

## Method for Determining Frying Oil Degradation by Near-Infrared Spectroscopy

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A rapid near-infrared (NIR) spectroscopic method for measuring degradation products in frying oils, including total polar materials (TPMs) and free fatty acids (FFAs), has been developed. Calibration models were developed using both forward stepwise multiple linear regression (FSMLR) and partial least-squares (PLS) regression techniques and then tested with two independent sets of validation samples. Derivative treatments had limited usefulness, especially in the longer (1100–2500 nm) wavelength region. When using a wavelength region of 700–1100 nm, PLS models gave improved results compared to FSMLR models. The best correlations ( $r$ ) between the NIR and wet chemical methods for TPM and FFA were 0.983 and 0.943, respectively. In the longer wavelength region (1100–2500 nm), FSMLR models were as good or better than PLS models. The best correlations for TPM and FFA obtained in this region were 0.999 and 0.983, respectively.

**KEYWORDS:** NIR; free fatty acids; total polar materials; forward stepwise multiple linear regression (FSMLR); partial least-squares (PLS) regression; frying oil quality

### INTRODUCTION

Frying is an established, inexpensive, rapid, and easy method of food preparation. For decades, consumers have desired deep-fat-fried products because of their unique flavor and texture combination. Products range from potato chips, French fried potatoes, doughnuts, extruded snacks, and fish sticks to traditional fried chicken products. Frying is used in homes, restaurants, and various segments of the food industry. In the United States, more than 500000 institutions and commercial restaurants use deep-fat frying (1).

Frying oil, used continuously and repeatedly at high temperature, is subject to a series of degradation reactions. The amount of degradation products increases with increased duration of heating at high temperatures. More than 400 different chemical compounds, including 220 volatile products, have been identified in deteriorated frying oil (2). These degradation products have a negative effect on the flavor and nutritional value of fried products. It is now a well-established fact that many degradation products of frying oil are harmful to human health because they destroy vitamins, inhibit enzymes, and can potentially cause mutations and gastrointestinal irritations (3). Also, Flickinger et al. (4) reported that cyclic fatty acid monomers led to significant reductions in  $\text{Ca}^{2+}$  ATPase activity and monolayer integrity in cultured porcine endothelial cells.

Measurement of oil degradation should be based on the changes after frying. Chemical analyses, including measurements of free fatty acid (FFA) content and total polar materials (TPMs), are reliable ways to measure degradation compounds

in the frying oil. However, these chemical analyses are often time-consuming, costly, and destructive to the sample. In addition, they require potentially hazardous reagents and also require reasonable analytical expertise. Because of these shortcomings, spectroscopic methods have been investigated as alternatives to chemical methods. Goburdhun et al. (5) monitored changes in soybean frying oil quality over time by measuring peroxide, iodine, and free fatty acid values and also collected mid-infrared transmission spectra of the oils. The authors found that hydroperoxides and free fatty acids increased in the oil over time and also that a decrease in the triacylglycerol ester linkage could be observed over time in the mid-IR spectra. The IR spectra also showed a loss of *cis* double bonds coupled with an increase in *trans* unsaturation. Innawong et al. (6) used Fourier transform mid-infrared spectroscopy, coupled with attenuated total reflectance sampling and principal component analysis, to classify restaurant frying oils as new, used, or degraded.

It may also be possible to monitor frying oil degradation using the technique of near-infrared (NIR) spectroscopy. Boot and Speek (7), using transmittance measurements obtained with a filter-based spectrometer, successfully screened frying oil samples for acid value and for dimer and polymer triglycerides (DPTGs). Stronger correlations were found for acid value than for DPTG. However, when Engelsen (8) used NIR/visible spectroscopy to predict degradation parameters including DPTG and FFA in frying oils, the most significant wavelengths corresponded to background scatter and blue-green color. This indicated that indirect correlations to color and amount of dispersed material were actually being measured. In more recent work, Kazemi et al. (9) obtained correlations  $>0.9$  for acid value, total polar components, and viscosity using a limited

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number of canola oil samples. Büning-Pfaue and Kehraus (10) obtained NIR transmittance measurements from used deep-frying oils and developed models to predict various measures of degradation, including acid value and total polar materials. Strong correlations ( $>0.98$ ) were obtained, but sample sets were again quite small.

If successful, NIR spectroscopy could be useful as a quantitative technique because it is relatively fast, accurate, and nondestructive. Also, little to no sample preparation is required. In addition, NIR analysis is very economical because labor requirements are low, reagents are not required, and no chemical wastes are produced.

The objectives of the present research were (1) to develop NIR methods for measuring TPM and FFA in degraded soy-based frying oil, (2) to evaluate different sample handling and data treatment techniques to maximize the correlation between spectral and chemical data, and (3) to evaluate the success of the methods developed by testing each with two independent sets of degraded frying oil samples.

## MATERIALS AND METHODS

**Sample Preparation.** Samples of FryMax 3500, a commercial partially hydrogenated soy-based frying oil distributed by ACH Food Co. Inc. (Memphis, TN), were obtained from a local Sam's Club store or from a local food wholesale distributor (Cash-Wa Distributing). Different levels of degradation were generated by heating the oils for 8 h per day at approximately 190 °C in two Presto (model no. 0600308) fryers, with water added by a metering pump at approximately 2% by weight per hour compared to the oil. A total of three different lots of FryMax frying oils were purchased on different days to encompass possible manufacturing variations that may occur over time. The first 2 lots of oil were used for the first 4 batches of heating that generated 103 samples. After several weeks, the third lot of oil was used to generate an additional external validation set that contained 28 samples from a fifth batch of heating.

Oil samples for chemical and spectroscopic analysis were collected at 4 h intervals during heating. In this study, TPM and FFA were used to measure oil degradation, because they are widely used in today's food industry as reliable chemical parameters for determining degradation of frying oils (11–13). Chemical analysis for FFA was performed each day on the samples collected. Samples for spectroscopic and TPM analyses were stored in brown glass bottles under nitrogen and kept in a freezer at –8 °C until analyzed.

**Reference Chemical Methods.** All chemical analyses were carried out in duplicate for each sample under the same conditions, and the means were calculated. Free fatty acid contents were determined by titration with standard 0.01 N NaOH and phenolphthalein using AOCS Official Method Ca 5a-40 (14).

Total polar materials were determined by solid-phase extraction, using a modified method based on the work of Bheemreddy et al. (15). TPM was defined as the amount of material remaining on a 500 mg silica column after elution of a 0.5 g sample with 50 mL of a 90:10 mixture of petroleum and diethyl ethers. First, a 500 mg silica SPE cartridge (Alltech, part no. 22935) was conditioned with 10 mL of elution solvent, a 90:10 mixture of petroleum, and diethyl ethers. Then a 0.5 g oil sample was dissolved in 5 mL of elution solvent. Next, the sample solution was transferred to the cartridge and then eluted with 50 mL of elution solvent and the eluent collected in a tared 125 mL round-bottom flask using a vacuum manifold. Then the solvent was evaporated from the eluent with a vacuum evaporator at 8 °C. After that, the final traces of solvent were removed in a vacuum oven at 60 °C for 60 min. Finally, the flask containing the nonpolar fraction eluted from the cartridge was weighed. The content of polar compounds (%) was calculated using the formula

$$\text{total polar materials (\%)} = \{(m - m_1)/m\} \times 100$$

where  $m_1$  = mass of the nonpolar fraction (g) and  $m$  = mass of the sample (g).

**Near-Infrared Spectroscopy.** Spectroscopic data were collected with a Foss NIRSystems model 6500 scanning spectrometer (NIRSystems Division of Foss Electric, Silver Spring, MD). NIRSystems Near Infrared Spectral Analysis Software (NSAS, version 3.53; Foss NIRSystems, Inc.), a commercial NIR software package, was used in this study to collect, store, and manipulate data. Spectroscopic data were collected from each sample as direct transmission measurements over a 400–2500 nm range, using a quartz cuvette with a 2 mm path length.

**Collection of Spectral Data.** To minimize temperature effects on spectral response (16), spectra of all samples were collected under the same conditions. All samples were heated to 60 °C in a water bath prior to collection of spectra to liquefy the samples, as these samples had a melting point around 54 °C. Then each oil sample was placed into a 2 mm quartz cuvette, which was inserted into the cuvette holder in the instrument. A spectrum was obtained by immediately collecting and averaging 32 individual spectral scans.

**Calibration Development and Validation.** The 103 samples generated from the first 2 lots of oil were divided into calibration and validation sets by NSAS, using random selection. A total of 60 samples were chosen for the calibration set, and the remaining 43 samples were placed in a validation set (V1). The calibration samples were inspected to make sure that the extremes of TPM and FFA values were included. To further ensure validity of the calibration models, an additional external validation set of 28 samples (V2) was generated from a third lot of oil.

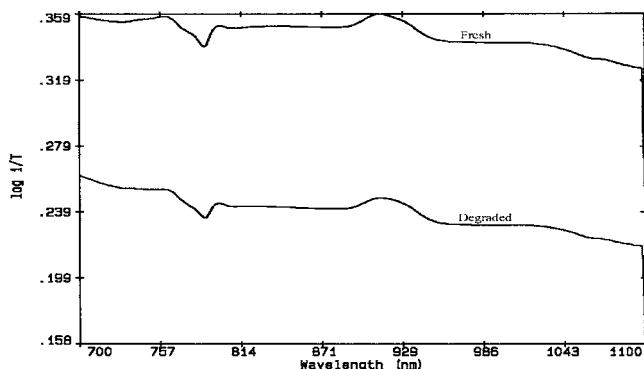
Calibration models were created by NSAS using forward stepwise multiple linear regression (FSMLR) and partial least-squares (PLS) regression. Because of the possibility of improved results, the spectra were also derivatized prior to calibration development, with parameters of wavelength gap and segment length optimized. A subroutine in NSAS was used to choose the optimum wavelength gap and segment length. Models were then developed from the  $\log(1/T)$  spectra, as well as first and second derivatives of  $\log(1/T)$ , where  $T$  is the transmittance of a sample at a specific wavelength. The correlation coefficient of calibration ( $R$ ), standard error of calibration (SEC), and mean square error of cross-validation (MSECV), for PLS, were used to determine the optimal number of wavelengths (FSMLR) or terms (PLS) to be included in a calibration model. A maximum of four wavelengths were used in the FSMLR models to prevent overfitting of the models.

Each generated model was validated with the V1 samples that were not used in calibration development and also validated with the external V2 samples. The correlation coefficient of prediction ( $r$ ) and root-mean-square of the differences (rmsd) were used to choose the best model; rmsd measures how well the model can predict samples in a validation set. The best calibration model to be used for prediction is the one with the highest  $r$  and lowest rmsd.

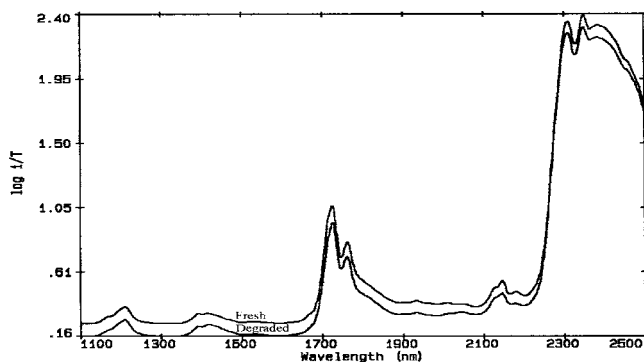
## RESULTS

**Frying Oil.** A total of three different lot numbers of FryMax frying oils were purchased. Before these frying oils were heated, their FFA and TPM values as determined by the reference methods ranged from 0.015% to 0.020% and from 2.18% to 3.84%, respectively.

**NIR Spectra.** Figures 1 and 2 show the near-infrared spectra of fresh and highly degraded soybean-based frying oil samples, in different wavelength ranges. Bands  $>2300$  nm appear distorted due to the extremely strong absorptions in this region. Osborne et al. (17) indicated that the major NIR absorption bands in a fat or oil are centered at 1200 nm for a  $\text{CH}_2$  second overtone, 1734 and 1765 nm for  $\text{CH}_2$  first overtones, and 2310 and 2345 nm for  $\text{CH}_2$  stretch–bend combinations. Besides the major absorption bands, absorptions at around 1700 nm are due to first overtones of C–H stretching vibrations, and absorptions at 1400, 2072, and 2078 nm are due to the first overtones of O–H stretches and combinations of O–H stretching and deformation. Osborne et al. (17) also indicated that absorption bands at 2140 and 2190 nm are due to *cis*-unsaturated fatty acid units and can be used to estimate the degree of unsaturation



**Figure 1.** Near-infrared spectra of fresh and highly degraded soybean-based frying oil samples in the shorter wavelength region.



**Figure 2.** Near-infrared spectra of fresh and highly degraded soybean-based frying oil samples in the longer wavelength region.

of a fat/oil. From **Figures 1** and **2**, differences in the spectra are observable. The spectrum of the highly degraded oil has lower absorption compared to the spectrum of the fresh oil. Some differences in the shape of the spectra are also observable. The appearance of a more prominent shoulder between 1800 and 1900 nm can be noted in the spectrum of the degraded oil. Also, small differences in the shape of the spectra can be seen between 900–950 and 2000–2150 nm.

**Reference Results.** Characteristics of the reference data for calibration and validation sets are listed in **Table 1**. TPM has been widely used to measure deterioration of frying oils. The polar materials are considered to include all nontriacylglycerol or altered triacylglycerol materials in the oil and polar materials released from foods. The value of TPM increased with an increase in heating time, and there was a linear relationship with heating time.

**NIR and TPM.** There were strong correlations between NIR predicted values and reference data for TPM in both the calibration and validation sample sets (**Table 2**). In the shorter wavelength region, PLS models for predicting TPM gave improved results compared to FSMLR models (**Table 2**). PLS models gave higher correlations and lower rmsd compared to FSMLR models. A PLS model using the first derivative of  $\log(1/T)$  gave slightly improved results compared to the PLS model using the original  $\log(1/T)$ , while a PLS model using the second derivative of  $\log(1/T)$  gave a worse result for V2, indicating overfitting of the model (data not shown). When using the first-derivative  $\log(1/T)$ , the  $r$  values between the NIR and wet chemical methods for V1 and V2 were 0.993 and 0.983, respectively, and the rmsd values for V1 and V2 were 1.730% and 2.690%, respectively. When using FSMLR regression to develop calibration models, the second-derivative treatment provided better results than the original  $\log(1/T)$  and first-

**Table 1.** Characteristics of Reference Data for Calibration and Validation Sets

sample set	no. of samples	constituent range (%)	
		TPM	FFA
calibration	60	2.18–62.93	0.020–0.568
V1	43	2.45–52.39	0.020–0.474
V2	28	5.02–47.31	0.030–0.346

derivative treatment (data not shown). When using second-derivative spectra, the  $r$  values between the NIR and wet chemical methods for V1 and V2 were 0.974 and 0.987, respectively, and the rmsd values for V1 and V2 were 3.450% and 4.910%, respectively.

In the longer wavelength region, PLS models did not provide an advantage over FSMLR models (**Table 2**). When using  $\log(1/T)$  with FSMLR, the  $r$  values between the NIR and wet chemical methods for V1 and V2 were 0.999 and 0.999, respectively, and the rmsd values for V1 and V2 were 0.719% and 0.801%, respectively. Derivative treatments with FSMLR models did not improve the results (data not shown). Instead, the derivative treatments may have resulted in overfitting of the models. Models using derivative treatments gave much higher rmsd values for V2 than for V1 due to a bias. Wavelengths of 1712, 2070, 2126, and 2254 nm were chosen by the program as the key wavelengths for measuring the TPM value when using original  $\log(1/T)$  spectra. According to the table of chemical assignments of Osborne et al. (17), absorption at 1712 nm is characterized by first overtones of C–H stretching vibrations, and the absorption at 2254 nm is due to C–H stretching and deformation vibrations. Absorptions at 2126 and 2070 nm may be due to O–H stretching and deformation vibrations. PLS calibration models gave results similar to those of FSMLR models. Derivative treatments overfitted the models, resulting in a bias when V2 samples were predicted (data not shown). Thus, models using derivative treatments were only good to predict V1 samples. In this study, eliminating the wavelength region  $>2250$  nm in PLS models improved the results. This may be because the region above 2250 nm does not contain any useful information about the constituent of interest, but adds noise to the models.

**FFA Value.** Free fatty acids are the main degradation products of hydrolysis. FFA content is a measure of the amount of fatty acid chains hydrolyzed off the triacylglycerol backbone (12). Generally, FFA values increased with an increase in frying time, but fluctuations were observed due to the easy degradation of FFAs to other polar compounds caused by heat, oxidation, and light (18).

**NIR and FFA.** The NIR predicted values were strongly correlated with the reference data in both calibration and validation sample sets, but correlations were not as strong as for the TPM value (**Table 3**). In the shorter wavelength region, the PLS models gave slightly improved results compared to the FSMLR models (**Table 3**). When using  $\log(1/T)$  in a PLS model, the  $r$  values between the NIR and wet chemical methods for V1 and V2 were 0.962 and 0.943, respectively, and the rmsd values for V1 and V2 were 0.033% and 0.035%, respectively. Use of derivative treatments with PLS modeling did not give improved results (data not shown). On the other hand, first- and second-derivative treatments improved the results when using FSMLR to develop calibration models. The  $r$  values for V1 and V2 were 0.926 and 0.913, respectively, and the rmsd

**Table 2.** Best FSMLR and PLS Calibration Models for TPM Prediction Using Two Wavelength Regions

data treatment	wavelengths (nm) or no. of factors	calibration		V1 (SD = 14.370)			V2 (SD = 12.647)		
		multiple R	SEC (%)	r	rmsd (%)	RPD	r	rmsd (%)	RPD
FSMLR									
second derivative segment 20 nm gap 30 nm	738, 898, 982, 848	0.982	2.890	0.974	3.450	4.165	0.987	4.910	2.576
log(1/T)	1712, 2070, 2126, 2254	0.999	0.721	0.999	0.719	19.986	0.999	0.801	15.789
PLS									
first derivative <sup>a</sup> segment 20 nm gap 20 nm	9	0.996	1.454	0.993	1.730	8.306	0.983	2.690	4.701
log(1/T) <sup>b</sup>	6	0.999	0.734	0.999	0.731	19.658	0.999	0.824	15.348

<sup>a</sup> Wavelength region 700–1100 nm. <sup>b</sup> Wavelength region 1100–2250 nm.

**Table 3.** Best FSMLR and PLS Calibration Models for FFA Prediction Using Two Wavelength Regions

data treatment	wavelengths (nm) or no. of factors	calibration		V1 (SD = 0.114)			V2 (SD = 0.087)		
		multiple R	SEC (%)	r	rmsd (%)	RPD	r	rmsd (%)	RPD
FSMLR									
second derivative segment 20 nm gap 30 nm	738, 908, 930, 970	0.947	0.045	0.926	0.043	2.651	0.913	0.036	2.417
first derivative segment 30 nm gap 30 nm	2052, 2016, 1284, 1226	0.987	0.023	0.978	0.026	4.385	0.940	0.030	2.900
PLS									
log(1/T) <sup>a</sup>	7	0.973	0.033	0.962	0.033	3.455	0.943	0.035	2.486
log(1/T) <sup>b</sup>	10	0.995	0.015	0.987	0.019	6.000	0.983	0.018	4.833

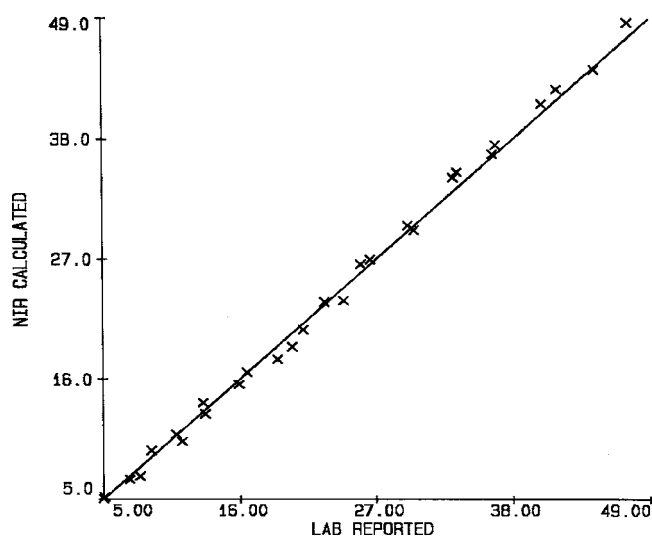
<sup>a</sup> Wavelength region 700–1100 nm. <sup>b</sup> Wavelength region 1100–2250 nm.

values for V1 and V2 were 0.043% and 0.036%, respectively, when using a second-derivative spectral treatment in an FSMLR model.

In the longer wavelength region, the PLS model using log(1/T) gave a better result compared to the corresponding FSMLR model (Table 3). For the PLS model using log(1/T), the *r* values between the NIR and wet chemical methods for V1 and V2 were 0.987 and 0.983, respectively, and the rmsd values for V1 and V2 were 0.019% and 0.018%, respectively. With PLS models, the derivative treatments overfitted the models (data not shown). On the other hand, use of a first-derivative treatment in an FSMLR model gave slightly improved results compared to that of the log(1/T) model.

## DISCUSSION

Results of the best models are summarized in Tables 2 and 3. Comparisons of NIR and reference data from V2 are also shown in Figures 3 and 4. Overall, predictions of TPM were better than the predictions of FFA, as demonstrated by comparing their RPD (ratio of SD to rmsd) values. The RPD value is a simple statistic that enables the evaluation of a standard error of prediction (SEP) or rmsd in terms of the standard deviation (SD) of the reference data of the prediction set and allows comparison of results obtained from sets of data expressed in different units. In general, the RPD of a method should be 5 or higher for the method to be useful for quality control applications (19). Factors that may cause the weaker results for FFA are (a) the titration procedure always has a difficult end point when applied to highly colored or emulsified oils (12), which may cause incorrect readings, thus introducing uncertainty into

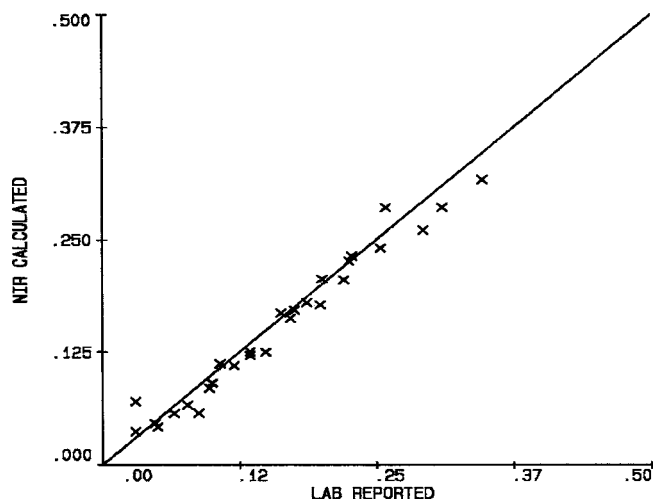


**Figure 3.** Scatter plot diagram comparing TPM (%) of V2 samples, as determined by log(1/T) and FSMLR calibration using the longer wavelength region, to TPM (%) as determined by the reference method.

the reference values, and (b) FFAs easily turn into a variety of other polar compounds due to heat, light, and oxidation, so values are not as stable as TPM.

In summary, calibration models have been successfully developed that can be used to directly measure TPM and FFA values in soy-based frying oils from their NIR spectra. On the basis of the results of this study, the following conclusions can be made: (a) predictions using a longer wavelength region





**Figure 4.** Scatter plot diagram comparing FFA (%) of V2 samples, as determined by  $\log(1/T)$  and PLS calibration using the longer wavelength region, to FFA (%) as determined by the reference method.

(1100–2500 nm) were better than the predictions using a shorter wavelength region (700–1100 nm) for the conditions used; (b) predictions of TPM were better than the predictions of FFA; (c) in the longer wavelength region, FSMLR models were as good or better than PLS models for predicting TPM; (d) in the shorter wavelength region, PLS models were better than FSMLR models for predicting both TPM and FFA; (e) derivative treatments generally had limited usefulness, especially in the longer wavelength region; (f) high values of  $r$  and low values of rmsd were obtained in this study, meaning that NIR is effective in measuring values of TPM and FFA in soy-based frying oil.

Once an instrument is precalibrated, TPM and FFA in frying oils can be quantitatively measured by NIR in less than 3 min, and thus, it can be immediately determined whether an oil is deteriorated on the basis of the TPM and FFA values. The NIR method is fast, simple, accurate, and nondestructive. In addition, it is economical because it is not labor intensive and large amounts of chemical reagents are not used in the test. Additional research is currently under way to determine whether frying different types of foods in an oil affects the reliability of the TPM and FFA measurements.

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