Designation: D7861 – 14$^{\varepsilon 1}$

Standard Test Method for
Determination of Fatty Acid Methyl Esters (FAME) in Diesel Fuel by Linear Variable Filter (LVF) Array Based Mid-Infrared Spectroscopy[1]

This standard is issued under the fixed designation D7861; 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.

ε^1 NOTE—The equation for repeatability in subsection 14.1.1 was corrected editorially in December 2015.

1. Scope

1.1 This test method determines fatty acid methyl esters (FAME or biodiesel) in diesel fuel oils. FAME can be quantitatively determined from 1.0 % to 30.0 % by volume. This test method uses linear variable filter (LVF) array based mid-infrared spectroscopy for monitoring FAME concentration.

NOTE 1—See Section 6 for a list of interferences that could affect the results produced from this method.

1.2 This test method uses a horizontal attenuated total reflectance (HATR) crystal and a univariate calibration.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D975 Specification for Diesel Fuel Oils
D1298 Test Method for Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method
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
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 and Lubricants
D6751 Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels
D7371 Test Method for Determination of Biodiesel (Fatty Acid Methyl Esters) Content in Diesel Fuel Oil Using Mid Infrared Spectroscopy (FTIR-ATR-PLS Method)
D7467 Specification for Diesel Fuel Oil, Biodiesel Blend (B6 to B20)
E168 Practices for General Techniques of Infrared Quantitative Analysis (Withdrawn 2015)[3]
E1655 Practices for Infrared Multivariate Quantitative Analysis

3. Terminology

3.1 *Definitions:*

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

3.1.2 *biodiesel blend (BXX), n*—blend of biodiesel fuel with diesel fuel oils.

3.1.2.1 *Discussion*—In the abbreviation, BXX, the XX represents the volume percentage of biodiesel fuel in the blend.

3.1.3 *diesel fuel, n*—petroleum-based middle distillate fuel.

3.1.4 *univariate calibration, n*—aprocess for creating a calibration model in which a single measured variable, for

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum 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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

example, the absorbance at a particular wavelength, is correlated with the concentration or property values for a set of calibration samples.

3.2 *Acronyms:*

3.2.1 *ATR, n*—attenuated total reflectance

3.2.2 *BXX, n*—see 3.1.2

3.2.3 *FAEE, n*—fatty acid ethyl esters

3.2.4 *FAME, n*—fatty acid methyl esters

3.2.5 *HATR, n*—horizontal attenuated total reflectance

3.2.6 *LVF, n*—linear variable filter

4. Summary of Test Method

4.1 A sample of diesel fuel or biodiesel blend (BXX) is placed onto a HATR sample crystal. Infrared light is imaged through the sample, then through the LVF and finally onto a detector array. The LVF separates the infrared light into specific wavelengths so that the response of the detector array generates an infrared spectrum. Spectral corrections are performed to eliminate interferences caused by diesel and biodiesel variations. A wavelength region of the absorption spectrum that correlates highly with biodiesel is selected for analysis. The area of the selected region is determined. A calibration curve converts the selected area of an unknown sample to a concentration of biodiesel.

4.2 This test method uses a LVF array based mid-infrared spectrometer with an HATR crystal. The absorption spectrum shall be used to calculate a calibration curve.

5. Significance and Use

5.1 Biodiesel is a fuel commodity primarily used as a blending component with diesel fuel. It is important to check the concentration of biodiesel in the diesel fuel in order to make sure it is either not below the minimum allowable limit and or does not exceed the maximum allowable limit.

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

6. Interferences

6.1 The hydrocarbon composition of diesel fuels can affect the accuracy of the calibration. When possible it is advised that diesel fuels used in calibration be similar to the unknown samples to be analyzed.

6.2 *Undissolved Water and Particulates*—Samples containing undissolved water, particulates, or both will result in erroneous results. If the sample is cloudy or water saturated after it has been equilibrated between 15 °C to 27 °C, filter the sample through a qualitative filter paper until clear prior to their introduction onto the instrument sample crystal.

6.3 The primary spectral interferences are vegetable oils or animal fats, or both. Other means of analysis or separate calibrations may be required if fuel is suspected to be contaminated with vegetable oils or animal fats, or both.

6.4 Due to the inherent variability in LVFs, calibrations cannot be transferred between instruments. Each instrument shall be calibrated separately prior to use.

6.5 This test method is not appropriate for fatty acid ethyl esters (FAEE). FAEEs will cause a negative bias.

7. Apparatus

7.1 *Mid-Infrared Spectrometer:*

7.1.1 *LVF Array Based Mid-Infrared Spectrometer*—The type of apparatus suitable for use in this test method employs an IR source, a HATR crystal, a LVF paired to a detector array, an A/D converter, a microprocessor, and controller software. Specifications of sub parts of the analyzer listed below will determine the applicability of an instrument to this test method.

7.1.2 The noise level shall be established by acquiring a single beam spectrum of air. The single beam spectrum may be an average of multiple instrument scans but the total collection time shall not exceed 60 s. The noise of the spectrum at 100 % transmission shall be less than 0.3 % in the range of 5.50 µm to 5.90 µm (1818 cm^{-1} to 1725 cm^{-1}).

7.2 *Detector Array/Linear Variable Filter Specifications*—The infrared detector array shall have at least 128 detection channels. This detector array shall be paired to a LVF with a range that includes the region of 5.4 µm to 6.0 µm. At least ten detector channels shall be within the range of 5.4 µm to 6.0 µm. The filter shall have a resolution of at least 50 cm^{-1}.

7.3 *Horizontal Attenuated Total Reflection Crystal*—A horizontal attenuated total reflectance (ATR) crystal, with zinc selenide element mounted on a horizontal plate shall be used. Any number of internal reflections (bounces) may be used, however the absorbance at 1745 cm^{-1} shall not exceed 1.1 absorbance units for the highest concentration calibration standard used in the calibration range. Therefore, for higher concentration measurements, careful consideration of element length and face angle shall be made to maximize sensitivity without exceeding 1.1 absorbance units at 1745 cm^{-1}.

7.4 Note that other spectrometer configurations can provide adequate results; however, the precision and bias data listed with this test method was collected based on these apparatus specifications. Any modifications can result in precision and or bias that differ from the numbers listed in this test method.

8. Reagents and Materials

8.1 *Purity of Reagents*—Spectroscopic grade (preferred) or reagent grade chemicals shall be used in tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the committee on analytical reagents of the American Chemical Society, where such specifications are available.[4] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.1.1 Hexane, anhydrous [110-54-3] or Heptane [142-82-5] for use as a cell cleaning agent.

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[4] Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, D.C. For suggestions on the testing of reagents not listed by the American Chemical Society, see Annual Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K., and the United States Pharmacopeia and National Formulary, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

8.1.2 B100 used for calibration, qualification, and quality control standards are recommended to be compliant with Specification D6751 or similar FAME specifications. The biodiesel (B100) shall be FAME. A BQ-9000 certified producer for the biodiesel is recommended to ensure quality of product.[5]

8.1.3 Middle distillate fuel used for calibration, qualification, and quality control standards are recommended to be compliant with Specification D975 or similar diesel fuel specifications, be free of biodiesel or biodiesel oil precursor, or both. If possible, middle distillate fuel shall be representative of diesel fuels anticipated for blends to be analyzed (crude source, 1D, 2D, blends, winter/summer cuts, low aromatic content, high aromatic content, and so forth).

9. Sampling, Test Specimens, and Test Units

9.1 *General Requirements:*

9.1.1 Fuel samples to be analyzed by this test method shall be sampled using procedures outlined in Practice D4057 or D4177, where appropriate. Do not use "sampling by water displacement." FAME is more water-soluble than the hydrocarbon base in a biodiesel blend.

9.1.2 Protect samples from excessive temperatures prior to testing.

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

9.2 *Sample Handling During Analysis:*

9.2.1 When analyzing samples using this method, the sample temperature needs to be within the range of 15 °C to 27 °C. Equilibrate all samples to the temperature of the test site (15 °C to 27 °C) prior to analysis by this test method.

9.2.2 After the analysis, if the sample is to be retained, reseal the container before storage.

9.2.3 Avoid using plastic materials for sampling and do not use rubber caps or plastic bottles for storage of the sample.

10. Preparation of Apparatus

10.1 Before use, the instrument needs to be calibrated according to the procedure described in Annex A1. This calibration may be performed by the instrument manufacturer prior to delivery of the instrument to the end user. If, after maintenance, the instrument calibration is repeated, the qualification procedure shall also be repeated.

10.2 Before use, the instrument shall be qualified according to the procedure described in Annex A1. The qualification need only be carried out when the instrument is initially put into operation, recalibrated, or repaired.

11. Calibration and Standardization

11.1 Information on calibration and qualification of the apparatus can be found in Annex A1.

11.2 Confirm the in-statistical-control status of the test method each day it is used by measuring the biodiesel concentration of at least one quality control sample that is similar in composition and matrix to samples routinely analyzed. For details on quality control sample selection, preparation, testing, and control charting, refer to Practice D6299.

11.3 A system that is found to be out of statistical control cannot be used until the root cause(s) of out-of-control is identified and corrected.

11.4 If correction of out-of-control behavior requires repair to the instrument or recalibration of the instrument, the qualification of instrument performance described in A1.3 shall be performed and the in-statistical control status shall be confirmed.

12. Procedure

12.1 Equilibrate the samples to between 15 °C and 27 °C before analysis.

12.2 Clean the sample crystal of any residual fuel or other contamination according to the manufacturer's recommendation. Hexane or heptane has been determined to be suitable for cleaning the sample cell. It is recommended that the sample crystal be cleaned at least twice before a baseline spectrum is obtained since a clean baseline spectrum is critical for ensuring correct results.

12.3 Obtain a baseline spectrum in the manner established by the manufacturer of the equipment.

12.4 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(s) to the known value(s) for the QC standard(s).

12.5 Introduce the unknown fuel sample in the manner established by the manufacturer. Ensure that the entire crystal surface is covered with fuel.

12.6 Obtain the digitized spectral response of the fuel sample in the manner established by the manufacturer of the equipment in a spectral range containing 5.4 µm to 6.0 µm.

12.7 Determine and record the biodiesel concentration according to the calibration curve generated in Annex A1.

12.8 Wipe the sample off of the sample crystal and clean thoroughly according to manufacturer's specification.

12.9 Biodiesel and biodiesel blends containing high concentrations of biodiesel are difficult to remove from the ATR crystal surface. The sample crystal should be cleaned thoroughly between each sample. When in doubt, repeat steps 12.5 – 12.8 and compare the results to ensure adequate cleaning occurred.

13. Report

13.1 Report the following information:

13.1.1 Volume Percent Biodiesel by Test Method D7861, to the nearest 0.1 %.

14. Precision and Bias

14.1 The precision of this test method is based on an interlaboratory study conducted in 2011. A total of twelve

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[5] A current list of BQ9000 producers can be found at the National Biodiesel Accreditation Program's website http://www.bq-9000.org/ or by contacting them at 573-635-3893.

laboratories participated in this study, testing samples of eighteen different diesel blends for specified biodiesel contents. Not every laboratory was able to submit results for every diesel/biodiesel combination, however each "test result" reported represents an individual determination, and all participants were asked to report triplicate test results for each diesel/biodiesel pairing. Practice D6300 was followed for the analysis of the data.[6,7]

14.1.1 *Repeatability (r)*—The difference between repetitive results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

$$\text{repeatability } (r) = 0.011 \times (X + 6.485) \text{ \% by volume}$$

$$\text{applicable range: } 1.0 \text{ \% to } 30.0 \text{ \% by volume}$$

where:

X = biodiesel concentration determined.

14.1.2 *Reproducibility (R)*—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

$$\text{reproducibility } (R) = 0.043 \times (X + 6.485) \text{ \% by volume}$$

$$\text{applicable range: } 1.0 \text{ \% to } 30.0 \text{ \% by volume}$$

where:

X = biodiesel concentration determined.

NOTE 2—Only soy, canola, and waste vegetable oil biodiesel were used in the determination of the repeatability and reproducibility of this test method. The repeatability and reproducibility of other biodiesels could be different.

14.2 *Bias*—No known reference materials were tested as part of this study, therefore no statement on bias can be made at this time.

15. Keywords

15.1 biodiesel; biodiesel blend; biodiesel concentration; FAME; fatty acid methyl esters; infrared spectroscopy

TABLE 1 Example Calculations for r and R

Concentration (Volume Percent)	*r*	*R*
1.0	0.08	0.32
5.0	0.13	0.49
10.0	0.18	0.71
15.0	0.24	0.92
20.0	0.29	1.14
25.0	0.35	1.35
30.0	0.40	1.57

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[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting RR:D02-1795.

[7] The following equipment, as listed in RR:D02-1795, InfraSpec VFA-IR Spectrometer available from Wilks Enterprise Inc. was used to develop the precision statement. This listing is not an endorsement or certification by ASTM International.

ANNEXES

(Mandatory Information)

A1. CALIBRATION AND QUALIFICATION OF THE APPARATUS

A1.1 *Calibration Matrix*—Calibration standards shall be prepared in accordance with Practice D4307 or D5854 where appropriate. It is recommended that the blend components be compliant with Specification D975 or similar diesel fuel specifications (for base petroleum diesel components) and Specification D6751 or similar FAME specifications (for B100 biodiesel components).

A1.1.1 *Calibration Standards*—To obtain the best precision and accuracy of the calibration, prepare a biodiesel calibration set from 0 % to 30 % biodiesel as set forth in Table A1.1.

TABLE A1.1 Instrument Calibration Set

Sample	Biodiesel (Volume Percent)	Solvent
1	0	Ultra Low Sulfur Diesel
2	2	Ultra Low Sulfur Diesel
3	5	Ultra Low Sulfur Diesel
4	10	Ultra Low Sulfur Diesel
5	15	Ultra Low Sulfur Diesel
6	20	Ultra Low Sulfur Diesel
7	25	Ultra Low Sulfur Diesel
8	30	Ultra Low Sulfur Diesel

A1.1.2 Measure the density for each of the components to be mixed and of the calibration standards according to either Test Method D1298 or D4052.

A1.1.3 For each of the calibration standards, convert the mass percent biodiesel to volume percent biodiesel according to the Eq A1.1 presented in A1.1.3.1. If the densities of the calibration standards cannot be measured, it is acceptable to convert to volume percent using the densities of the individual components measured using Test Method D1298 or D4052.

A1.1.3.1 *Conversion to Volume Percent of Biodiesel*—To convert the calibration and qualification standards to volume percent, use Eq A1.1.

$$V_b = M_b(D_f / D_b) \qquad (A1.1)$$

where:

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 Test Method D1298 or 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 Test Method D1298 or D4057.

A1.2 *Calibration*:

A1.2.1 Equilibrate all samples to the temperature of the laboratory (15 °C to 27 °C) prior to analysis. Apply calibration standards to the crystal in accordance with Practice E168 or in accordance with the manufacturer's instructions. See Table A1.1 for a list of recommended calibration standards to be used.

A1.2.2 Allow the instrument to warm up for at least one hour before attempting a calibration.

A1.2.3 Clean the sample crystal of any residual fuel or other contamination according to the manufacturer's specification. It is recommended that the sample crystal be cleaned at least twice before a baseline spectrum is obtained since a clean baseline spectrum is critical for ensuring a quality calibration.

A1.2.4 Obtain a baseline spectrum in the manner established by the manufacturer of the equipment.

A1.2.5 Introduce the lowest concentration calibration standard to the instrument in the manner established by the manufacturer. Ensure that the entire crystal surface is covered with fuel.

A1.2.6 Obtain the digitized spectral response of the fuel sample in the manner established by the manufacturer of the equipment in a spectral range containing 5.4 µm to 6.0 µm. The infrared spectrum is the negative logarithm of the ratio of the transmittance obtained with a sample in the infrared light beam and the transmittance obtained without the sample in the infrared light beam.

A1.2.7 Clean the sample crystal between each sample in the manner established by the manufacturer.

A1.2.8 Repeat this process for each calibration standard, from lowest to highest concentration.

A1.2.9 Repeat steps A1.2.3 – A1.2.8 twice more, so that each calibration standard is run three times.

A1.2.10 Apply a linear baseline correction to each spectrum, using 5.6 µm to 5.65 µm (1786 cm^{-1} to 1770 cm^{-1}) and 5.85 µm to 5.90 µm (1709 cm^{-1} to 1695 cm^{-1}) as correction regions. This is done by fitting a line to the absorbance values located inside the two correction regions on a wavelength versus absorbance plot. Subtract this line from the spectrum of each calibration standard. This may be done using supporting software.

A1.2.11 Integrate the area under the absorbance curve for each corrected spectrum in the range of 5.65 µm to 5.75 µm (1770 cm^{-1} to 1739 cm^{-1}).

A1.2.12 Plot each area value on an absorbance area versus concentration plot and generate a double exponential fit to the data. This double exponential curve shall be used to determine the biodiesel concentration of unknown samples. Software capable of plotting and fitting this data should be used in generating the calibration curve.

A1.3 *Qualification of Instrument Performance*—Once a calibration has been established, qualify the calibrated instrument to ensure that the instrument accurately and precisely measures biodiesel in the presence of typical compression-ignition engine fuel compounds that, in typical concentrations, present spectral interferences. This qualification need only be carried out when the instrument is initially put into operation, is recalibrated, or repaired.

A1.3.1 *Preparation of Qualification Samples*—Prepare qualification standards of the biodiesel by mass according to Practices D4307 or D5854, where appropriate. Prepare the qualification samples of different concentrations of biodiesel over a range that spans at least 95 % of that for the calibration standards. The numbers of required standards are suggested by Practice E1655. In general, will be three times the number of independent variables in the calibration equation.

A1.3.2 *Acquisition of Qualification Data*—For each of the qualification standards, measure the biodiesel concentration, expressed in volume percent, according to the procedure established in Section 12.

A1.3.3 *Qualifications Calibration Procedures*—Calculate the standard error of qualification as follows:

A1.3.3.1 The standard error of qualification (SEQ) is calculated as follows:

$$SEQ = \sqrt{\frac{\sum_{i-1}^{q}(\hat{y}_i - y_i)}{q}} \qquad (A1.2)$$

where:

q = number of surrogate qualification mixtures,
y_i = component concentration for the i$^{\text{th}}$ qualification sample, and
$\hat{y}_i$ = estimate of the concentration of the i$^{\text{th}}$ qualification sample.

A1.3.3.2 If SEQ is less than PSEQ (the pooled standard error of qualification for the round robin instruments), then the instrument is qualified to perform the test.

A1.3.3.3 If SEQ is greater than PSEQ, calculate an *F* value by dividing the square of SEQ by the square of PSEQ. Compare the *F* value to the critical *F* value with *q* degrees of freedom in the numerator and DOF (PSEQ) degrees of freedom in the denominator. Values of PSEQ and DOF (PSEQ) are given in Table A1.2 and the critical *F* values for the DOF (PSEQ) for the instruments used in the interlaboratory study.

A1.3.3.4 If the *F* value is less than or equal to the critical *F* value from the table, then the instrument is qualified to perform the test.

A1.3.3.5 If the *F* value is greater than the critical *F* value from the table, then the instrument is not qualified to perform the test.

TABLE A1.2 Pooled Standard Errors of Qualification

	ILS
PSEQ	1.08
DOF (PSEQ)	924

Critical F Values for DOF (PSEQ) = 924[A]

[A] From Standard Mathematical Tables, Chemical Rubber Publishing Co, Cleveland (1961).

TABLE A1.3 Critical F Value

DOF (Numerator SEQ)	Critical *F* Value
10	1.83
15	1.67
20	1.57
30	1.46
50	1.35
100	1.25

A2. EFFECT OF DIESEL FUEL TYPES

A2.1 *Diesel Fuel Types*—The ILS included samples made from three different diesel fuels:

A2.1.1 *Low Aromatic*—Fischer-Tropsch product with no aromatics.

A2.1.2 *Mid Aromatic*—Cetane #53 with aromatics content 21.1 % by volume.

A2.1.3 *High Aromatic*—Cetane #43.6 with aromatics content 29 % by volume.

A2.2 *Results*—The ILS results for each of these diesels are shown in Figs. A2.1-A2.3

A2.2.1 The analysis for biodiesel in the Fischer-Tropsch fuel (Fig. A2.1) clearly has similar precision to those of the other analyses but a distinctly different slope. For analysis of biodiesel in fuels of this type a separate calibration or adjustment of this calibration would be necessary.

A2.2.2 The statistics in Table A1.2 were derived from only the data represented in Figs. A2.2 and A2.3.

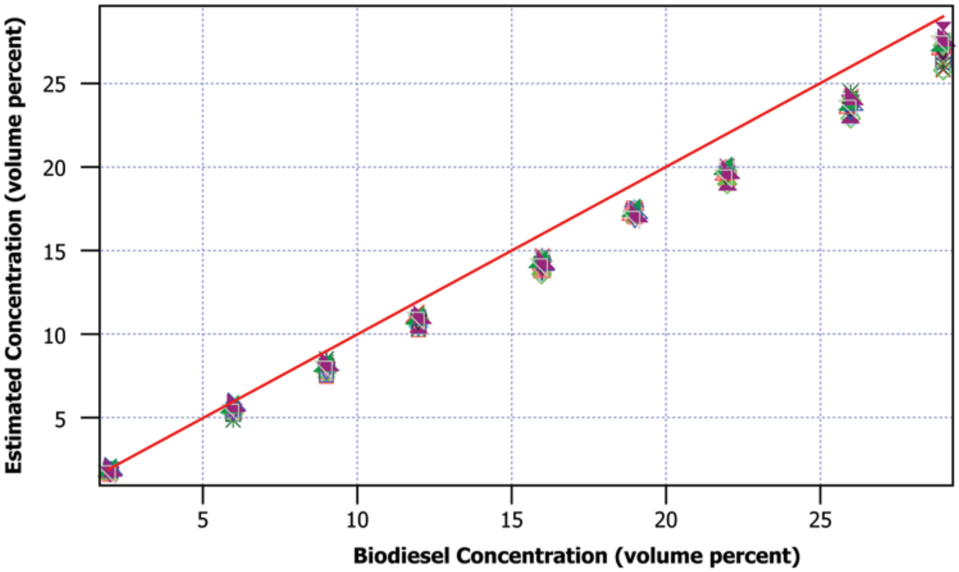

FIG. A2.1 Biodiesel in Fischer-Tropsch Diesel Fuel, Aromatics Content 0 % by Volume

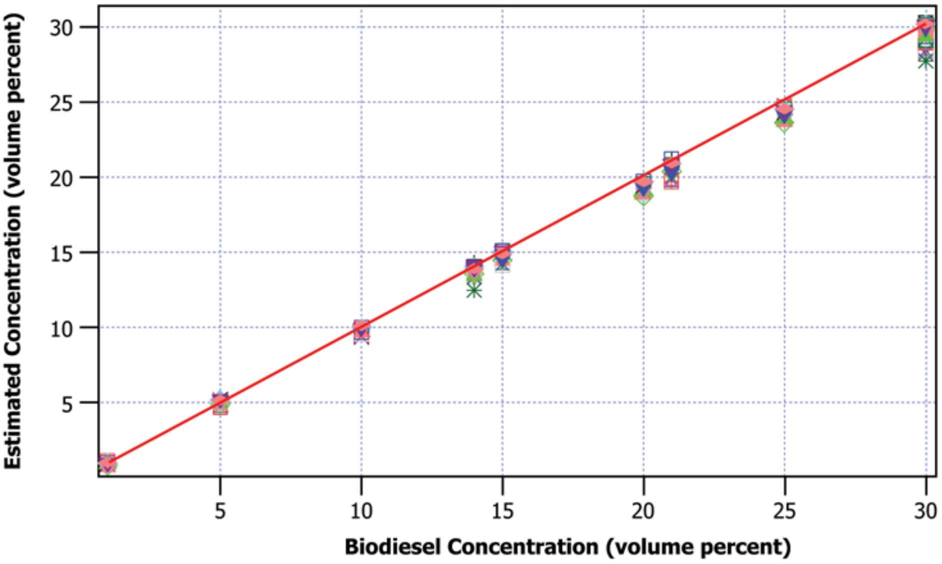

FIG. A2.2 Biodiesel in Diesel Fuel, Cetane #53, Aromatics Content 21.1 % by Volume

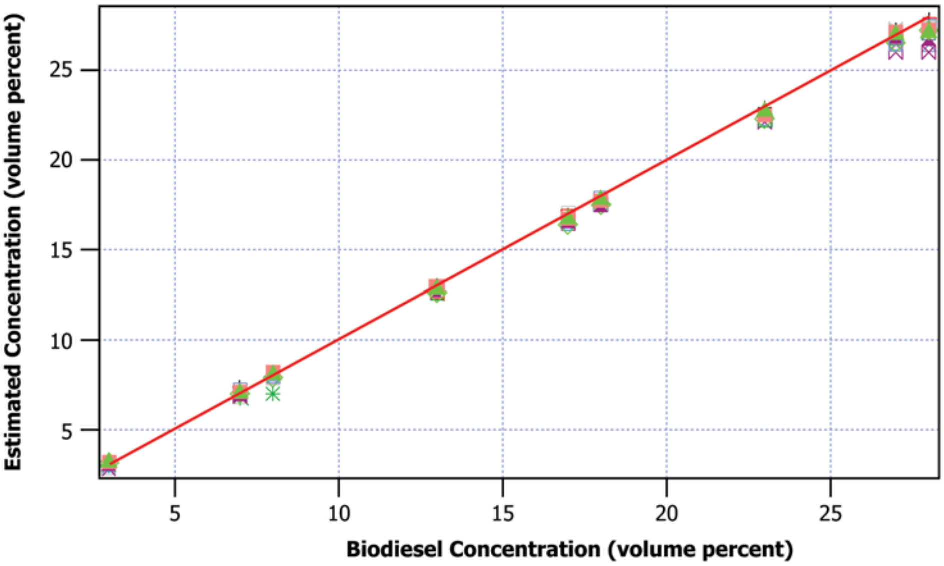

FIG. A2.3 Biodiesel in Diesel Fuel, Cetane #42.5, Aromatics Content 29 % by Volume

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