Designation: ~~D7806 – 12~~ D7806 – 20

Standard Test Method for Determination of ~~the Fatty~~Biodiesel (Fatty Acid Methyl ~~Ester (FAME) Content of a Blend of Biodiesel and Petroleum-Based~~ Ester) and Triglyceride Content in Diesel Fuel Oil Using Mid-Infrared Spectroscopy (FTIR Transmission Method)[1]

This standard is issued under the fixed designation D7806; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. ~~Scope~~ Scope*

1.1 This test method covers the determination of fatty acid methyl ester (FAME) biodiesel and triglyceride (TAG) concentrations in traditional diesel and renewable diesel fuel blends using a portable mid-infrared spectrometer.

1.2 The method applies to samples with biodiesel concentrations from 3 % to 40 % by volume. Additionally, it applies to samples with biodiesel concentrations from 2 % to 27 % by volume which contain triglycerides concentrations from 1 % to 10 % by volume. Triglycerides from 2 % to 10 % by volume can be determined in samples of diesel having biodiesel concentrations from 3 % to 27 % by volume. FAME and triglyceride can be simultaneously determined outside these stated ranges, but the stated precision estimates do not apply.

1.3 The method is not able to distinguish TAG and FAME if the TAG concentrations is below 0.142× the measured FAME concentrations.

1.4 This ~~test method covers the determination of the content of biodiesel (fatty acid methyl esters—FAME) in diesel fuel oils. It is applicable to concentrations from 1 to 30 volume %. This procedure is applicable only to FAME. This test method is~~ procedure is not appropriate for the determination of the concentration of biodiesel ~~that is~~ in the form of fatty acid ethyl esters ~~(FAEE).~~(FAEE), see Section 6 for further discussion of possible interferences.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate ~~safety~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D975 Specification for Diesel Fuel

D1298 Test Method for Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method

D1655 Specification for Aviation Turbine Fuels

D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum ~~Products~~ Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.04.0F on Absorption Spectroscopic Methods.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

***A Summary of Changes section appears at the end of this standard**

D4307 Practice for Preparation of Liquid Blends for Use as Analytical Standards
D5854 Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products
D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance
D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products, Liquid Fuels, and Lubricants
D6751 Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels
D7418 Practice for Set-Up and Operation of Fourier Transform Infrared (FT-IR) Spectrometers for In-Service Oil Condition Monitoring
E131 Terminology Relating to Molecular Spectroscopy
E168 Practices for General Techniques of Infrared Quantitative Analysis
E1655 Practices for Infrared Multivariate Quantitative Analysis
E2056 Practice for Qualifying Spectrometers and Spectrophotometers for Use in Multivariate Analyses, Calibrated Using Surrogate Mixtures

3. Terminology

3.1 *Definitions:*

3.1.1 *biodiesel, n*—a fuel composed of mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fats, designated B100 in Specification D6751.

3.1.2 *biodiesel blend, BXX, n*—a blend of biodiesel fuel with petroleum-based diesel fuel.

3.1.2.1 *Discussion*—

In the abbreviation BXX, the XX represents the percentage by volume of biodiesel fuel in the blend.

3.1.3 *diesel fuel oil, n*—a petroleum-based diesel fuel, as described in Specification D975.

3.1.4 *FAME, n*—a biodiesel composed of long chain fatty acid methyl esters derived from vegetable or animal fats.

3.1.5 ~~*Mid-Infrared Spectroscopy,*~~*mid-infrared spectroscopy, n*—uses the mid-infrared region of the electromagnetic spectrum, as described in Terminology E131.

3.1.6 *oxygenate-free middle distillate, n*—a petroleum-based fuel meeting the specifications of D975 or E1655, free of compounds containing esters, acids, or ketones.

3.1.7 *renewable diesel fuel, n*—a biomass-based fuel, meeting the specifications in D975.

3.1.8 *triglycerides, TAG, n*—a naturally occurring ester formed from glycerol and three fatty acid groups, which are the main constituents of natural fats and oils, biodiesel feedstocks, fats and/or oils, that have not been transesterified into biodiesel.

3.2 *Acronyms:*

3.2.1 *AU*—absorption unit

3.2.2 *CLS*—classical least squares

3.2.3 *FAEE*—fatty acid ethyl esters

3.2.4 *FAME*—fatty acid methyl esters

3.2.5 *FTIR*—Fourier transform infrared spectrometer

3.2.6 *IR*—infrared

3.2.7 *PL*—path length

3.2.8 *TAG*—triglyceride

4. Summary of Test Method

4.1 This method utilizes a Fourier transform mid infrared spectrometer fitted with a transmission sample cell with a specified path length. A sample of diesel fuel or biodiesel blend is introduced into ~~a liquid sample cell having a specified path length.~~ the sample cell. A beam of infrared light is ~~imaged~~passed through the sample ~~onto a detector, and the detector response is determined. Wavelengths of the absorption spectrum that correlate highly with biodiesel or interferences are selected for analysis. Mathematical analysis converts the detector response for the selected areas or peaks~~ and the motion of the spectrometer mirrors enables determination of the sample absorption spectrum. Specific regions of the spectrum ~~of an unknown to a concentration of biodiesel.~~where FAME and triglycerides show strong absorption are used for the analysis.

~~4.2 This test method can utilize two different types of spectrometers.~~

~~4.2.1 A Fourier Transform Mid-IR Spectrometer fitted with a transmission sample cell can be used. The absorbance spectrum is baseline corrected to eliminate linear and constant background from the spectrum. Linear regression calibration is calculated without considering the influence of interferences.~~

~~4.2.2 A filter-based Mid-IR spectrometer fitted with a transmission cell can be used. The absorbance values at specified wavenumbers are used to develop a multiple linear regression calibration.~~

5. Significance and Use

5.1 Biodiesel is a fuel commodity primarily used as a value-added blending component with diesel fuel.

~~5.2 This test method is fast and simple to run.~~

5.2 This test method is applicable for quality control in the production and distribution of diesel fuel and biodiesel blends containing FAME.

5.3 The use of triglycerides in fuels is not approved for transportation applications within any ASTM specification. This test method allows the quantification of triglyceride concentration in biodiesel blends, thus enabling detection of out-of-specification blending.

5.4 This test method is fast, simple to run, inexpensive and requires no sample preparation.

6. Interferences

6.1 The ~~primary spectral interferences are vegetable oils, or animal fats, or both.~~presence of carbonyl containing compounds including carboxylic acids, ketones and aldehydes, as well as fatty acid ethyl esters (FAEE), may result in spectral interference.

6.2 The hydrocarbon composition of the diesel fuel has a ~~significant~~minor impact on the calibration model. ~~Therefore, for a robust calibration model, it is important that the diesel fuel in the biodiesel fuel blend is represented in the calibration set.~~The proper choice of the evaluation routine as described in this standard can minimize interferences from the hydrocarbon composition of the diesel fuel.

~~6.3 Proper design of a calibration matrix, utilization of multivariate calibration techniques, and evaluation routines as described in this standard can minimize interferences.~~

6.3 *Water Vapor Interference*—~~This procedure is applicable only~~ The calibration and analysis bands in used for the calculations in Section 12~~to FAME. The concentration of fatty acid ethyl esters (FAEE) cannot be determined using this test method.~~ lie in regions where significant signals due to water vapor can appear in the infrared spectrum. This shall be accounted for to permit calibration and measurements at the low end concentrations.

NOTE 1—Ideally, the spectrometer should be purged with dry air or nitrogen to remove water vapor. The purge should be allowed to stabilize over several hours before analytical work is pursued, due to the rapid changes in the air moisture content within the spectrometer during early stages of the purge. In cases where water vapor prevention or elimination is not possible using a purge, the operator should measure a reference background spectrum for correction of the ratioed spectrum for each sample spectrum measured. This operation is generally automated in today's spectrometer systems and the operator should consult the manufacturer of the spectrometer for specific instructions for implementing automated background correction routines. The spectrometer should be sealed and desiccated to minimize the effect of water vapor variations, and any accessory should be sealed to the spectrometer.

6.4 ~~*Undissolved Water*~~—*High Lauric Acid Methyl Esters Interference*—Samples ~~containing undissolved water will result in erroneous results. Filter cloudy or water saturated samples through a dry filter paper until clear prior to their introduction into the instrument sample cell.~~with high lauric acid methyl ester (that is, coconut biodiesel) content are known to cause a bias when used in the calibration model. Unless the method is being used to calibrate specifically and exclusively for samples containing high levels of lauric acid methyl esters, feedstocks containing high levels of these compounds should be avoided.

7. Apparatus

7.1 *Mid-IR Spectrometric Analyzer:*

7.1.1 *Fourier Transform Mid-IR Spectrometer (FT-IR)*—The type of apparatus suitable for use in this test method employs an IR source, a liquid transmission cell, a scanning interferometer, a ~~deuterated triglycine sulfate~~ detector, an analog-to-digital converter, a microprocessor, and a method to introduce the sample. The following performance specifications must be met:

~~scan range~~	~~4000 to 650 cm^{-1}~~
spectral resolution	4 cm^{-1}
~~digital resolution~~	~~1 cm^{-1}~~

NOTE 2—~~To obtain a digital resolution of 1 cm~~The noise of the single^{-1} ~~for a~~ beam spectrum ~~recorded at 4 cm~~at 100 % transmission shall be less than 0.3 % peak-to-peak in the region from 1725 cm^{-1} ~~requires that~~to 1765 cm^{-1} ~~the interferogram be zero filled prior to Fourier transformation. Consult the FT-IR manufacturer's instructions for the appropriate zero fill parameter settings to achieve this digital resolution.~~. The single beam spectrum obtained can be the average of multiple of FTIR scans but the total collection time shall not exceed 60 seconds.

~~7.1.1.1 The noise level shall be established by taking and ratioing two successive single beam spectra of dry air. The single beam spectra obtained can be the average of multiple of FTIR scans. The noise of the spectrum at 100 % transmission shall be less than 0.3 % peak-to-peak in the region from 1765 to 1725 cm^{-1}.~~

~~7.1.2 *Filter-based Mid-IR Test Apparatus*—The type of apparatus suitable for use in this test method minimally employs an IR source, an infrared transmission cell, wavelength discriminating filters, a chopper wheel, a lithium tantalate detector, an analog-to-digital converter, a microprocessor, and a method to introduce the sample. The frequencies and bandwidths of the filters are specified in Table 1.~~

7.2 ~~*Transmission*~~*Absorption Cell*—The cell shall be a transmission cell made from non-hygroscopic materials having a significant transmission in the ~~appropriate IR wavelength region.~~relevant spectral range (1050 cm^{-1} to 1850 cm^{-1}). The nominal path length of the cell shall be ~~0.10 (± 0.01) mm,~~ 0.10 mm ± 0.015 mm, appropriate to measure the ~~peak regions (as defined~~ relevant peaks involved in ~~Table 1) of samples in scope~~ the spectral analysis without going into saturation. ~~If path length information from the manufacturer is not available,~~The path length should be determined in accordance to Practice D7418~~use cyclohexane as a path length check sample (see~~ . ~~A1.2).~~

8. Reagents and Materials

8.1 *Standards for Calibration, Qualification, and Quality Control Check Standards*—~~As~~Because this ~~test~~ method is intended to quantify FAME and triglyceride content in commercial biodiesel blends there are no high purity standard chemical reference materials that are appropriate for development of multivariate calibration models.

8.1.1 B100 (Neat Biodiesel) used for calibration, qualification and quality control standards must be Specification D6751 compliant. ~~The B100 shall be a methyl fatty acid ester derived from soy. The B100 used to generate the precision of this test method was derived from soy. See~~If the origin of FAME is known, this type shall be used for calibration. If the type of biodiesel is not known, use soy methyl ester ~~Annex A2 for further discussion.~~or the FAME most commonly used in the geographical area where the samples are obtained.

8.1.2 The triglycerides shall be food grade oils liquid at room temperature, preferably soy oil or the type of oil most commonly found in the local market, available for purchase commercially. See Annex A2 for further discussion on different biodiesel and triglyceride varieties.

8.1.3 Middle distillate fuel used for calibration, qualification and quality control standards must be Specification D975 compliant, free of biodiesel or biodiesel ~~oil precursor, or both, and so~~ precursor compounds and should as far as possible ~~should~~ be representative of the petroleum base stocks anticipated for blends to be analyzed (that is, crude source, 1D, 2D, blends, winter/summer cuts, etc). See Annex A2 for ~~calibration set.~~further discussion on calibration fuels.

TABLE 1 ~~Filter Frequencies and Bandwidths~~

~~Center Wave Number (±0.15 % of wave number)~~	~~Bandwidth (in wavelength units) (full width at half height)~~		
~~1745 cm^{-1}~~	~~1605 cm^{-1}~~	~~1 % of λc~~	~~1 % of λc~~
~~1159 cm^{-1}~~	~~1 % of λc~~		
~~915 cm^{-1}~~	~~1 % of λc~~		
~~769 cm^{-1}~~	~~1 % of λc~~		
~~698 cm^{-1}~~	~~1 % of λc~~		

TABLE 1 Precision Estimate Examples for FAME in Diesel / no TAG Detected, FAME in Diesel / TAG Detected, and TAG / FAME Detected for Different Concentrations

C (% Vol)	FAME / no TAG Detected		FAME / TAG Detected		TAG / FAME Detected	
	r	R	r	R	r	R
2			0.10	0.20	0.11	0.37
3	0.06	0.37	0.17	0.33	0.20	0.67
5	0.12	0.56	0.31	0.60	0.41	1.38
7	0.19	0.74	0.45	0.90	0.67	2.22
10	0.31	0.99	0.69	1.36		
15	0.53	1.38	1.12	2.20		
20	0.78	1.75	1.57	3.09		
25	1.04	2.10	2.04	4.02		
30	1.33	2.44				
40	1.95	3.08				

8.1.3 *Diesel Cetane Check Fuel*—Low (DCCF-Low).[3]
8.1.4 *Diesel Cetane Check Fuel*—High (DCCF-High).[3]
8.1.5 *n-Hexane [110-54-3]*—Reagent grade. (**Warning—**Flammable.)
8.1.6 *Hexadecane [544-76-3]*—With a minimum purity of 99.0 volume percent.
8.1.7 *Acetone [67-64-1]*—Reagent grade. (**Warning—**Flammable.)
8.1.8 *Toluene [108-88-3]*—Reagent grade. (**Warning—**Flammable.)
8.1.9 *Cyclohexane [110-82-7]*—Reagent grade. (**Warning—**Flammable.)
8.1.10 *Methanol [67-56-1]*—Reagent grade. (**Warning—**Flammable.)
8.1.11 *Triple Solvent*—A mixture of equal parts by volume of toluene, acetone, and methanol. (**Warning—**Flammable.)

9. Sampling and Sample Handling

9.1 *General Requirements:*

9.1.1 Fuel samples to be analyzed by the test method shall be sampled using procedures outlined in Practices D4057 or D4177, where appropriate. Do not use the "Sampling by Water Displacement" procedure.

9.1.2 Protect samples from excessive (<0 °C and >60 °C) temperatures prior to testing.

9.1.3 Until test samples are known to contain <4.0 % by volume TAG, avoid storage of samples in refrigerated conditions (≤ ≈10 °C).

9.1.4 Do not test samples stored in leaking containers. Discard and obtain a new sample if leaks are detected.

9.2 *Sample Handling During Analysis:*

9.2.1 Equilibrate all samples to the typical temperature of the laboratory (15(15 °C to 27°C)27 °C) prior to analysis by this test method.

9.2.2 After analysis, if the sample is to be saved, reseal the container before storing.

10. Calibration and Qualification of the Apparatus

10.1 Calibrate the instrument according to the procedure described in Annex A1. This calibration canmay be performed by the instrument manufacturer prior to delivery of the instrument to the end user. Perform this qualification procedure anytime the instrument is calibrated.

10.2 Perform this qualification procedure when an instrument is initially put into operation, when it is recalibrated, or when it is repaired. The qualification procedure is described in Annex A1.

11. Quality Control Checks

11.1 Each day it that the instrument is to be used, confirm that the instrument it is in statistical control by measuring the biodiesel concentration using the procedure outlined in Section 12 on at least one quality control sample of known biodiesel content. The preparation of samples with known biodiesel concentration is described in 11.1.1 and 11.1.2. For details on quality control testing and control charting refer to Practice D6299.

11.1.1 Standard(s) of known biodiesel and triglyceride concentration shall be prepared by mass according to A1.1.1and converted to volume % volume percent using the measured density as outlined in Section 1313.1. At least one standard shall be prepared for each calibration range. For example, 2 volume % may be used for the low calibration range, 20 volume % for high calibration range. Additional standards including 0 0 % by volume percent may also be prepared and used for quality control checks.

11.1.2 Standard(s) should be prepared in sufficient volume to allow for a minimum of 30 quality control measurements to be made on one batch of material. Properly package and store the quality control samples to ensure that all analyses of quality control samples from a given lot are performed on essentially identical material.

11.2 If the biodiesel volume % volume percent value estimated for the quality control sample exceeds the action limits described specified in Practice D6299 or equivalent, then the measurement system is out-of-control and cannot be used to estimatemeasure biodiesel concentrations until the cause of the out-of-control behavior is identified and corrected.

11.3 If correction of out-of-control behavior requires repair to the instrument or recalibration of the instrument, the qualification of instrument performance described in A1.4 shall be performed before the system is used to measure biodiesel content on samples.

12. Procedure

12.1 Equilibrate all samples to the temperature of the laboratory (15 °C to 27 °C) prior to analysis by this test method.

12.2 *Background Spectrum*—Record a single beam infrared spectrum of dry air or nitrogen.

[3] The sole source of supply of the apparatus known to the committee at this time is Chevron Phillips Chemical Company LP, 10001 Six Pines Drive, The Woodlands, TX 77380. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at aSupporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-2002meeting of the responsible technical committee,. Contact ASTM Customer Service at[1] which you may attend.service@astm.org.

12.3 ~~*FTIR Procedure:*~~ Prior to the analysis of unknown test samples, establish that the equipment is running properly by collecting the spectrum of the quality control standard(s) and comparing the estimated biodiesel concentration to the known value for the QC standard(s).

~~12.1.1 If the FTIR instrument is used, remove the fuel by flushing the cell and inlet-outlet lines with sufficient solvent, described in 8.1.11. Evaporate the residual solvent with dry air.~~

~~12.1.2 *Background Spectrum*—Record a single beam infrared spectrum of dry air. This spectrum can be used as a background spectrum for 6 h.~~

~~12.1.3 Prior to the analysis of unknown test samples, establish that the equipment is running properly by collecting the spectrum of the quality control standard(s) and comparing the estimated biodiesel concentration to the known value for the QC standard(s). Introduce enough standard to the cell to ensure that the cell is washed by a volume of at least three times the dead volume of the sample introduction system.~~

~~12.1.4 Equilibrate the unknown fuel sample to the typical temperature of the laboratory (15 to 27°C) before analysis.~~

~~12.1.5 Introduce enough of the fuel sample to the cell to ensure the cell is washed by a volume of at least three times the dead volume of the sample introduction system.~~

~~12.1.6 Obtain the digitized spectral response of the fuel sample over the frequency region from 4000 to 650 cm^{-1}.~~

~~12.1.7 Measure the absorption spectrum and note the maximum absorption value of the peak in the region 1765 to 1720 cm^{-1}.~~

~~12.1.8 Biodiesel and high concentrations of biodiesel in biodiesel blends are difficult to remove from the cell surface. Flush several times with sample or use a solvent rinse between samples. When in doubt, repeat steps 12.1.6 through 12.1.8 and compare result to ensure adequate rinsing occurred.~~

~~12.1.9 For FTIR instruments using a baseline correction step and a linear regression calibration, determine the biodiesel concentration using the calibration models developed in A1.3 by following the steps outlined as follows.~~

~~12.1.9.1 If the absorption value (determined in 12.1.8) is smaller or equal to 1.0, calculate the baseline corrected absorption spectrum. The baseline is defined through the absorption values at the wavenumber 1708 and 1785 cm^{-1}. Calculate the area from the wavenumber 1713 to 1784 cm^{-1}. Estimate the biodiesel concentration by applying the low concentration linear regression calibration (see A1.3.3.1).~~

~~12.1.9.2 If the absorption value (determined in 12.1.7) is greater than 1.0, calculate the baseline corrected absorption spectrum. The baseline is defined through the absorption values at the wavenumber 1126 and 1225 cm^{-1}. Calculate the area from the wavenumber 1126 to 1220 cm^{-1}. Estimate the biodiesel concentration by applying the high concentration linear regression calibration (see A1.3.3.2).~~

12.4 Rinse the absorption cell according to the manufacturer's instructions. Biodiesel and high concentrations biodiesel blends are difficult to remove from the cell. Flush the cell with sample or use a solvent rinse between measurements to ensure that carry-over between measurements is minimal. Measuring a blank (oxygenate-free middle distillate or jet fuel) after the measurement of the high range quality control sample can be used to verify adequate rinsing.

12.5 Introduce the unknown sample into the sample cell and obtain the digitized absorption spectrum (AS) of the fuel sample over the frequency range from 1050 cm^{-1} to 1850 cm^{-1}.

12.6 Normalize the absorption spectrum to 0.1 mm path length, that is, multiply the spectrum with the factor 0.1/PL, where PL is the path length of the cell in mm. The absorption spectrum after normalization should be used for the subsequent analysis.

12.7 Perform a classical least squares (CLS) (A1.1.8) fit of the spectrum of the unknown sample as described in A1.1.7 and A1.1.8. Determine the respective concentrations using the CLS calibration (A1.1.9).

12.8 Determine the relative contribution of biodiesel and triglycerides.

$$X_i = \frac{c_i}{\sum_i c_i}, i \in \text{FAME and TAG} \quad (1)$$

If $X_{TAG} < 0.14$, set $X_{TAG} = 0$ and $X_{FAME} = 1$.
If $X_{FAME} < 0.14$, set $X_{FAME} = 0$ and $X_{TAG} = 1$.

12.9 Let P be the maximum of the absorption spectrum between 1735 cm^{-1} and 1758 cm^{-1} without any baseline correction.

12.10 If $X_{FAME} > 0$ and $X_{TAG} = 0$, the sample contains only biodiesel.

12.10.1 If P > 1.55 AU, calculate the peak area from 1230 cm^{-1} to 1288 cm^{-1} using the baseline points at 1105 cm^{-1} and 1330 cm^{-1} and use the high range FAME calibration to determine the biodiesel concentration (A1.1.11). Report the triglyceride concentration as zero.

12.10.2 If P ≤ 1.55 AU, calculate the peak area from 1730 cm^{-1} to 1766 cm^{-1} using the baseline points at 1673 cm^{-1} and 1838 cm^{-1} and use the low concentration FAME calibration to determine the biodiesel concentration (A1.1.10). Report the triglyceride concentration as zero.

12.11 If $X_{FAME} = 0$ and $X_{TAG} > 0$, the sample contains only triglycerides.

12.11.1 If P > 1.55 AU, report the concentration determined by the CLS calibration, c_{TAG} (A1.1.9). Report the biodiesel concentration as zero.

12.11.2 If P ≤ 1.55 AU, calculate the peak area from 1730 cm^{-1} to 1766 cm^{-1} using the baseline points at 1673 cm^{-1} and 1838 cm^{-1} and use the low concentration TAG calibration to determine the triglyceride concentration (A1.1.10). Report the biodiesel concentration as zero.

12.12 ~~*Filter-Based Mid-IR Instruments:*~~ If X_{FAME} > 0 and X_{TAG} > 0, the sample contains a mixture of biodiesel and triglycerides.

~~12.2.1 Equilibrate the unknown fuel sample to the typical temperature of the laboratory (15 to 27°C) before analysis.~~

12.12.1 ~~Introduce enough of the fuel sample to the cell to ensure~~If P > 1.55 AU, report the biodiesel and triglyceride concentrations determined by the CLS calibration, c_{TAG} ~~the cell~~and c_{FAME} ~~is~~ (A1.1.9~~washed by a volume of at least three times the dead volume of the sample introduction system.~~).

12.12.2 ~~For the filter-based Mid-IR test apparatus~~If P ≤ 1.55 AU, calculate the peak area from 1730 cm^{-1} to 1766 cm^{-1}, using the baseline points at 1673 cm^{-1} and 1838 cm^{-1}~~determine the biodiesel concentration using the calibration models developed in~~ and use the low concentration FAME calibration to determine the sum of triglyceride and biodiesel concentrations, C (~~A1.4~~A1.1.10 ~~by following the steps outlined as follows.~~). Calculate the biodiesel and triglyceride concentrations as $C \cdot X_{FAME}$ and $C \cdot X_{TAG}$ respectively.

~~12.2.3.1 Estimate the FAME concentration using the universal equation developed in A1.4.2.~~

~~12.2.3.2 If the estimated FAME concentration is ≤6.0 volume percent use the low concentration equation developed in A1.4.3 to determine the FAME concentration.~~

~~12.2.3.3 If the estimated FAME concentration is >6.0 volume percent but ≤30.0 volume percent use the high concentration equation developed in A1.4.4 determine the FAME concentration.~~

~~12.2.3.4 The precision of the analysis may cause the result obtained from the narrow range calibration to not correspond to the result obtained from the universal calibration at the interface between the narrow calibrations (6.00 volume percent). If the result from the universal calibration and the result from the indicated narrow calibration agree to within the cross method reproducibility then the result using the narrow calibration is the accepted result. If the two results do not agree then check the instrument performance using a check standard.~~

13. Calculation

13.1 *Conversion to ~~Volume %~~ Volume Percent of Biodiesel*—To convert the calibration and qualification standards to ~~volume %~~ volume percent use Eq ~~1~~2:

~~$V_b = M_b(D_f / D_b)$~~ (2)

$$V_b = M_b(D_f / D_b) \quad (2)$$

where:

~~V_b = biodiesel volume %,~~
~~M_b = biodiesel mass %,~~
~~D_f = relative density at 15.56°C of the calibration or qualification standard being tested as determined by Practice D1298 or Test Method D4052, and~~
~~D_b = B100 biodiesel blend stock relative density at 15.56°C of the calibration or qualification standard being tested as determined by Practice D1298 or Test Method D4052.~~

V_b = biodiesel volume percent,
M_b = biodiesel mass percent,
D_f = relative density at 15.56 °C of the calibration or qualification standard being tested as determined by Practice D1298 or Test Method D4052, and
D_b = B100 biodiesel blend stock relative density at 15.56 °C of the calibration or qualification standard being tested as determined by Practice D1298 or Test Method D4052.

13.2 ~~*Calculation of the Peak Area*~~—*Conversion to Volume Percent of Triglyceride*—To ~~calculate the peak area~~ convert the calibration and qualification standards to volume percent, use Eq ~~2~~3:

$$A_{v_1-v_2} = \sum_{i=v_1}^{v_2-1} \frac{x_i + x_{i+1}}{2} \quad (3)$$

$$V_t = M_t(D_f / D_t) \quad (3)$$

where:

~~$A_{v_1-v_2}$ = area of the absorption spectrum in the range from v_1 to v_2,~~
~~v = wave number in cm^{-1},~~
~~x_i = absorbance at wave number i, and~~
~~i = enumeration index.~~