* values for the Val-2 (LG) samples. The SDD_{*a*} is about ±0.50% *trans*, significantly better than ±1.1% *trans* for Cal-1.

Discriminate analysis. When implementing a PLS calibration, the PLS-predicted values for samples whose spectra differ significantly from those of the training set are not reliable. This is well illustrated by the results presented above for Cal-3. Because the training set for this calibration was restricted to four specific oil types (LG oil types A–D), the *trans* predictions obtained for Val-1 oils were off by 20% or more. Similarly, if a sample were atypical (e.g., containing excess free fatty acids or heavily oxidized relative to the samples in the training set), it may not be accurately predicted. Thus, practical implementation of a PLS calibration should incorporate a means for detecting “outliers,” i.e., samples that are not well represented spectroscopically by the samples used to derive the calibration.

The AIRS software includes discriminate analysis capability, which can be used to determine whether spectra of

samples are consistent with the characteristics of the calibration training set. As detailed in a previous paper (9), the use of discriminate analysis for outlier detection can be based on a variety of criteria, including the PLS-predicted value of the parameter of interest, the spectral residual, or the factor scores. These three types of discrimination criteria were devised for Cal-1. Based on these criteria, none of the samples in Val-1 and Val-2 were identified as outliers. This provides further evidence that the Val-2 samples were adequately modeled by the general Cal-1 training set. Refining this calibration by the addition of LG samples to the training set (Cal-2) did not provide a significant improvement.

In the case of Cal-3, all the samples in Val-1 were flagged as outliers when tested against the discrimination criteria established for this calibration, as would be expected on the basis of the makeup of the training set (Table 2). All the samples in Val-2 passed the Cal-3 discrimination criteria. Subsequently, four calibrations, Cal-4(a–d), were developed specifically for each LG oil type, and discrimination criteria were set to determine whether the AIRS discriminate analysis routine could distinguish among the four oil types. Since the *trans* ranges of oil types C and D overlap appreciably (Table 1), difficulty in distinguishing between oils of these two types might be anticipated. However, the AIRS discriminate analysis routine was readily able to distinguish among samples of all four oil types. This capability could be very useful, as it not only provides a means of ensuring that the calibration in place is appropriate for the sample being analyzed but also can be used to automate the selection of the most appropriate calibration from among a set of available calibrations. The AIRS software tests the sample spectrum using the discrimination criteria for each calibration and uses this information to select the most

TABLE 4
MD and SDD for *r* and *a* Obtained for Samples Analyzed Using the General Lipton Group Calibration (Cal-3) Using the Two Validation Sets (Val-1 and Val-2)^a

Validation set	Reference method	Cal-3 statistics	
Val-1 <i>n</i> = 21 CBG	AOCS	MD _{<i>r</i>} = -0.76	SDD _{<i>r</i>} = 0.91
	SB-ATR	MD _{<i>a</i>} = -14.11	SDD _{<i>a</i>} = 17.30
Val-2 <i>N</i> = 20 LG	AOCS GC	MD _{<i>r</i>} = 0.18	SDD _{<i>r</i>} = 0.61
		MD _{<i>a</i>} = 0.48	SDD _{<i>a</i>} = 0.62

^aSee Table 3 for abbreviations.

appropriate calibration to predict the *trans* content of the sample. Thus, samples of different oil types can be analyzed in random order without any need for operator intervention to select an appropriate calibration.

Based on our experience to date (9), we are finding that discriminate analysis is a very powerful and important tool, which, in our opinion, should be an integral part of any IR spectroscopic analytical method to be implemented in an industrial situation. The application of discriminate analysis routines helps eliminate errors such as mixing up a sample or analyzing one that is spoiled or contaminated, thereby providing an extra degree of confidence in the analytical results.

This study demonstrates that despite the lack of a discernible *trans* absorption band in the NIR portion of the spectrum, the *trans* content of edible fats and oils can be predicted from their FT-NIR spectra through the application of PLS. By employing a training set comprising of a wide variety of oil types, a generalized calibration was developed that allowed the *trans* content of unknown samples, analyzed in disposable glass vials, to be predicted with an accuracy of $\sim \pm 1.1\%$ *trans*. Supplementing the calibration training set with a subset of a specific oil type or group does not appear to improve the accuracy of the basic calibration. On the other hand, it is possible to obtain more accurate predictions by calibrating on a more limited training set of oils having specific characteristics (e.g., canola oils undergoing a standardized hydrogenation process). These more restricted calibrations are strictly limited to oils having the characteristics reflected by the training set, and discriminate analysis is an important supplementary tool for these types of calibrations because it provides the capability to flag samples that do not meet this requirement. The generalized FT-NIR *trans* calibration does not match the performance characteristics of the mid-FT-IR SB-ATR method, but when specific calibrations are developed for well-defined oil types, the accuracy approaches that of the mid-FT-IR method. NIR spectroscopy is clearly a competitive, alternative approach to mid-IR spectroscopy for the rapid determination of the *trans* content of fats and oils. It has the advantage of using more robust instrumentation and a simpler sample handling system better suited to the industrial QC environment. Supplemented with the AIRS discriminate analysis package, the FT-NIR system evaluated provides a practical means for the edible oil sector to comply with the new *trans* labeling requirements recently proposed by the FDA.

ACKNOWLEDGMENTS

The authors thank Mike Adam of Lipton as well California Oils Co. and Bunge Foods for supplying oil samples used in this study and Dr. Magdi M. Mossoba of the U.S. Food and Drug Administration for providing the ultra-degummed bleached expelled soybean oil. Special thanks to the Natural Sciences and Engineering Research Council of Canada (NSERC) and Bomem Inc. for the financial assistance to allow this research to be carried out under the auspices of the Collaborative Research and Development (CRD) program.

REFERENCES

1. Haumann, B.F., Dietary Fats Still Spark Controversy, *inform* 5:358 (1994).
2. Aro, A., Epidemiological Studies of *trans* Fatty Acids and Coronary Heart Disease, in *Trans Fatty Acids in Human Nutrition*, edited by J.L. Sebedio and W.W. Christie, The Oily Press, Dundee, 1998, pp. 235–260.
3. *Federal Register*, Vol. 64, No. 221 (Nov. 17, 1991).
4. *Official and Tentative Methods of the American Oil Chemists' Society*, edited by V.C. Mehlenbacher, American Oil Chemists' Society, Chicago, 1946, Method Cd 14-61.
5. *Official Methods and Recommended Practices of the American Oil Chemists' Society*, 5th edn., edited by D. Firestone, AOCS Press, Champaign, 1998, Methods Ce 1g-96, Cd 14d-97, and Ce 1f-97.
6. van de Voort, F.R., A.A. Ismail, and J. Sedman, A Rapid Automated Method for the Determination of *cis* and *trans* Content of Fats and Oils by Fourier Transform Infrared Spectroscopy, *J. Am. Oil Chem. Soc.* 72:873–880 (1995).
7. Li, H., F.R. van de Voort, J. Sedman, and A.A. Ismail, Rapid Determination of *cis* and *trans* Content, Iodine Value, and Saponification Number of Edible Oils by Fourier Transform Near-Infrared Spectroscopy, *Ibid.* 76:491–497 (1999).
8. Anonymous, A Global Iodine Value Calibration for the Oil and Fat Analyzer, *Application Note ABB*, Bomem Inc., Quebec, Canada (1999).
9. Li, H., F.R. van de Voort, A.A. Ismail, J. Sedman, R. Cox, C. Simard, and H. Buijs, Discrimination of Edible Oil Products and Quantitative Determination of Their Iodine Value by FT-NIR Spectroscopy, *J. Am. Oil Chem. Soc.* 77:29–36 (2000).
10. Youden, W.J., and E.H. Steiner, *Statistical Manual of the AOAC*, Association of Official Analytical Chemists, Arlington, 1975.
11. Adam, M., M.M. Mossoba, T. Dawson, M. Chew, and S. Wasserman, Comparison of Attenuated Total Reflection Infrared Spectroscopy to Capillary Gas Chromatography for *trans* Fatty Acid Determination, *J. Am. Oil Chem. Soc.* 76:375–378 (1999).

[Received January 19, 2000; accepted July 17, 2000]