Quantitative Differential Scanning Calorimetric Analysis for Determining Total Polar Compounds in Heated Oils

C.P. Tan and Y.B. Che Man*

Department of Food Technology, Faculty of Food Science and Biotechnology,
Universiti Putra Malaysia, 43400 UPM Serdang, Malaysia

ABSTRACT: A new differential scanning calorimetry (DSC) method was developed for the determination of total polar compounds (TPC) in heated oils. Three different types of edible oils, refined, bleached, and deodorized corn oil (CO), palm olein (RBDPO), and soybean oil (SO), were used in this study. Each type of edible oil was heated at 180°C in a deep fryer to obtain a range of TPC concentrations. In this study, the cooling thermograms of oil samples at a scanning rate of 1°C/min from −30 to −85°C showed a well-defined single crystallization peak. The study found that six DSC parameters, namely, peak temperature (PT), enthalpy (EN), onset (ON) and offset (OF) temperatures, peak height (HT), and the range of temperatures (RT) (the difference between onset and offset temperature) of this single crystallization peak could predict well the TPC of heated oils by using stepwise multiple linear regression analysis. These six parameters were used as independent variables while values from standard method were used as dependent variables. The coefficient of determination (R^2) of calibration models for CO, RBDPO, and SO were 0.9996, 0.9709, and 0.9980, respectively. Calibration models were validated with an independent set of samples. The R^2 of validation models were 0.9995, 0.9559, and 0.9961, respectively. Based on the results obtained, DSC appears to be useful instrumental method in determining the TPC of edible oils, and it may have the potential to replace the time- and chemical-consuming standard method.

Paper no. J9073 in *JAOCS 76*, 1047–1057 (September 1999).

KEY WORDS: Bleached and deodorized palm olein, corn oil, differential scanning calorimetry, heated oil, refined, soybean oil, total polar compounds.

Edible oils undergo a complex series of thermolytic and oxidative reactions during heating or frying processes (1). These reactions have been widely studied in recent decades by many researchers who have isolated and identified various degradation compounds in heated oils (2–4). More than 400 different chemical compounds have been identified in deteriorated heated oils (5). Generally, these compounds are of great interest and importance to the food industry and are of much concern for human health (6). Numerous analytical methods, such as free fatty acid content, iodine, peroxide, anisidine, and thiobarbituric acid values, have been described for the measurement of changes that occur in heated oils (7,8). Nowadays, these analytical methods and the knowledge of novel degradation compounds have advanced progressively. Therefore, in many countries, there are regulations that control the quality of oils used in heating or frying processes (9).

Recent research has indicated that the presence of polar compounds is one of the best indicators of heated oil quality (10). Regulatory agencies in European countries have established the level of 25–27% total polar compounds (TPC) as the rejection point for heated oils (9,11). By definition, TPC are those materials that remain on the silica gel column after the first elution when a heated oil is tested for polar compounds using AOCS Official Method Cd 20-91 (12). They include all nontriacylglycerols such as free fatty acids, mono- or diacylglycerols, glycerol, and polymers. Pokorný (13) demonstrated that increases in the polar compounds resulted in an eventual decline in fried-food quality. However, the main drawback with the used of polar compounds in regulatory and quality controls is that the standard chemical method is time-consuming (14). It may take 3.5 h to run one sample (1), and the analysis is chemical-intensive.

Edible oils, like other naturally occurring fats and oils, are composed of a complex mixture of 96 to 99% triacylglycerols. The nature of the fatty acid chains bound to glycerol and the wide range of fusion temperatures cause these triacylglycerol mixtures to have complex crystallization behaviors and thermal properties (15). When subjected to the crystallization conditions from the melted state, edible oils behave like high polymers, i.e., they pass through the stages of nucleation, activation, and crystal growth, and finally reach the final state of a crystal lattice (16). Much research has been conducted to investigate the crystallization behavior of various fat and oil products (16,17). According to Jacobsberg and Ho (18), factors influencing the crystallization of palm oil are: the presence of impurities such as free fatty acids, partial glycerides, and oxidation products. Since these impurities are known to increase during heating or frying processes, the crystallization behavior is particularly useful for predicting the degree of thermal degradation in the heated oil.

Instrumental methods have been developed in order to replace the analytical methods for online measurement of oil degradation in the food industry. These instrumental methods include the Fritest®, which is based on colorimetric measure-

*To whom correspondence should be addressed.
E-mail: yaakub@fsb.upm.edu.my

ment of carbonyl compounds formed during thermal oxidation, the Spot test, the Shortening Monitor test, which is based on calorimetric measurement of free fatty acid content as an indicator of hydrolytic rancidity, and the Food Oil Sensor, which is based on changes in the dielectric constant of heated oils (19). However, differential scanning calorimetry (DSC) techniques have the potential to be used as a nonchemical method of determining oil quality parameters. DSC analysis offers a direct method of studying the thermal properties of various materials (20). It is the most widely used of all thermoanalytical techniques with applicability to polymers, pharmaceuticals, fats, oils, and various inorganic materials (21). It provides unique energy profile information, which specifically measures the temperatures and heat flows associated with material transitions as a function of time and temperature. Such measurements provide quantitative and qualitative information about endothermic or exothermic processes. In the fats and oils industry, the DSC cooling/melting thermograms give valuable information on the thermal properties of various fats and oils products (20). A few applications of statistical and mathematical techniques used to determine various quality parameters for fats and oils, in DSC thermogram data can be found in the literature. They include the determination of the iodine value in palm-based products (22), the solid-fat index of oils (23), and an estimation of the amount of saturation present in transesterified blends, such as jojoba wax esters (24). However, current literature lacks any exploration into the possibility of applying DSC techniques in determining the quality parameters of heated oil. Therefore, the objective of this study was to develop the DSC technique for the determination of TPC in heated oils.

MATERIALS AND METHODS

Materials and treatments. Three different edible oils were used in this study. Commercial refined, bleached, and deodorized corn oil (CO), palm olein (RBDPO), and soybean oil (SO) were obtained from a local retailer [Ngo Chew Hong Oils and Fats (M) Ltd. Inc., Semenyih, Malaysia] and were used for all experiments. Oil samples (4.5 kg) were placed in open deep fryers (Berto's electrical fryer ALT series, ELT 8B, Montegrotto T., Padova, Italy) and heated at 180°C for a period of time, to obtain samples of increasing polar compound concentration. For each type of edible oil, a total of 42 samples were produced, with 30 assigned for use as a calibration set for method development, and 12 as a validation set. The concentration ranges of TPC for each edible oil are given in Table 1. The concentration range of the calibration set was chosen to model the likely range that is normally found in heated or frying oils. The validation samples were chosen such that their concentrations fell within the concentration range of the calibration set. All chemicals and solvents used were of analytical grade unless otherwise specified.

Chemical analysis. AOCS Official Method Cd 20-91 was used to determine the percentage of TPC in the oil samples (12).

Instrumental analysis. A Perkin Elmer differential scan-

TABLE 1
Concentrations of Polar Compounds in Edible Oils
Used for Calibration and Validation Analysis
(analyzed by standard chemical methods)[a]

	TPC (%)	
Samples	Calibration	Validation
CO	7.91–35.16	8.78–34.82
RBDPO	7.81–34.97	7.93–27.97
SO	7.21–40.50	7.63–33.01

[a]Abbreviations: TPC, total polar compounds; CO, corn oil; RBDPO, refined, bleached, and deodorized palm olein; SO, soybean oil.

ning calorimeter, DSC-7 (Perkin Elmer Corp., Norwalk, CT), equipped with a dry box, Intracooler II (liquid nitrogen cooling unit), TAC 7/DX thermal analysis instrument controller, and a Hewlett-Packard (Palo Alto, CA) DeskJet 660C printer, were used for the thermal analysis of the oil samples. Nitrogen (99.999% purity) was the purge gas and flowed at approximately 20 mL/min (20 psi). The DSC instrument was calibrated using indium (m.p. 156.6°C, ΔH_f = 28.45 J/g) and *n*-dodecane (m.p. −9.65°C, ΔH_f = 216.73 J/g). Samples of *ca.* 6–12 mg were weighed into aluminum pans to the nearest 0.1 mg, and covers were hermetically sealed into place. An empty, hermetically sealed aluminum pan was used as reference. Prior to analysis of samples, the baseline was obtained with an empty hermetically sealed aluminum pan. After the sample and reference pans were placed in the chamber at 30°C, the cell block of the DSC was heated rapidly to 80°C at 200°C/min and held for 5 min to destroy crystal nuclei, cooled rapidly at 200°C/min to −30°C and held for 5 min to allow for polymorphic changes and finally cooled at 1°C/min to −85°C to define the cooling profile. The cooling thermograms of the samples showed a single exothermic/crystallization peak. The following DSC parameters were extracted from each cooling thermogram: peak temperature (PK, °C), peak height in milliwatts (HT, mW), peak onset (ON) and offset (OF) temperatures (°C), range of temperature (RG, the difference between onset and offset temperature), and peak enthalpy of fusion (EN) in joules per gram (J/g). These parameters were calculated with the 7 Series/UNIX DSC software library (25). The construction and designation of these parameters are shown in Figure 1.

Statistical analysis. All measurements were replicated two times. The SAS/STAT (SAS, Cary, NC) release 6.08 program was used for stepwise multiple linear regression (SMLR) analysis (26). The significance level of an independent variable for entry and stay in the calibration model was set to 0.15 during execution of the stepwise variable selection in SAS procedure "REG".

RESULTS AND DISCUSSION

The principle technique of DSC is to compare the rate of heat flow to the sample and to reference materials that are heated or cooled at the same rate. This technique measures net changes in enthalpy and weight as a result of the many reac-
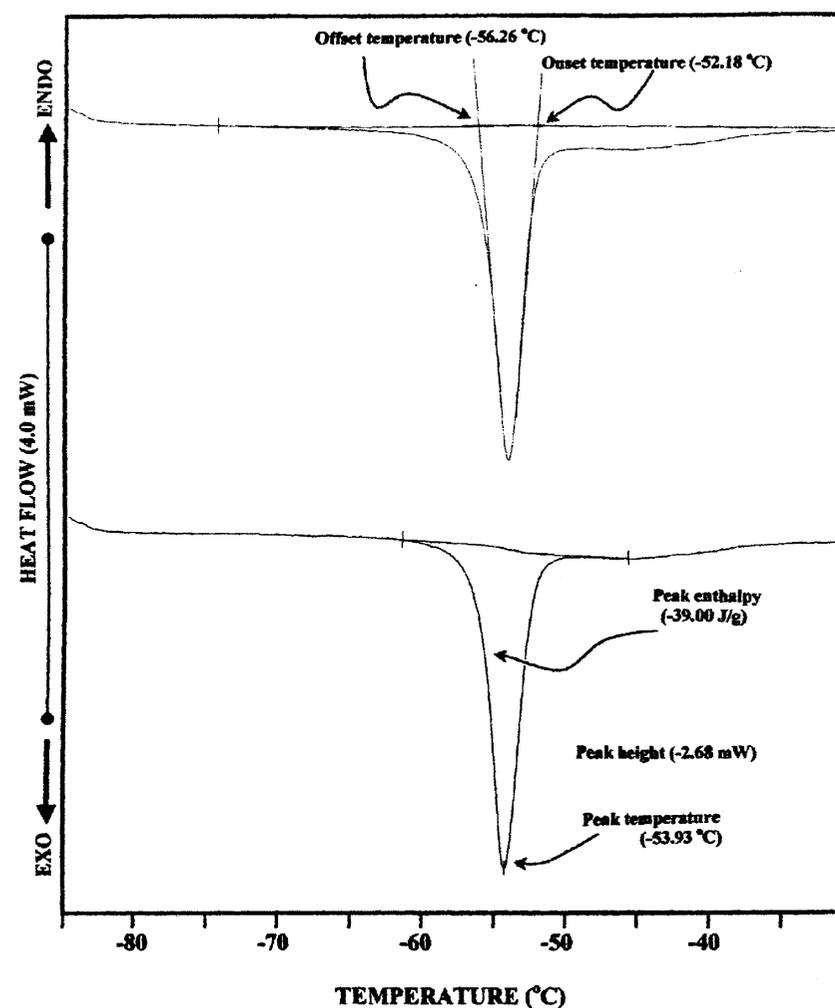
FIG. 1. Differential scanning calorimetry (DSC) cooling thermograms of corn oil. The techniques used for determination of the DSC parameters are illustrated by construction lines. (a) The onset and offset temperatures corresponded closely to the intersection of the extrapolated baseline and the tangent line (leading edge) of the peak. (b) A sigmoidal/curved baseline is constructed before the calculation of peak temperature, enthalpy and height by using 7 Series/UNIX software library (Perkin Elmer Corp., Norwalk, CT).

tions taking place simultaneously and is particularly useful for indicating the temperature range and the rate of thermal processes as well as giving considerable information on physical and chemical changes. Changes in the sample that are associated with the absorption or evolution of heat cause a change in the different heat flow, which is then recorded as a peak. Naturally occurring fats and oils, such as an edible oil composed of a wide variety of triacylglycerols, melt/crystallize over a wide range of temperatures (27). Thermal properties of edible oils are closely related to those of triacylglycerols. In the heating thermogram of edible oil, complex features were not easily interpretable. This is a consequence of the known phenomenon of polymorphism of fats and oils that is strongly dependent on the thermal history of the sample. Therefore, we decided to focus our attention on the cooling thermograms of the oils, which were influenced only by the chemical composition of the sample and not by the initial crystalline state as the analysis is initiated from a melted stage. We devised statistical and mathematical indices based on DSC cooling thermogram parameters to define the level of TPC in edible oils.

In this study, heated oils exhibited a simple thermogram after cooling in the DSC with a well-defined single crystallization peak as shown in Figures 2, 3, and 4 for CO, RBDPO, and SO, respectively. From these figures, it is apparent that the DSC traces are affected in a systematic way by the increasing TPC level in heated oils. In general, as the concentration of polar compounds increased, the peak of crystallization shifted to lower temperatures and became broader (an increase in the peak temperature range). At the same time, the
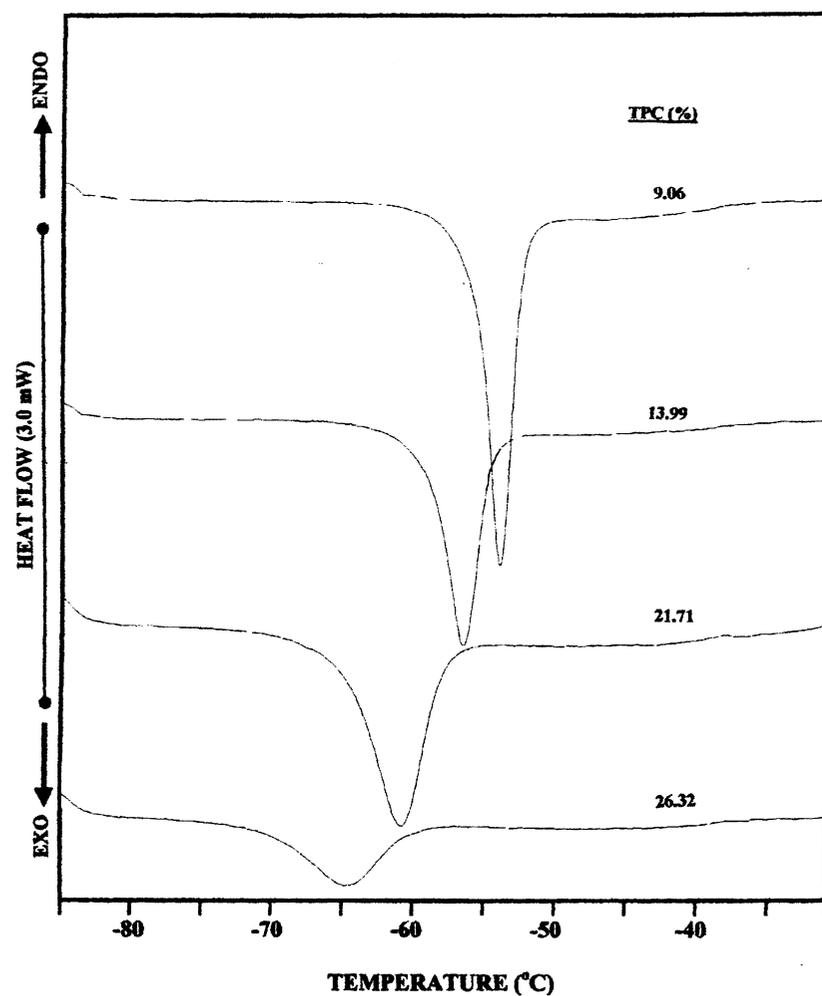
FIG. 2. Cooling thermograms of corn oil samples with different concentrations (percentage) of total polar compounds (TPC).

peak height and enthalpy decreased dramatically. Consequently, the ON and OF were set at a lower temperature. According to Hagemann and Tallent (28), slow-scan cooling curves of unsaturated triacylglycerols generally showed an exotherm at approximately the melting point of the α-form. Berger and Akehurst (29) also reported that the typical cooling thermogram of the edible oil shows a dominant exothermic peak at supercooling temperature range. Since the contents of unsaturated fatty acids were higher in CO and SO in comparison to RBDPO, the crystallization peaks in CO and SO were more apparent than RBDPO.

In heated oils, the amount of polar compounds increased during the heating process. As this level increased, the compounds contributed to the changes in DSC crystallization peak parameters. It is well known that the presence of free fatty acids, partial glycerides, and oxidation products in oil tends to shift the melting range to lower temperatures (30). The same

phenomenon is expected for the changes in DSC crystallization peak parameters. Further study on the crystallization behavior of polar and nonpolar fraction of heated oils derived evidence supportive of this phenomenon. In Figure 5 (A, B, and C), the result showed that the crystallization peak was not observed in the polar fraction of heated oils. This may be attributed to the disappearance of unsaturated triacylglycerols and the formation of degradation products in polar fraction.

The crystallization peak analyses enable the determination of the TPC level in a heated oil sample. From each sample, six DSC parameters (PK, HT, ON, OF, RG, and EN) were derived from the single crystallization peak. These six parameters served as independent variables in the SMLR analysis, with TPC obtained from standard chemical method as the dependent variable. To build a DSC method for TPC determination, 30 samples were used as the calibration set for CO, RBDPO, and SO, respectively. In CO samples, one out of the
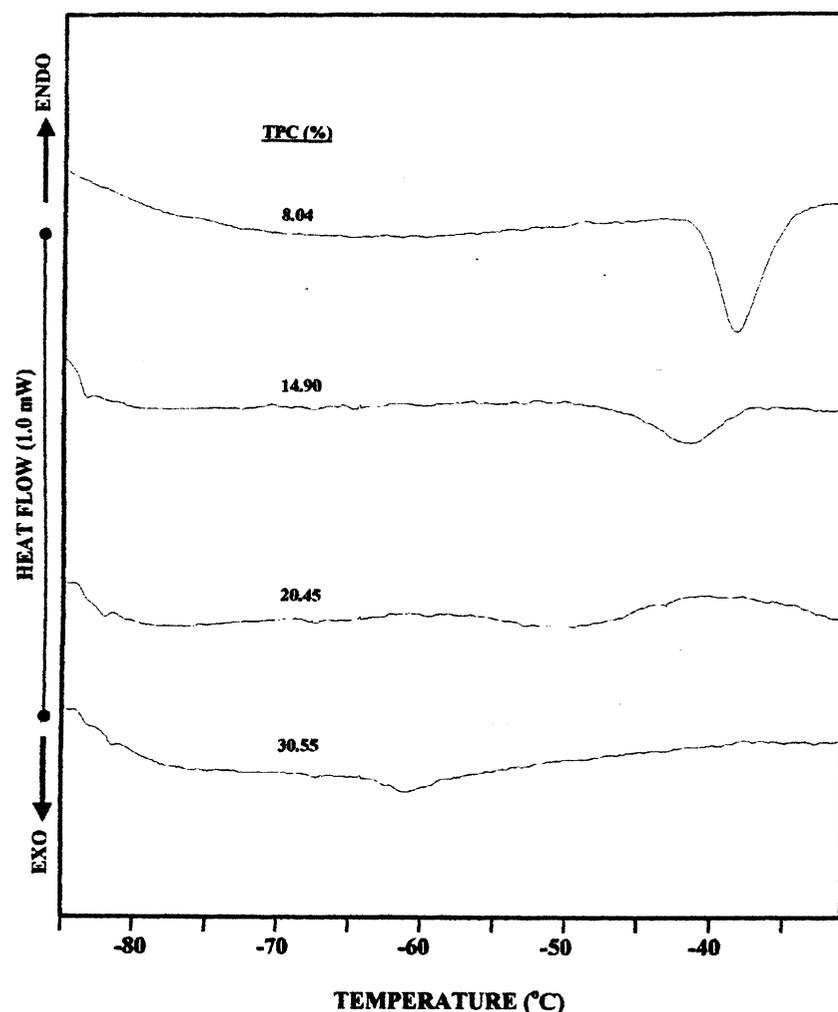
FIG. 3. Cooling thermograms of refined, bleached, and deodorized palm olein (RBDPO) samples with different concentrations (percentage) of TPC. See Figure 2 for abbreviation.

30 samples exhibited comparatively large residuals as indicated by Cook's distance measure in the model selected by stepwise selection procedure (31). This sample may therefore influence greatly the regression parameter estimates. Indeed, exclusion of the sample improved the fit of the models slightly but was, conversely, not advantageous for predictions made on the validation data set. Therefore, the unmodified regression equation was taken for further calculations. The summary of the SMLR analyses for CO, RBDPO, and SO are presented in Tables 2, 3, and 4, respectively.

The SMLR analysis showed that from six independent variables, only five, four, and three variables were necessary to predict the TPC in CO, RBDPO, and SO, respectively. The SMLR analysis was superior in predicting the TPC in these heated oils, as judged by the coefficient of determination (R^2) values. The regression models used to predict TPC by DSC as based on the highest R^2 value and smallest SE of calibration are shown in the following equations:

$$TPC = -113.2691 - 2.2635\ PK - 0.2366\ EN$$
$$+ 3.0528\ HT - 1.3669\ ON + 1.3012\ OF \qquad [1]$$

with $R^2 = 0.9996$ and SE $= 0.1615\%$;

$$TPC = 4.0944 - 0.2211\ PK + 1.6828\ EN$$
$$- 16.5013\ HT + 0.5036\ RG \qquad [2]$$

with $R^2 = 0.9709$ and SE $= 1.3887\%$;

$$TPC = -8.1615 + 0.3792\ EN + 0.6617\ RG - 0.4563\ OF \qquad [3]$$

with $R^2 = 0.9980$ and SE $= 0.4595\%$, where Equations 1, 2, and 3 are for CO, RBDPO, and SO, respectively.

The effectiveness of the regression models for future prediction was checked by means of the validation data set. Each validation set contained 12 samples that were not represented in the calibration data. No outlier was observed from any of the validation data sets. The outcomes of the validation tests are summarized in Table 5. As expected from the results of the calibration data, predictions made on the validation data
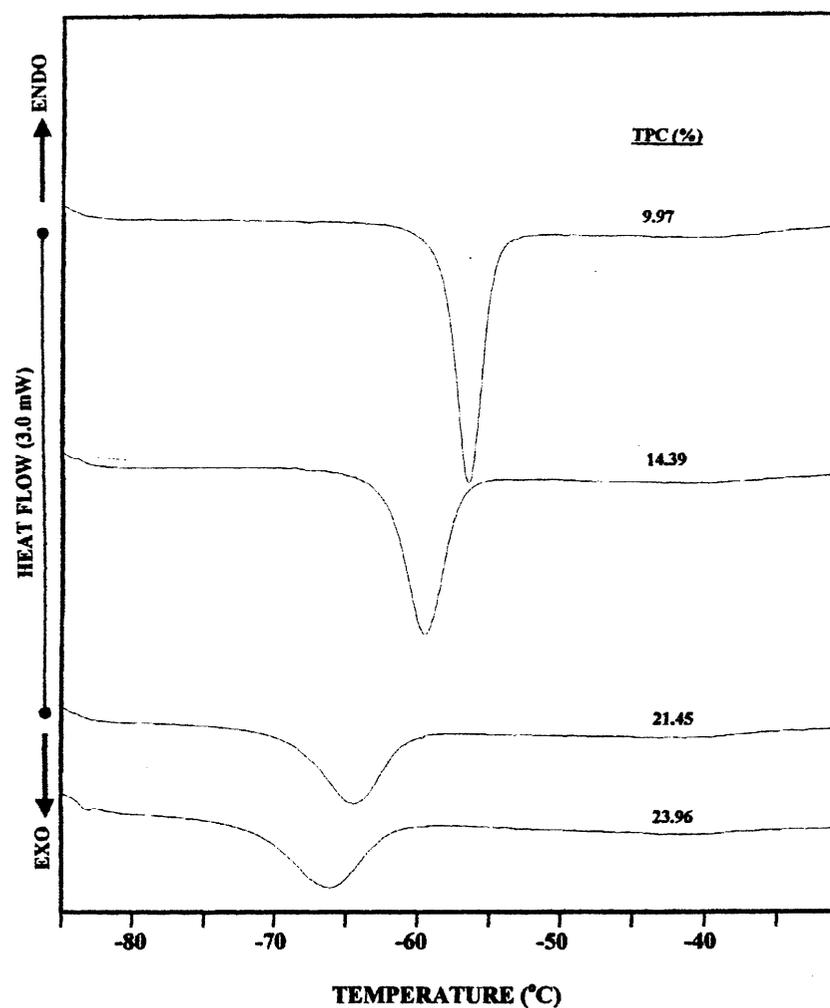
FIG. 4. Cooling thermograms of soybean oil samples with different concentration (percentage) of TPC. For abbreviation see Figure 2.

TABLE 2
Summary of Stepwise Regression Analysis with DSC Crystallization Peak Parameters as Independent Variables (Corn Oil)[a]

Step	Regression equation	R^2
1	TPC = −95.3161 − 2.0144 ON	0.9488
2	TPC = −78.8958 − 0.4537 PK − 1.2423 ON	0.9596
3	TPC = −122.5095 − 2.2429 PK − 1.8114 ON + 1.4911 OF	0.9958
4	TPC = −98.6327 − 2.1121 PK + 1.7626 HT − 1.4115 ON + 1.3597 OF	0.9984
5	TPC = −113.2691 − 2.2635 PK − 0.2366 EN + 3.0528 HT − 1.3669 ON + 1.3012 OF	0.9996

[a]Abbreviations: EN, peak enthalpy; HT, peak height; OF, offset temperature; ON, onset temperature; PK, peak temperature; R^2, coefficient of determination; DSC, differential scanning calorimetry; for other abbreviation see Table 1.

TABLE 3
Summary of Stepwise Regression Analysis with DSC Crystallization Peak Parameters as Independent Variables (RBDPO)[a]

Step	Regression equation	R^2
1	TPC = −24.3898 − 0.8952 PK	0.9388
2	TPC = −9.1407 − 0.6699 PK + 1.3542 EN	0.9558
3	TPC = 1.4341 − 0.3054 PK + 1.2167 EN + 0.4087 RG	0.9678
4	TPC = 4.0944 − 0.2211 PK + 1.6828 EN −16.5013 HT + 0.5036 RG	0.9709

[a]RG, temperature range (difference between ON and OF). For other abbreviations see Tables 1 and 2.

TABLE 4
Summary of Stepwise Regression Analysis with DSC Crystallization Peak Parameters as Independent Variables (Soybean Oil)[a]

Step	Regression equation	R^2
1	TPC = −62.7941 − 1.2294 OF	0.9919
2	TPC = −42.3597 + 0.6459 RG − 0.8466 OF	0.9961
3	TPC = −8.1615 + 0.3792 EN + 0.6617 RG − 0.4563 OF	0.9980

[a]See Tables 1–3 for abbreviations.

TABLE 5
Summary of Regression Analysis on Validation Data[a]

Samples	Regression equation	R^2
CO	$TPC_A = 1.0815\ TPC_p − 0.4561$	0.9995
RBDPO	$TPC_A = 0.9628\ TPC_p + 0.6647$	0.9559
SO	$TPC_A = 1.0320\ TPC_p − 0.3696$	0.9961

[a]TPC_A, actual total polar compounds (standard chemical method); TPC_P, predicted total polar compounds (DSC method). See Tables 1 and 2 for other abbreviations.
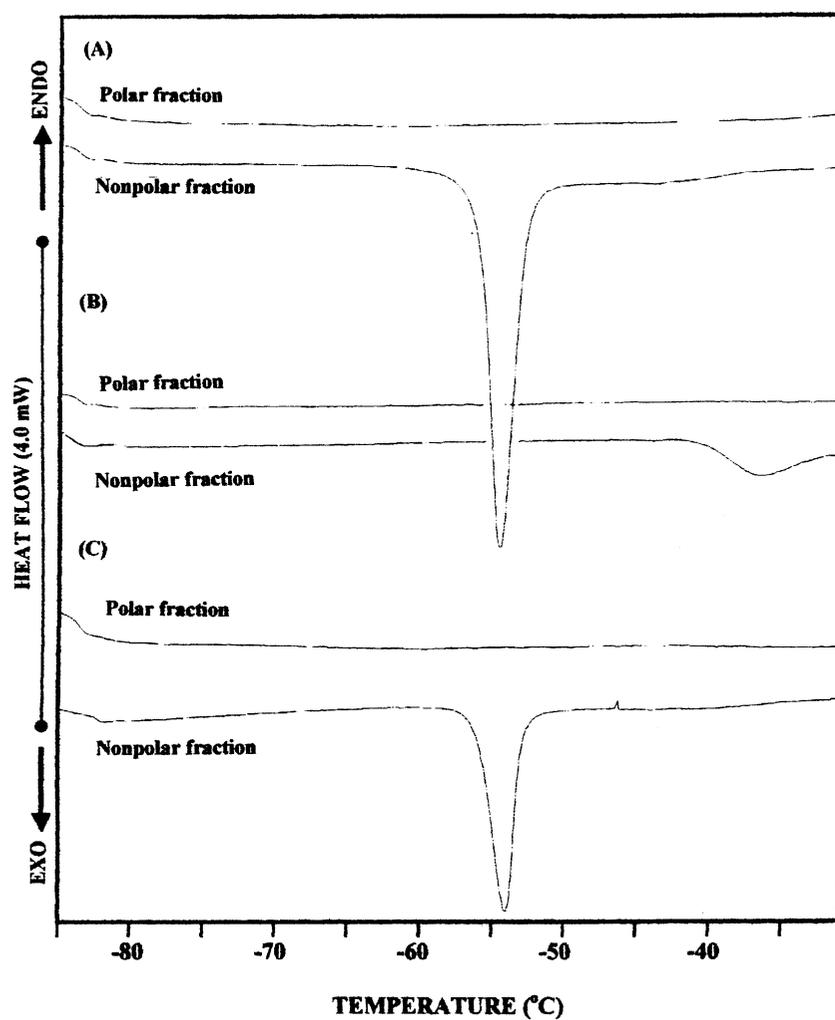
FIG. 5. Cooling thermograms of polar and nonpolar fraction of (A) corn oil sample with 23.84% TPC; (B) RBDPO sample with 19.97% TPC; and (C) soybean oil with 25.82% TPC. See Figures 2 and 3 for abbreviations.
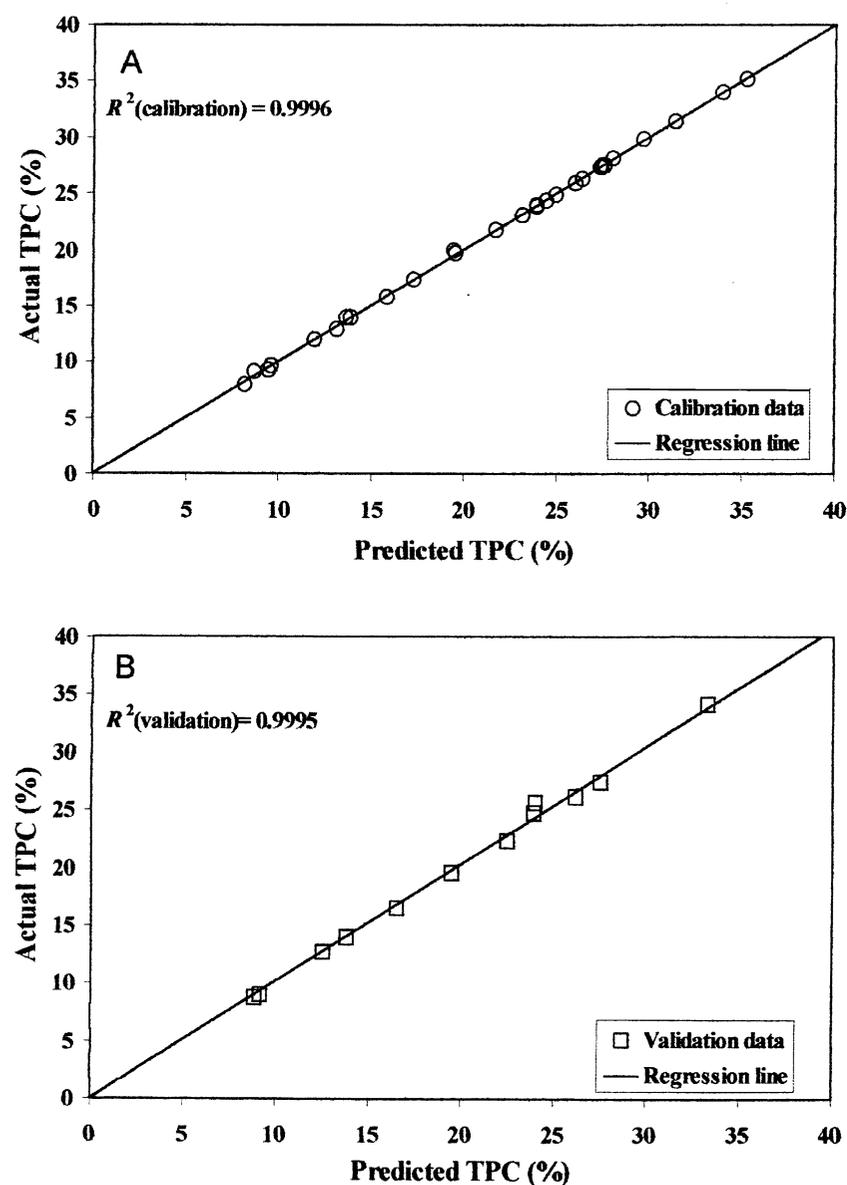
FIG. 6. Scatter plot showing calibration (A) and validation (B) data of actual vs. predicted TPC percentage of corn oil. Each value represents the mean of two independent determinations. R^2, coefficient of determination; for other abbreviation see Figure 2.

were also superior for CO, RBDPO, and SO, respectively. For these three different types of edible oil, the R^2 values for validation samples were close to those of calibration samples. The corresponding SE of validation are 0.5850, 1.4119 and 0.4898% for CO, RBDPO, and SO, respectively. The SE of validation was higher than the SE of calibration in these edible oils.

Figures 6, 7, and 8 illustrate the correlation plot of the actual TPC vs. the predicted TPC for CO, RBDPO, and SO, respectively. In these figures, the correlation plot is presented for the calibration and validation data set, respectively. For CO, RBDPO, and SO samples, the slope of regression line and y-intercept for calibration data are 1.07 and −1.07%, 0.9628 and 0.6647%, 1.0185 and 0.4561%, respectively (Fig. 6A, 7A, and 8A). These results demonstrate the closeness of DSC and standard chemical methods. For the validation set, the correlation between the actual TPC and predicted TPC when the calibration models are used is illustrated in Figures 6B, 7B, and 8B, respectively. The slope and the y-intercept of regression analysis are 1.0185 and −4.4561%, 0.9628 and 0.6647%, and 1.0320 and −0.3696%, respectively. These studies indicate that TPC predicted by DSC method is linearly correlated to the actual TPC obtained by standard chemical methods throughout the ranges tested for CO, RBDPO, and SO, respectively. The DSC
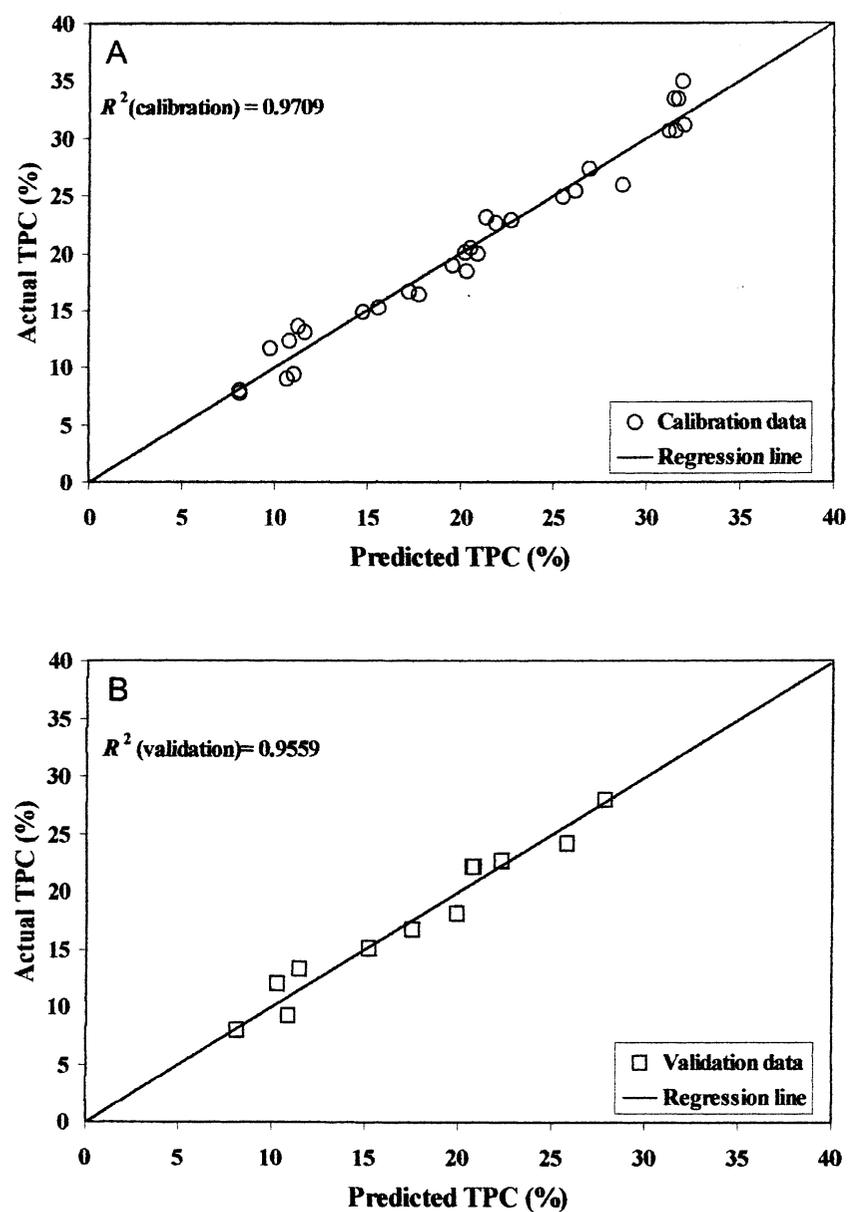
FIG. 7. Scatter plot showing calibration (A) and validation (B) data of actual vs. predicted TPC in percentage of RBDPO. Each value represents the mean of two independent determinations. For abbreviations see Figures 3, 5, and 6.

method provides accurate results that closely approach the accuracy limit of standard chemical methods in the determination of TPC. Table 6 summarizes all data for this study in terms of mean differences (MD) and standard deviations of differences (SDD) for both reproducibility and accuracy to compare the overall performance of these two methods. For each type of edible oil, data comparison between two methods by a one-way analysis showed that there were no significant differences ($P > 0.01$) between the two data sets. The results obtained allow us to conclude that the DSC method can be a useful technique which may replace the AOCS method for determining TPC in CO, RBDPO, and SO.

The use of DSC, as presented here, may offer a quick, accurate, and routine technique to determine TPC. This bodes well for the wider application of DSC in quality control. The speed and ease of data manipulation brought about by computer control during this study represent the further benefits of the DSC method. It is especially attractive to develop a DSC method so that the tedious time- and chemical-consuming standard chemical method can be avoided in future routine testing. With some extra work during calibration for different edible oils, this time-saving method can be carried forward in the form of an easy-to-use DSC method.
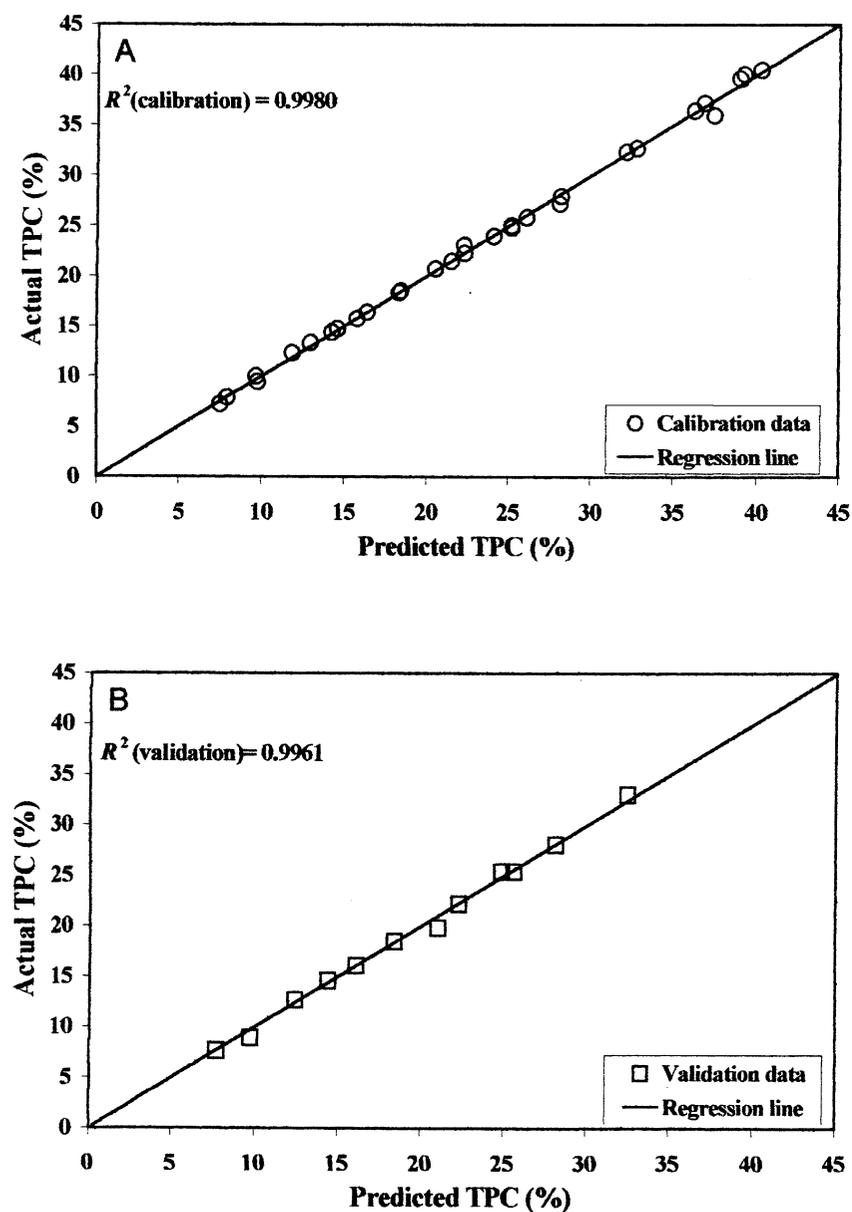
FIG. 8. Scatter plot showing calibration (A) and validation (B) data of actual vs. predicted TPC in percentage for soybean oil. Each value represents the mean of two independent determinations. For abbreviations see Figure 6.

TABLE 6
Reproducibility and Accuracy Data for Total Polar Compounds Determination by the DSC and AOCS Official Methods[a]

Statistic	CO		RBDPO		SO	
	AOCS official method	DSC method	AOCS official method	DSC method	AOCS official method	DSC method
Mean	21.2603	21.1838	19.6003	19.5973	22.0789	22.1066
MD_r	−0.3176	0.2095	−0.2785	−0.2517	−0.1601	−0.2357
SDD_r	0.0800	0.1996	−0.2184	0.2622	0.2913	0.0190
MD_a		0.0765		0.0030		−0.0276
SDD_a		0.1608		0.0569		0.0401

[a]MD, mean difference; SDD, standard deviation of the difference; r, reproducibility; a, accuracy. For other abbreviations see Table 1.

ACKNOWLEDGMENTS

REFERENCES

1. White, P.J., Methods for Measuring Changes in Deep-Fat Frying Oils, *Food Technol. 45*:75–80 (1991).
2. Chung, T.Y., J.P. Eiserich, and T. Shibamoto, Volatile Compounds Identified in Headspace Samples of Peanut Oil Heated under Temperatures Ranging from 50 to 200°C, *J. Agric. Food Chem. 41*:1467–1470 (1993).
3. Wu, C.M., and S.Y. Chen, Volatile Compounds in Oils After Deep Frying or Stir-Frying and Subsequent Storage, *J. Am. Oil Chem. Soc. 69*:858–865 (1992).
4. Gardner, D.R., R.A. Sanders, D.E. Henry, D.H. Tallmadge, and H.W. Wharton, Characterization of Used Frying Oils. Part 1: Isolation and Identification of Compound Classes, *Ibid. 69*:499–508 (1992)
5. Gere, A., Study of the Changes in Edible Fats During Heating and Frying, *Die Nahrung 26*:923–926 (1982).
6. Clark, W.L., and G.W. Serbia, Safety Aspects of Frying Fats and Oils, *Food Technol. 45*(2):84–86,88,89,94 (1991).
7. Robards, K., A.F. Kerr, and E. Patsalides, Rancidity and Its Measurement in Edible Oils and Snack Foods, *Analyst 113*:213–222 (1988).
8. Fritsch, C.W., Measurements of Frying Fat Deterioration: A Brief Review, *J. Am. Oil Chem. Soc. 58*:272–274 (1981).
9. Paul, S., and G.S. Mittal, Regulating the Use of Degraded Oil/Fat in Deep-Fat/Oil Food Frying, *Crit. Rev. Food Sci. Nutr. 37*:635–662 (1997).
10. Blumenthal, M.M., A New Look at the Chemistry and Physics of Deep-Fat Frying, *Food Technol. 45*(2):68–71,72 (1991).
11. Firestone, D., R.F. Stier, and M.M. Blumenthal, Regulation of Frying Fats and Oils, *Ibid. 45*(2):90–94 (1991).
12. AOCS, *Official Methods and Recommended Practices of the American Oil Chemists' Society*, 4th edn., AOCS Press, Champaign, 1993, Method Cd 20–91.
13. Pokorný, J., Flavor Chemistry of Deep Fat Frying in Oil, in *Flavor Chemistry of Lipid Foods*, edited by D.B. Min, and T.H. Smouse, American Oil Chemists' Society, Champaign, 1989, pp. 113–155.
14. Blumenthal, M.M., Frying Technology, in *Bailey's Industrial Oil and Fat Products*. 5th edn., edited by Y.H. Hui, John Wiley & Sons, Inc., New York, Vol. 3 1996, pp. 429–482.
15. Herrera, M.L. and M.C. Anon, Crystalline Fractionation of Hydrogenated Sunflowerseed Oil. II. Differential Scanning Calorimetry (DSC), *J. Am. Oil Chem. Soc. 68*:799–803 (1991).
16. Kawamura, K., The DSC Thermal Analysis of Crystallization Behavior in Palm Oil, *Ibid. 56*:753–758 (1979).
17. Md. Ali, A.R., and P.S. Dimick, Thermal Analysis of Palm Mid-Fraction, Cocoa Butter, and Milk Fat Blends by Differential Scanning Calorimetry, *Ibid. 71*:299–302 (1994).
18. Jacobsberg, B., and O.C. Ho, Studies in Palm Oil Crystallization, *Ibid. 71*:609–617 (1976).
19. Melton, S.L., S. Jafar, D. Sykes, and M.K. Trigiano, Review of Stability Measurements for Frying Oils and Fried Food Flavor, *Ibid. 71*:1301–1308 (1994).
20. Biliaderis, C.G., Differential Scanning Calorimetry in Food Research—A Review, *Food Chem. 10*:239–265 (1983).
21. Dollimore, D., Thermal Analysis, *Anal. Chem. 68*:63R–71R (1996).
22. Haryati, T., Y.B. Che Man, A.B. Asbi, H.M. Ghazali, and L. Buana, Determination of Iodine Value of Palm Oil by Differential Scanning Calorimetry, *J. Am. Oil Chem. Soc. 74*:939–942 (1997).
23. Walker, R.C., and W.A. Bosin, Comparison of SFI, DSC, and NMR Methods for Determining Solid–Liquid Ratios in Fats, *Ibid. 48*:50–53 (1971).
24. Sessa, D.J., T.C. Nelsen, R. Kleiman, and J.D. Arquette, Differential Scanning Calorimetry Index for Estimating Level of Saturation in Transesterified Wax Esters, *Ibid. 48*:271–273 (1996).
25. *7 Series/UNIX DSC7 Users Manual*, Version 4.0, Perkin Elmer Corporation, Norwalk, CT, 1995.
26. SAS, *Statistical Analysis System User's Guide: Basic Statistics*, SAS Institute, Cary, 1989.
27. Barbano, P., and J.W. Sherbon, Phase Behavior of Tristearin/Trioctanoin Mixtures, *J. Am. Oil Chem. Soc. 55*:478–481 (1978).
28. Hagemann, J.W., and W.H. Tallent, Differential Scanning Calorimetry of Single Acid Triglycerides: Effect of Chain Length and Unsaturation, *Ibid. 49*:119–123 (1972).
29. Berger, K.G., and E.E. Akehurst, Some Application of Differential Thermal Analysis to Oils and Fats, *J. Food Technol. 1*:237–247 (1966).
30. Che Man, Y.B., and P.Z. Swe, Thermal Analysis of Failed-Batch Palm Oil by Differential Scanning Calorimetry, *J. Am. Oil Chem. Soc. 72*:1529–1532 (1995).
31. Miller, J.N., Outliers in Experimental Data and Their Treatment, *Analyst 118*:455–461 (1993).