Upgrading the AOCS Infrared *trans* Method for Analysis of Neat Fats and Oils by Fourier Transform Infrared Spectroscopy

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ABSTRACT: An automated protocol for the direct, rapid determination of isolated *trans* content of neat fats and oils by Fourier transform infrared (FTIR) spectroscopy was devised, based on a simple modification of the standard AOCS *trans* method, eliminating the use of CS_2 and methylation of low *trans* samples. Through the use of a commercially available, heated transmission flow cell, designed specifically for the analysis of neat fats and oils, a calibration (0–50%) was devised with trielaidin spiked into a certified, *trans*-free soybean oil. The single-beam spectra of the calibration standards were ratioed against the single-beam spectrum of the base oil, eliminating the spectral interference caused by underlying triglyceride absorptions, facilitating direct peak height measurements as per the AOCS IR *trans* method. The spectrometer was preprogrammed in Visual Basic to carry out all spectral manipulations, measurements, and calculations to produce *trans* results directly as well as to provide the operator with a simple interface to work from. The derived calibration was incorporated into the software package, obviating the need for further calibration because the program includes an automatic recalibration/standardization routine that automatically compensates for differences in optical characteristics between instruments, instrument drift over time, and cell wear. The modified AOCS FTIR analytical package was evaluated with Smalley check samples for repeatability, reproducibility, and accuracy, producing SD of ± 0.07, 0.13, and 0.70 *trans,* respectively, the FTIR predictions being linearly related to the Smalley means ($r = 0.999$; SD = ± 0.46), and well within one SD of the Smalley sample means. Calibration transfer was assessed by implementing the calibration on a second instrument and reanalyzing the Smalley check samples in cells of two different pathlengths (25- and 50-µm). There were no statistically significant differences between the FTIR *trans* predictions obtained for the Smalley samples from the two instruments and two cells, indicating that the software was able to adjust the calibrations to compensate for differences in instrument response and cell pathlength. The FTIR isolated *trans* analysis protocol developed by the McGill IR Group has the benefit of being based on the principles of an AOCS-approved method, matches its accuracy, and allows the analysis to be performed on both neat fats and oils, producing *trans* predictions in less than 2 min per sample. It is suggested that this integrated approach to *trans* analysis, which requires a minimum level of sample manipulation and operator skill, be considered as a modification of the proposed Recommended Practice CD14b-95.

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KEY WORDS: Fats and oils, Fourier transform infrared spectroscopy, FTIR spectroscopy, lipid analysis, oil analysis, *trans* analysis, triglycerides.

Edible fats and oils vary in their triglyceride makeup, relative degree and forms of unsaturation (*cis* and *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 and leads to increased levels of *trans* fatty acids, now of increased concern to health professionals because of their association with heart disease (1). *Trans* fatty acid levels can reach values of 40% or more in hardened fats, and the United States Food and Drug Administration has been petitioned to require the inclusion of *trans* fatty acids as part of saturated fat content in the labeling of fat-based products, such as margarines, spreads, and frying fats (2,3). *Trans* analysis by infrared (IR) spectroscopy was developed over 40 yr ago with dispersive IR instrumentation and is a well-established AOCS official method (4). The limitations of the method have been well documented, specifically the fact that the method is not applicable to oils that contain conjugated *trans/trans* and *cis/trans* bonds, requires the use of a volatile and noxious solvent (CS_2), and is affected by overlap of the *trans* peak by underlying triglyceride absorptions, which contribute significantly to the *trans* peak height measurement at low *trans* levels (<15%), requiring the oil to be saponified and methylated to overcome this interference. The official methodology has recently been updated (5) to take advantage of Fourier transform IR (FTIR) instrumentation and computerized data analy-

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sis techniques; however, the inherent limitations noted previously remain.

The McGill IR Group has been carrying out research into the development of simple, rapid, and accurate methods of edible oil analysis based on FTIR spectroscopy (6–13). One of the practical developments of this work has been an FTIR-based edible oil analysis package, which is capable of simultaneously determining iodine value (IV), saponification number (SN), and *cis* and *trans* content (IV/SN/*cis*/*trans*) in a single analysis on a neat fat or oil sample in less than 2 min (11). The system is preprogrammed and precalibrated and uses a heated sample-handling accessory (11) to allow both fats and oils to be analyzed in their liquid state at 80°C. This system is being used by a number of edible oil processors; however, the fact that it employs a relatively sophisticated multicomponent analysis approach based on partial least squares (PLS) regression, the principles of which are not well understood by most users, may hinder its general acceptance. For many analysts in industry and regulatory agencies, reference to an approved methodology is highly desirable and often a requirement; hence, there is a place for a simplified, improved, and automated technique based on the well-defined and accepted analytical concepts associated with the official AOCS *trans* method. This paper describes an updated and automated version of the AOCS *trans* analysis procedure that eliminates the need for CS_2 and methylations by taking advantage of the ratioing capabilities of FTIR spectroscopy and making use of a dedicated sample-handling accessory that allows the *trans* analysis to be carried out in less than 2 min per sample.

MATERIALS AND METHODS

Instrumentation/spectral acquisition. The instruments used were Nicolet Magna and Protégé FTIR spectrometers (Madison, WI), controlled by 486 PCs running under Nicolet's Omnic 3.0 software; the two instruments utilized the same type of source (Globar) and detector [deuterated triglycine sulfate (DTGS)]. Each instrument was equipped with a heated sample-handling accessory (Fig. 1), manufactured by Dwight Analytical (Toronto, Ontario, Canada), and fitted with either a 50- or 25-μm KCl flow cell maintained at 80°C. Both spectrometers were continuously purged with CO_2-free dry air, supplied by a Balston dryer (Balston, Lexington, MA), to minimize spectral interferences from water vapor and carbon dioxide. All spectra were collected by co-adding 64 scans (calibration standards) or 32 scans (samples) recorded at a resolution of 4 cm^{-1} and a gain of 1.0.

Calibration. A *trans* calibration set was devised by gravimetrically spiking trielaidin (Sigma Chemical Co., St. Louis, MO) into an unhydrogenated soybean oil with a *trans* content of <0.1% as determined by gas chromatography (AOCS Method Ce 1c-89), to cover a range of 0–80% trielaidin. The calibration was performed by recording the spectra of these standards in a 25-μm cell inserted into the sample-handling accessory (maintained at 80°C) installed in the sample-handling compartment of the Magna spectrometer and ratioing

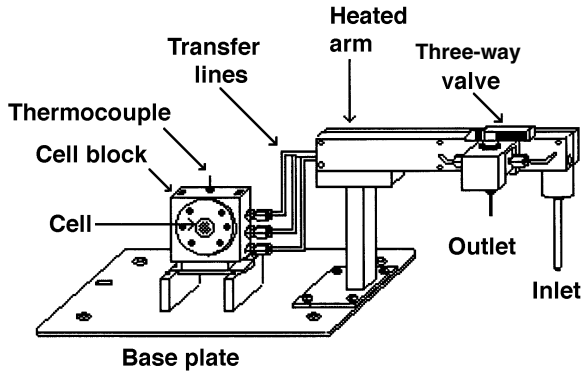

FIG. 1. Schematic diagram of the heated transmission flow cell and sample-handling accessory used for the analysis of neat fats and oils.

the spectrum of each standard against that of the *trans*-free base oil to produce a "differential spectrum." The concentrations of trielaidin in these standards were 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}. A calibration equation was derived by linear regression of peak height vs. the gravimetric *trans* content added to the base oil. A program was written in Microsoft Visual Basic (Redmond, WA) to carry out peak height measurements and calculate the *trans* content by using the calibration equation derived. In addition, the program developed for the analysis included a simple menu to guide the operator in collecting the spectra, as well as calibration diagnostics and automatic recalibration routines.

Samples/analyses. To assess the performance of the modified AOCS method, seven Smalley *trans* check samples were obtained from the 1995 series, for which results were already available. These samples were analyzed by FTIR spectroscopy with our preprogrammed ratio approach, here termed the modified AOCS FTIR method. The spectra of all analyzed samples were ratioed against the same *trans*-free soybean oil spectrum as employed in deriving the calibration. Prior to analysis, the samples were prewarmed in a microwave oven to ~80°C to melt any solids and aspirated into the IR cell. The samples were run in duplicate to determine repeatability (back-to-back scans of the same sample) and reproducibility (two consecutive loadings). Accuracy was evaluated by comparing the FTIR results to the Smalley sample means, the paired data sets being assessed in terms of mean difference (MD) and standard deviation of the differences (SDD) (14). In subsequent experiments, calibration transfer was assessed by analyzing the Smalley check samples on a second spectrometer (Protégé) with two cells of different pathlengths (50 and 25 μm). The purpose was to test the ability of the software to compensate for both instrument and pathlength changes. After each calibration adjustment for any instrument or cell configuration change, the Smalley test samples were run and the results collated and subjected to analysis of variance (ANOVA) to determine if there were any sig-

nificant differences between the results obtained with different instruments and/or cell pathlengths. In addition, the *trans* values predicted with the IV/SN/*cis*/*trans* analysis package were compared to the data obtained with the modified AOCS FTIR method.

Spiking experiments. Canola, corn, coconut, soybean, and sunflower oil were purchased locally. These oils were gravimetrically spiked with trielaidin at levels of 1.5–7% and analyzed as described.

RESULTS

Figure 2A 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 soybean 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 2B. The latter 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 2A and 2B, are presented in Figures 3A and 3B, respectively. These plots are reasonably linear, although we and others (15) have noted that there is significant curvature beyond 50% *trans* and that accurate analysis beyond this value requires a quadratic fit. The equations obtained by simple linear regression over the range of 0–50% *trans* are presented in Equations 1 and 2, respectively.

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

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

where: % *trans* = *trans* content expressed as % trielaidin; A_u (966) = absorbance at 966 cm^{-1} relative to a baseline drawn between 995 and 937 cm^{-1} (raw spectrum); A_r (966) = absorbance at 966 cm^{-1} relative to a baseline drawn between 995 and 937 cm^{-1} (ratioed spectrum). 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 obtained by using the ratioing procedure. Equation 2 was incorporated into a Visual Basic program to allow for the direct prediction of *trans* values of neat fat or oil samples loaded into the IR cell. The program also included calibration update routines to adjust the calibration equation in order to compensate for changes in cell pathlength or instrument response.

Repeatability, reproducibility, and accuracy of FTIR data. One of the recurrent problems with validating *trans* methodology is obtaining reference samples with *trans* values that one can have confidence in. Because there are no absolute standards *per se*, other than pure *trans* triglycerides, it was determined that the best approach would be to rely on the AOCS Smalley check samples, which provide a range of *trans* levels (~0–40% *trans*) and have been analyzed by a number of laboratories (15–18 laboratories, depending on the samples in question) by using the conventional AOCS IR method. After outlier removal, the average *trans* values provide a benchmark value for each sample as well as a standard deviation around its mean. Using the seven samples available, we evaluated the repeatability, reproducibility, and accuracy of our modified AOCS method in terms of MD and SDD (Table 1).

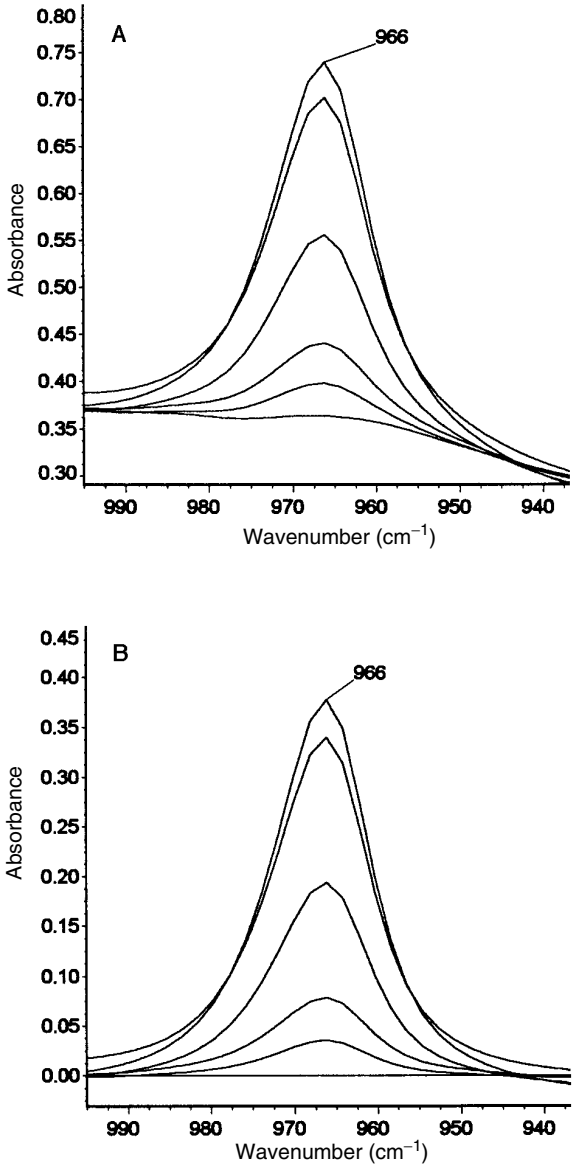

FIG. 2. (A) Overlaid spectra in the *trans* absorption region of calibration standards prepared by addition of various amounts of trielaidin to a *trans*-free soybean base oil; (B) same spectra as in (A) after ratioing out the spectrum of the base oil.

In terms of repeatability and reproducibility, duplicate analyses show no bias relative to each other in terms of MD, which ideally would be zero. SDD, a measure of the variability around MD, is less than ± 0.1 in terms of repeatability of two consecutive scans of the same samples, while for the re-

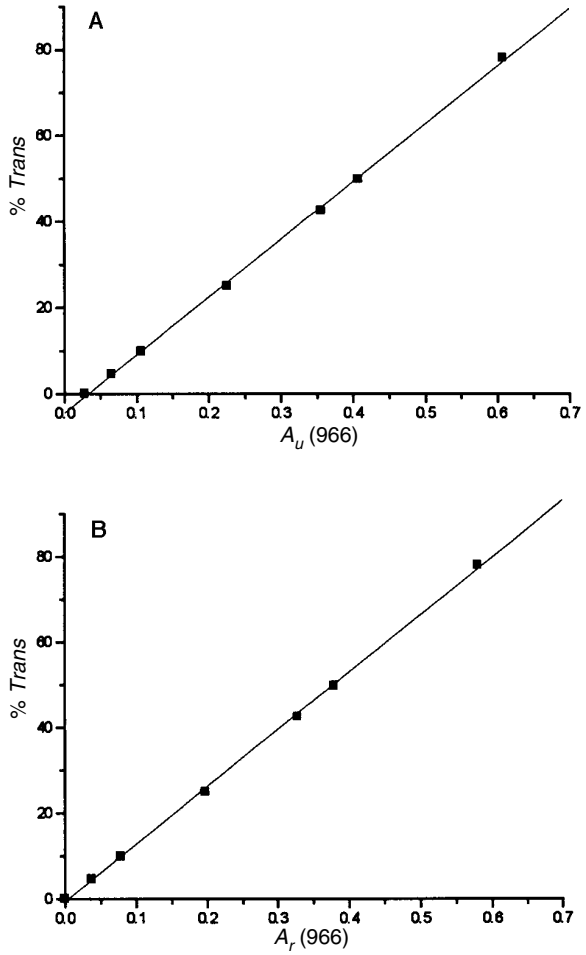

FIG. 3. Calibration curves obtained for unratioed (A_u) and ratioed (A_r) spectra of trielaidin in a *trans*-free oil, illustrating the removal of the intercept caused by underlying absorptions when the spectra are ratioed. These plots have some curvilinearity and are better fitted by a quadratic function if concentrations above 50% are considered.

producibility for two separate loadings, it rises to ± 0.13 *trans*. These data provide an assessment of the stability of the FTIR data and are an indication of excellent temperature control. In terms of accuracy, the means of the Smalley check samples were used as the reference values, compared to the means of the reproducibility runs, with the MD being close to zero and the SDD being ± 0.7 *trans*. Figure 4 presents a plot of our FTIR results vs. the Smalley check sample means. The Smalley means plus their SD as well as the means minus their SD are also plotted on the y axis for comparison to illustrate the spreads around the Smalley means.

Figure 4 illustrates that there is an excellent linear relationship between the FTIR means relative to the Smalley check sample means. The data effectively pass through the center of the Smalley SD limits, with the exception of the lowest value, which was determined to be zero *trans* by the modified FTIR method vs. the Smalley mean value of 1.37% *trans*. The equation for the line was

TABLE 1
Raw Data for Repeatability, Reproducibility, and Accuracy for *trans* Analysis of the Smalley Check Samples by the Modified AOCS Fourier Transform Infrared (FTIR) Method[a]

Smalley	Repeatability[b]		Reproducibility[b]		Accuracy[b]	
sample	FTIRa	FTIRb	FTIR1	FTIR2	FTIR12	Smalley
1	16.2	16.3	16.3	16.1	16.20	15.985
2	0.0	0.1	0.0	0.0	0.00	1.372
3	3.9	3.9	4.0	3.9	3.95	4.067
4	8.5	8.6	8.6	8.4	8.50	8.829
5	16.3	16.3	16.4	16.1	16.35	16.191
6	32.3	32.2	32.4	32.0	32.20	31.38
7	40.7	40.7	40.8	40.6	40.70	40.291
Mean	16.842	16.871	16.928	16.728	16.842	16.873
MD	−0.028		0.200		0.030	
SDD	0.075		0.129		0.696	
CV	0.40%		0.76%		4.30%	

[a]Statistical comparisons are summarized in terms of overall mean for each sample as well as mean difference (MD), standard deviation of the differences (SDD), and coefficient of variation (CV).
[b]Data obtained with 25-µm cell in Magna spectrometer (Nicolet, Madison, WI).

FTIR *trans* = −0.602 + 1.038 Smalley *trans* r = 0.999 SE = 0.478 [3]

These analytical data provide evidence that the developed FTIR methodology produces results that fall within one SD of the mean values obtained by other laboratories that carried out the same analysis by the traditional AOCS method.

Subsequent series of analyses of the Smalley check samples were carried out on a second spectrometer with two cells of two different pathlengths to test the recalibration and standardization routines associated with the analytical software package as well as to assess the reproducibility of the analysis under variable conditions. The results of these analyses, as well as the corresponding *trans* results obtained using the PLS-based IV/SN/*cis*/*trans* analysis package, are presented in Table 2. The means obtained with instrument 2 are similar

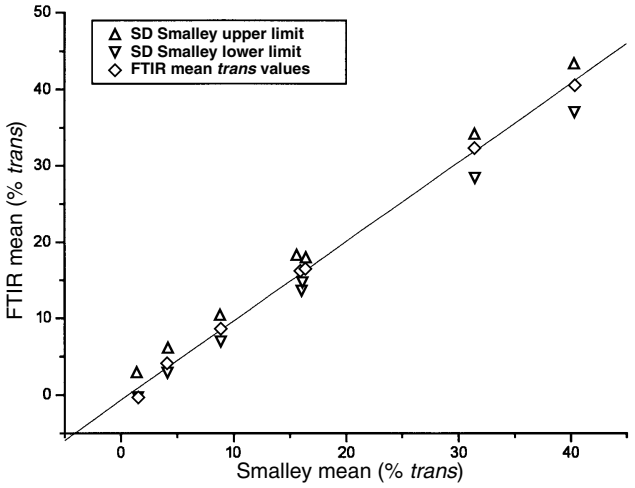

FIG. 4. A plot of the Fourier transform infrared (FTIR) predictions for the Smalley check samples obtained by the modified AOCS method vs. the Smalley means, with the triangles above and below the regression line representing one SD around the Smalley means.

TABLE 2
Smalley Check Sample FTIR Results Obtained by Transferring the Calibration Equation Obtained with a 25-μm Cell in a Magna Spectrometer (Instrument 1) to a Protégé Spectrometer (Instrument 2) with 50- and 25-μm Cells[a]

Smalley sample number	Modified AOCS Instrument 1[b] 25-μm Cell	Modified AOCS Instrument 2[b] 25-μm Cell	Modified AOCS Instrument 2 50-μm Cell	PLS FTIR Instrument 2 25-μM Cell	Smalley mean
1	16.2	16.3	15.7	15.3	15.98
2	0.0	0.0	0.0	0.0	1.37
3	3.9	4.0	3.7	3.8	4.07
4	8.5	8.5	8.2	8.0	8.83
5	16.3	16.3	15.9	15.5	16.19
6	32.2	32.2	31.7	32.2	31.38
7	40.7	41.7	40.3	40.4	40.29
Mean	16.84	17.00	16.50	16.46	16.87

[a]These data are compared to the *trans* values obtained with a partial least squares regression (PLS)-based calibration model as well as the Smalley mean *trans* values.
[b]Instrument 1: Magna spectrometer (Nicolet, Madison, WI). Instrument 2: Protégé spectrometer (Nicolet). For abbreviations see Table 1.

to those for instrument 1, presented in Table 1. ANOVA and the Duncan Multiple Range tests indicated that there were no significant differences ($P < 0.01$) between the results obtained for the two instruments or the two cells, indicating that the base calibration produces consistent results when the calibration is updated. The *trans* data produced by the PLS multicomponent analysis package were also not significantly different from the data produced by the peak-height method. These results corroborate our previous conclusion that the PLS approach produces *trans* values that are in agreement with those obtained by the modified FTIR AOCS method (Sedman, J., F.R. van deVoort, A.A. Ismail, and P. Maes, submitted for publication).

Spiking experiments. The accuracy of the *trans* predictions obtained from the FTIR ratioing method is expected to depend to some extent on how closely the triglyceride composition of the samples being analyzed resembles that of the *trans*-free reference oil employed because this will affect the accuracy of the ratioing out of the underlying triglyceride absorptions. Accordingly, the validation study with the Smalley check samples (hydrogenated soybean oils) described previously represents the optimal situation in which the samples are of the same oil type as the reference oil. To assess the analytical accuracy obtained with other types of oils, we prepared a set of samples by spiking pure trielaidin into five different oils (canola, coconut, corn, soybean, and sunflower) at levels of 1.5–7%. The samples were then analyzed by the FTIR ratioing method in the same manner as described above for the Smalley check samples, i.e., we used the *trans*-free soybean oil as the reference oil and employed the calibration equation derived from the trielaidin-spiked soybean standards (Eq. 2). The FTIR *trans* predictions for all these spiked samples were within one percentage point of the gravimetric values, demonstrating that the ratioing procedure eliminated the bulk of the contribution of the underlying triglyceride absorptions, even though the five oils employed in these spiking experiments vary widely in triglyceride composition. The *trans* content of each spiked sample was subsequently repredicted after ratioing its spectrum against that of the corresponding unspiked oil to estimate the error contributed by imperfect ratioing of the triglyceride absorptions (as well as to eliminate the possibility that the results of these experiments were confounded by the presence of *trans* isomers in the unspiked oils). The MD between the two sets of *trans* predictions was −0.14, with a SDD of 0.34. These results indicate that the error due to the variability in the underlying triglyceride absorptions among these oils is within the experimental error of the method.

DISCUSSION

The traditional AOCS method for *trans* analysis by IR spectroscopy has a number of well-known drawbacks, particularly the tendency to significantly overpredict *trans* values at the low end owing to underlying triglyceride absorptions, which is circumvented by converting oils to their methyl esters prior to analysis, and the use of noxious and volatile CS_2 as a solvent. Much effort has gone into overcoming these problems, and several authors have worked on means of eliminating CS_2 (15–17) by measuring the *trans* content from the IR spectrum of neat oils (or melted fats) or their methyl esters with attenuated total reflectance (ATR) sampling techniques or short-pathlength (~0.1 mm) transmission cells. Although both approaches are workable, each has limitations. The ATR technique is particularly appealing because it requires only that a neat sample be poured onto the surface of the ATR crystal. However, the technique suffers from difficulties in cleaning the ATR crystal (cross contamination) as well as extreme sensitively to changes in the alignment of the crystal. For short-pathlength transmission cells, the introduction of viscous oil samples into conventional IR cells can be difficult and awkward, and solvent rinses can be problematic in an industrial setting. In most of the previous work (15–17) on *trans* analysis of neat oil samples, the critical issue of temperature con-

trol has not been addressed, and the sample accessories used were not designed to operate at elevated temperatures and thus were not suitable for the routine analysis of solid fats. Even properly designed heated ATR cells tend to be problematic. Cleaning the crystal surface with solvents changes the surface temperature as a result of evaporative cooling and can lead to solubilization of the epoxy that holds the crystal in place, allowing leakage of solvent and sample to the underside of the crystal, causing gross analytical errors and posing an explosion hazard. To overcome these sampling problems, the McGill IR Group decided to design its own dedicated oil analysis accessory, based on a heated flow-through transmission cell that is designed specifically for the analysis of neat fats and oils (11). The elements of this sealed system, illustrated in Figure 1, are a simple three-position valve (off, bypass, and load cell) to facilitate sample handling, a heated cell-bypass line that allows one to rinse the previous sample out of the lines, and an interchangeable transmission flow cell inserted into a stainless- steel cell housing. The cell and lines are all maintained at 80°C (±0.2), and the sample is prewarmed prior to loading to liquify it if necessary. Our experience has shown that precise temperature control is essential in obtaining reproducible results, and the mass of the cell housing is crucial in minimizing temperature fluctuations.

Aside from sample handling, the other major limitation in conventional IR *trans* analysis has been the issue of errors caused by underlying triglyceride absorptions. This matter is easily addressed through the use of FTIR spectroscopy by ratioing the single-beam FTIR spectrum of the fat or oil being analyzed against the single-beam spectrum of a similar reference oil that is free of *trans* double bonds. The ratioing process and the ability to do it accurately is one of the major advantages of FTIR spectroscopy, allowing one to eliminate the common features associated with any two spectra by producing a "differential spectrum" (18) that accentuates the differences between similar samples. As pointed out by a number of authors (16,19,20), ratioing the single-beam spectrum of the sample against that of a *trans*-free oil cancels out the common underlying features under the *trans* band that are associated with the triglyceride backbone and removes the variable baseline tilt caused by the proximity of strong triglyceride fingerprint bands, which make it difficult to draw a proper baseline in a conventional absorbance spectrum. The underlying absorptions cause significant overprediction of the *trans* content in low-*trans* samples, while the sloping baseline gives rise to uncertainty in the peak height measurement. With the ratio approach, the isolated *trans* content can be obtained unambiguously by the AOCS peak height calculation, without any need to convert the oil to methyl esters. This drastically simplifies the method. As shown in Table 2 and discussed in detail elsewhere (Sedman, J., F.R. van de Voort, A.A. Ismail, and P. Maes, submitted for publication), similar results can be obtained with PLS, a form of factor analysis that mathematically eliminates the constant underlying absorptions and baseline fluctuations from the quantitative relation devised from a serial dilution of trielaidin in an oil. However, as noted previously, many analysts are not familiar with this approach.

In making use of the ratio approach, the suitability of a particular reference oil, and hence the range of applicability of the calibration equation developed with that reference oil, is subject to limitations imposed by the variability of the underlying absorptions among different triglycerides. For example, when analyzing partially hydrogenated soybean oils, Mossoba *et al.* (16) found that the *trans* values obtained by using triolein as the reference oil were 2.6 percentage points higher than those obtained when a refined, bleached, and deodorized soybean oil served as the reference material. They attributed this difference primarily to the more accurate ratioing out of the underlying triglyceride absorptions in the latter case. In the present work, this issue was further investigated by spiking five oils of different types with trielaidin at levels of 1.5–7%. We selected this concentration range as a worst-case scenario, because any error due to underlying absorptions would be greatest in samples containing low levels of *trans* isomers. As reported above, these spiking experiments indicated that the residual underlying triglyceride absorptions after the ratioing procedure did not contribute a significant error because the FTIR *trans* predictions obtained were within one percentage point of the gravimetric values for all spiked samples. This suggests that the application of the ratioing method based on the use of a single reference oil and the corresponding calibration equation may serve as a generalized procedure for *trans* determination in a wide variety of edible fats and oils.

The spectral ratioing approach is a simple means by which to eliminate the need for either solvents or methylation, and these advantages have led to ratioing being proposed for adoption by the AOCS as Recommended Practice CD14b-95 for the quantitation of isolated *trans* isomers at levels equal to or greater than 1% (19). We concur with this recommendation but suggest that the methodology be further standardized and automated by using a reliable, functional heated transmission cell and programming the spectrometer. Although most FTIR spectrometers come equipped with basic software that allows for the measurement of peak heights and areas and have simple quantitative packages that allow for construction of Beer's law plots, etc., it can be cumbersome to carry out the appropriate baseline selection, measure peak heights, and produce the corresponding *trans* values. One of the goals of the McGill IR Group is to take FTIR spectroscopy out of the technical environment and make it a routine quality control tool, independent of specialized expertise. This can be done by programming the spectrometer, in this case with Visual Basic algorithms, to produce a user-friendly interface between the operator and the instrument. The operator's sole function is to present the sample to the instrument, and the software takes care of the rest of the analysis and data presentation. This approach has many benefits in the industrial and even the analytical setting, in that there is no major learning curve associated with the analysis, making it possible for

plant personnel to carry out *trans* analyses on a routine basis. Accordingly, a software package based on our modified FTIR AOCS protocol has been developed; it simply presents the results in a spreadsheet after each sample has been aspirated into the IR cell. The analysis takes about 2 min per sample.

The software package also implements concepts of calibration transfer, whereby the calibration can be implemented directly from the software package and adjusted to suit the spectrometer and cell pathlength being used, eliminating the requirement to physically carry out a calibration. The program incorporates a calibration derived for a 25-µm KCl cell, which is the optimal pathlength for analysis over the range of 0–100% *trans*. In terms of sensitivity, the optimal pathlength depends on the maximum *trans* value to be measured. A 50-µm cell provides optimal sensitivity in the 0–50% *trans* range without exceeding the limit of detector linearity. The calibration transfer routine allows the 25-µm calibration to be restandardized for use with longer pathlength cells through the use of a gold standard, which is capable of compensating for pathlength differentials up to 100%. This calibration transfer routine also allows for the calibration to be implemented on another instrument and compensates for background and instrument drift over time and cell wear. These features are standard in several of our other oil packages and have performed well.

This study indicates that the basic AOCS *trans* analysis methodology can be updated and dramatically simplified through the use of the ratioing capabilities of FTIR spectrometers as well as a custom-designed sample-handling accessory. The methodology, as packaged and programmed by the McGill IR Group, using a dedicated, temperature-controlled oil analysis accessory, produced results that are consistent with the mean results obtained for the 1995 Smalley check sample set by a number of laboratories using the traditional AOCS method. The devised protocol eliminates the need for solvents and methylation, and because the calibration equation is part of the software, which can be installed on any FTIR spectrometer operating under Omnic software, calibration can be eliminated. Standardization of the methodology with proper accessories, temperature control, and programming could help to facilitate the routine determination of *trans* content in fats and oils. It is suggested that the McGill IR Group *trans* analysis protocol, which requires a minimum of sample manipulation and operator skill, be considered as a modification of the newly proposed Recommended Practice CD14b-95.

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