Designation: PS 121 – 99

Provisional Specification for Biodiesel Fuel (B100) Blend Stock for Distillate Fuels[1]

This provisional standard is issued under the fixed designation PS 121; the number immediately following the designation indicates the year of original adoption.

1. Scope

1.1 This provisional specification[2] covers low sulfur biodiesel (B100) for use as a blend component with diesel fuel oils defined by Specification D 975 Grades 1-D, 2-D, and low sulfur 1-D and 2-D.[3]

1.2 This specification, unless otherwise provided by agreement between the purchaser and the supplier, prescribes the required properties of biodiesel fuel at the time and place of delivery.

1.3 Nothing in this specification shall preclude observance of federal, state, or local regulations which may be more restrictive.

NOTE 1—The generation and dissipation of static electricity can create problems in the handling of distillate fuel oils with which biodiesel may be blended. For more information on the subject, see Guide D 4865.

2. Referenced Documents

2.1 *ASTM Standards:*

D 93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester[4]
D 130 Test Method for Detection of Copper Corrosion from Petroleum Products by the Copper Strip Tarnish Test[4]
D 189 Test Method for Conradson Carbon Residue of Petroleum Products[4]
D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)[4]
D 524 Test Method for Ramsbottom Carbon Residue of Petroleum Products[4]
D 613 Test Method for Cetane Number of Diesel Fuel Oil[5]
D 664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration[4]
D 874 Test Method for Sulfated Ash from Lubricating Oils and Additives[4]
D 974 Test Method for Acid and Base Number by Color-Indicator Titration[4]
D 975 Specification for Diesel Fuel Oils[4]
D 976 Test Method for Calculated Cetane Index of Distillate Fuels[4]
D 1266 Test Method for Sulfur in Petroleum Products (Lamp Method)[4]
D 1796 Test Method for Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure)[4]
D 2500 Test Method for Cloud Point of Petroleum Oils[4]
D 2622 Test Method for Sulfur in Petroleum Products by Wavelength Dispersive X-Ray Fluorescence Spectrometry[6]
D 2709 Test Method for Water and Sediment in Middle Distillate Fuels by Centrifuge[6]
D 3117 Test Method for Wax Appearance Point of Distillate Fuels[6]
D 3120 Test Method for Trace Quantities of Sulfur in Light Liquid Petroleum Hydrocarbons by Oxidative Microcoulometry[6]
D 3242 Test Method for Acidity in Aviation Turbine Fuel[6]
D 3828 Test Method for Flash Point by Small Scale Closed Tester[6]
D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[6]
D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products[6]
D 4294 Test Method for Sulfur in Petroleum and Petroleum Products by Energy Dispersive X-Ray Fluorescence Spectrometry[6]
D 4530 Test Method for Determination of Carbon Residue (Micro Method)[6]
D 4737 Test Method for Calculated Cetane Index by Four Variable Equation[6]
D 4865 Guide for Generation and Dissipation of Static Electricity in Petroleum Fuel Systems[6]

2.2 *Government Standards*[7]

40 CFR Part 79—Registration of Fuels and Fuel Additives Section 211(b) Clean Air Act

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

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

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[1] This provisional specification is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.E on Burner, Diesel, and Gas Turbine Fuels.

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[2] Provisional standards exist for two years subsequent to the approval date.

[3] This fuel complies with 40 CFR Part 79.

[4] *Annual Book of ASTM Standards*, Vol 05.01.

[5] *Annual Book of ASTM Standards*, Vol 05.04.

[6] *Annual Book of ASTM Standards*, Vol 05.02.

[7] Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

3.1.1.1 *Discussion*—Biodiesel, as defined above, is registered with the US EPA as a fuel and a fuel additive under Section 211(b) of the Clean Air Act. There is, however, other usage of the term biodiesel in the market place. Due to its EPA registration and the wide spread commercial use of the term biodiesel in the US market place, the term biodiesel will be maintained for this specification.

3.1.1.2 *Discussion*—Biodiesel is typically produced by a reaction of a vegetable oil or animal fat with an alcohol such as methanol or ethanol in the presence of a catalyst to yield mono-alkyl esters and glycerin. The finished biodiesel derives approximately 10 % of its mass from the reacted alcohol. The alcohol used in the reaction may or may not come from renewable resources.

3.1.2 *biodiesel blend*, *n*—a blend of biodiesel fuel with petroleum-based diesel fuel designated BXX, where XX is the volume % of biodiesel.

3.1.3 *biodiesel fuel*, *n*—synonymn for *biodiesel.*

3.1.4 *diesel fuel*, *n*—a light or middle petroleum distillate fuel.

3.1.5 *free glycerin*, *n*—a measure of the amount of unbonded glycerin remaining in the fuel.

3.1.6 *total glycerin*, *n*—the sum of the free glycerin and the glycerin portion of any unreacted or partially reacted oil or fat.

4. Requirements

4.1 The biodiesel specified shall be mono-alkyl esters of long chain fatty acids derived from vegetable oils and animal fats.

4.2 A representative sample obtained using such techniques as Practice D 4057 or D 4177 shall be used for all analysis.

4.3 The biodiesel specified shall conform to the detailed requirements shown in Table 1.

NOTE 2—A considerable amount of experience exists in the US with a 20 % blend of biodiesel, primarily produced from soybean oil, with 80 % diesel fuel (B20). Experience with biodiesel produced from animal fat and other oils is similar. Although biodiesel (B100) can be used, blends of over 20 % biodiesel with diesel fuel (B20) should be evaluated on a case by case basis until further experience is available.

NOTE 3—In certain items of fuel injection equipment in compression ignition engines, such as rotary/distributor fuel pumps and injectors, the fuel functions as a lubricant as well as a source for combustion. Blending biodiesel fuel with diesel fuel typically adds lubricity.

5. Test Methods

5.1 The ASTM test methods identified in the specification are based on those for diesel fuel. These testing methods appear applicable for biodiesel testing. However precision and bias of these methods with biodiesel is unknown and is under evaluation.

5.2 The requirements enumerated in this specification shall be determined in accordance with the following methods.

5.2.1 *Flash Point*—Test Methods D 93, except where other methods are prescribed by law. Test Method D 3828 can also be used. Test Method D 93 shall be the referee method.

TABLE 1 Detailed Requirements for Biodiesel (B100)[A]

Property	Test Method[B]	Limits	Units
Flash point (closed cup)	D 93	100.0 min	°C
Water and sediment	D 2709	0.050 max	% volume
Kinematic viscosity, 40°C	D 445	1.9-6.0[C]	mm^2/s
Sulfated ash	D 874	0.020 max	% mass
Sulfur[D]	D 2622	0.05 max	% mass
Copper strip corrosion	D 130	No. 3 max	
Cetane number	D 613	40 min	
Cloud point	D 2500	Report to customer	°C
Carbon residue[E]	D 4530	0.050 max	% mass
Acid number	D 664	0.80 max	mg KOH/g
Free glycerin	[F]	0.020[G]	% mass
Total glycerin	[F]	0.240[G]	% mass

[A] To meet special operating conditions, modifications of individual limiting requirements may be agreed upon between purchaser, seller, and manufacturer.

[B] The test methods indicated are the approved referee methods. Other acceptable methods are indicated in 5.3.

[C] See X1.3.1.

[D] Other sulfur limits can apply in selected areas in the United States and in other countries.

[E] Carbon residue shall be run on the 100 % sample (see 5.2.10).

[F] See Annex 1 for test method. A gas chromatographic technique is being converted to a standard test method.

[G] The test method is under ASTM consideration by Subcommittee D02.04.OL

5.2.2 *Water and Sediment*—Test Method D 2709. Test Method D 1796 may also be used. Test Method D 2709 shall be the referee method.

5.2.3 *Viscosity*—Test Method D 445.

5.2.4 *Sulfated Ash*—Test Method D 874.

5.2.5 *Sulfur*—Test Method D 2622. Test Methods D 1266, D 3120 and D 4294 may also suitable for determining up to 0.05 % sulfur in biodiesel fuels. Test Method D 2622 shall be the referee test method.

5.2.6 *Corrosion*—Test Method D 130, 3 h test at 50°C.

5.2.7 *Cetane Number*—Test Method D 613.

5.2.8 *Cloud Point*—Test Method D 2500. Test Method D 3117 may also be used because the two are closely related. Test Method D 2500 shall be the referee test method.

5.2.9 *Acid Number*—Test Method D 664. Test Method D 3242 or D 974 may also be used. Test Method D 664 shall be the referee test method.

5.2.10 *Carbon Residue*—Test Method D 4530. A 100 % sample shall replace the 10 % residual, with percent residue in original sample reported using the 10 % residual calculation (see X1.9.1). Test Method D 189 or D 524 may also be used. Test Method D 4530 shall be the referee method.

5.2.11 *Total Glycerin*—See Annex A1

5.2.12 *Free Glycerin*—See Annex A1.

6. Workmanship

6.1 The biodiesel fuel shall be visually free of undissolved water, sediment, and suspended matter.

7. Keywords

7.1 alternative fuel; biodiesel fuel; diesel fuel oil; fuel oil; renewable resource; specification

ANNEX

(Mandatory Information)

A1. TEST METHOD FOR DETERMINATION OF FREE AND TOTAL GLYCERINE IN B 100 BIODIESEL METHYL ESTERS BY GAS CHROMATOGRAPHY

A1.1 Scope

A1.1.1 This test method provides for the quantitative determination of free and total glycerin in B100 methyl esters by gas chromatography. The range of detection for free glycerin is 0.005 to 0.05 mass %, and total glycerin from 0.05 to 0.5 mass %. This procedure is not applicable to vegetable oil methyl esters obtained from lauric oils, such as coconut oil and palmkernel oil.

A1.1.2 The values stated in SI units are to be regarded as the standard.

A1.1.3 *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.*

A1.2 Referenced Documents

A1.2.1 *ASTM Standards:*

D 4307 Practice for Preparation of Liquid Blends for Use as Analytical Standards[6]

E 355 Practice for Gas Chromatography Terms and Relationships[8]

E 594 Practice for Testing Flame Ionization Detectors Used in Gas or Supercritical Fluid Chromatography[8]

A1.3. Terminology

A1.3.1 *Definitions*:

A1.3.1.1 This test method makes reference to many common gas chromatographic procedures, terms, and relationships. Detailed definitions can be found in Practices E 355 and E 594.

A1.3.2 *Definitions of Terms Specific to This Standard:*

A1.3.2.1 *biodiesel (B100), n*—fuel comprised of mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fats.

A1.3.2.2 *total glycerin*, *n*—is the sum of free and bonded glycerin.

A1.3.2.3 *bonded glycerin*, *n*—is the glycerin portion of the mono-, di-, and triglyceride molecules.

A1.4 Summary of Test Method

A1.4.1 The sample is analyzed by gas chromatography, after silyating with N-methyl-N-trimethylsilyltrifluoracetamide (MSTFA). Calibration is achieved by the use of two internal standards and four reference materials. Mono-, di-, and triglycerides are determined by comparing to monoolein, diolein, and triolein standards respectively. Average conversion factors are applied to the mono-, di-, and triglycerides to calculate the bonded glycerin content of the sample.

A1.5 Significance and Use

A1.5.1 Free and bonded glycerin content reflects the quality of biodiesel. A high content of free glycerin may cause problems during storage, or in the fuel system, due to separation of the glycerin. A high total glycerin content can lead to injector fouling and may also contribute to the formation of deposits at injection nozzles, pistons, and valves.

A1.6 Apparatus

A1.6.1 *Chromatographic System*—See Practice D 355 for specific designations and definitions.

A1.6.1.1 *Gas Chromatograph (GC)*—the system must be capable of operating at the conditions given in Table A1.1.

A1.6.1.2 *Column*, open tubular column with a 5 % phenylpolydimethylsiloxane bonded and cross linked phase internal coating. The column should have an upper temperature limit of at least 400°C. Columns, either 10 m or 15 m in length, with a 0.32 mm internal diameter, and a 0.1 µm film thickness have been found satisfactory. Any column with better or equivalent chromatographic efficiency and selectivity can be used. It is recommended that a 2 to 5 metre 0.53 mm high temperature guard column be installed from the injector to the analytical column. This allows the use of autoinjectors and also increases column life.

A1.6.2 *Electronic Data Acquisition System:*:

A1.6.2.1 *Integrator or Computer*, capable of providing real time graphic and digital presentation of the chromatographic data is recommended for use. Peak areas and retention times shall be measured by computer or electronic integration.

A1.6.2.2 This device must be capable of performing multilevel internal-standard-type calibrations and be able to calculate the correlation coefficient (r^2) and internal standard calculations for each data set.

A1.7 Reagents and Materials

A1.7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee

TABLE A1.1 Operating Conditions

Injector		
Cool on column injection		
Sample size	1 µl	
Column Temperature Program		
Initial temperature	50°C	hold 1 min
Rate 1	15°C / min to 180°C	
Rate 2	7°C / min to 230°C	
Rate 3	30°C / min 380°C	hold 10 min
Type	Flame ionization	
Temperature	380°C	
Carrier Gas		
Type	Hydrogen or helium	measured at 50°C
Flow rate	3 mL/min	

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[8] *Annual Book of ASTM Standards*, Vol 14.02/

on Analytical Reagents of the American Chemical Society where such specifications are available.[9] Other grades may be used provided it is first ascertained that the reagent is of sufficient purity to permit its use without lessening the accuracy of the determination.

A1.7.2 *n-Heptane*, reagent grade.

A1.7.3 *N-Methyl-N-trimethylsilyltrifluoroacetamide (MSTFA)*, reagent grade.

A1.7.4 *Pyridine*, reagent grade.

A1.7.5 *Carrier Gas*, hydrogen or helium of high purity. Additional purification is recommended by the use of molecular sieves or other suitable agents to remove water, oxygen, and hydrocarbons. Available pressure must be sufficient to ensure a constant carrier gas flow rate.

A1.7.6 *Microlitre Syringes*, 100 µl and 250 µl capacity.

A1.7.7 *Screw Cap Vials*, with polytetrafluoroethylene (PTFE)-faced septa, 10 mL capacity.

A1.8 Preparation of Apparatus

A1.8.1 Install and condition the column in accordance with manufacturer or supplier's instructions. After conditioning, attach column outlet to flame ionization detector inlet and check for leaks throughout the system. If leaks are found, tighten or replace fittings and recheck for leaks before proceeding.

A1.9 Calibration and Standardization

A1.9.1 *Preparation of Calibration Standards*—Prepare standards using fresh compounds listed in Table A1.2 according to Practice D 4307. Weigh the components directly into the volumetric flasks specified and record the weight to the nearest 0.1 mg. Dilute the volumetric flasks to mark with pyridine. Store the calibration standards in a refrigerator when not in use.

A1.9.2 *Standard Solutions*—Prepare the five standard solutions in Table A1.3 by transferring the specified volumes by means of microlitre syringes to 10 mL septa vials. Add to each of the five standard solutions 100 µL of MSTFA. Close the vial and shake. Allow the vial to stand for 15 to 20 min at room temperature. Add approximately 8 mL n-Heptane to the vial and shake.

TABLE A1.2 Stock Solutions

Compound	CAS No.	Approximate Weight (mg)	Volumetric Flask Size (mL)
Glycrein	56-81-5	25	50
1-Mono [*cis*-9-octadecenoyl]-*rac*-glycerol (monoolein)	111-03-5	50	10
1,3-Di [*cis*-octadecenoyl]glycerol (diolein)	2465-32-9	50	10
1,2,3-Tri [*cis*-octadecenoy]glycerol (triolein)	122-32-7	50	10
(S) - (-) -1,2,4-Butanetriol	42890-76-6	25	25
1,2,3-Tridecanolylglycerol (tricaprin)	321-71-6	80	10

TABLE A1.3 Standard Solutions

Standard Solution Number	1	2	3	4	5
µL of glycerin stock solution	10	30	50	70	100
µL of monoolein stock solution	20	50	100	150	200
µL of diolein stock solution	10	20	40	70	100
µL of triolein stock solution	10	20	40	70	100
µL of butanetriol stock solution	100	100	100	100	100
µL of tricaprin stock solution	100	100	100	100	100

A1.9.3 *Chromatographic Analysis*—If using an automatic sampler, transfer an aliquot of the solution into a glass GC vial and seal with a TFE-fluorocarbonlined cap.

A1.9.4 *Standardization*:

A1.9.5 Analyze the calibration standards under the same operating conditions as the sample solutions. Inject 1 µL of the reaction mixture into the cool on-column injection port and start the analysis. Obtain a chromatogram and peak integration report. For each reference substance, determine the response ratio (rsp_i) and amount ratio (amt_i) for each component using Eq A1.1 and A1.2

$$rsp_i = (A_i / A_s) \tag{A1.1}$$

where:

A_i = area of reference substance, and
A_s = area of internal standard.

$$amt_i = (W_i / W_s) \tag{A1.2}$$

where:

W_i = mass of reference substance, and
W_s = mass of internal standard.

Prepare a calibration curve for each reference component by plotting the response ratios (rsp_i), as the y-axis, versus the amount ratios (amt_i), as the x-axis.

A1.9.6 Calculate the correlation coefficient r^2 value for each reference component in the calibration set using Eq A1.3. The r^2 value should be at least 0.99 or greater. If the above criteria for r^2 are not met, rerun the calibration or check instrument parameters and hardware.

$$r^2 = \frac{[\Sigma\, xy^2]}{[\Sigma\, x^2]\,[\Sigma\, y^2]} \tag{A1.3}$$

where:

$$\bar{x} = X_i - x \tag{A1.4}$$

$$\bar{y} = Y_i - y \tag{A1.5}$$

and:

X_i = amt_i ratio data point,
$\bar{x}$ = average values for all amt_i data points
Y_i = corresponding rsp_i data points,
$\bar{y}$ = average values for all rsp_i data points.

A1.9.7 *Calibration Functions*—For each reference calibration functions are calculated in the form:

$$W_x / W_{is} = b_x \times (A_x / A_{is}) + a_x \tag{A1.6}$$

where:

W_x = weight of reference substance, mg,
W_{is} = weight of internal standard, mg,
A_x = peak area of reference substance,

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[9] Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see Analar 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.

A_{is} = peak area of internal standard,
b_x = slope of the calibration function, and
a_x = intercept of the calibration function.

A1.10 Procedure

A1.10.1 Set the instrument operating variables to the values specified in Table A1.1. Weigh to the nearest 0.1 mg approximately 100 mg of sample directly into a 10 mL septa vial. Using microlitre syringes, add exactly 100 µL of each internal standard and MSTFA. Shake the vials, and allow to set for 15 to 20 min at room temperature. Add approximately 8 mL of n-Heptane to the vial and shake.

A1.10.2 Inject 1 µL of the reaction mixture into the cool on-column injection port and start the analysis. Obtain a chromatogram and peak integration report.

A1.10.3 *Peak Identification*—Identify peaks by comparison of retention times to the standards. For identification of additional peaks, use the relative retention times given in Table A1.4 and the reference chromatograms given in Fig. A1.1. The mono-, di, and triglycerides are separated according to carbon numbers (CN).

A1.10.4 Monoglycerides consist of the four overlapping peaks with relative retention times (RRT) of 0.76 and 0.83 to 0.86 with respect to the internal standard tricaprin. A pair of peaks, methyl esters with a carbon number of 24, may appear with RRT of 0.80 to 0.82, and should not be included in the calculation of monoglycerides.

A1.10.5 Diglycerides are also primarily separated according to carbon number, but due to varying double bonds in the molecules, baseline resolution of the peaks does not occur. The grouping of 3 to 4 peaks with RRT of 1.05 to 1.09 (CN 34, 36, and 38) shall be attributed to diglycerides. Carbon number also separates triglycerides. Peaks with RRT of 1.16 to 1.31 (CN 52, 54, 56, and 58) should be included in the calculation.

A1.11 Calculation and Report

A1.11.1 After identifying the peaks, measure the areas of the peaks identified as glycerin, mono, di-, and triglycerides. Using the slope and y-intercept of the calibration functions, calculate the mass of each as follows:

A1.11.1.1 *Glycerin:*

$$G = (b_g \times A_g/A_{is1} + a_g) \times W_{is1} \times 100/W \tag{A1.7}$$

where:
G = mass percentage of glycerin in sample,
A_g = peak area of glycerin,
A_{is1} = peak area of Internal Standard 1,
W_{is1} = weight of Internal Standard 1 in mg,
W = weight of sample in mg,
b_g = slope of the calibration function,
a_g = intercept of the calibration function.

A1.11.1.2 *Individual Glycerides:*

$$Gl_i = (b_o \times A_{gli}/A_{is2} + a_{o1}) \times W_{is2} \times 100/W \tag{A1.8}$$

where:
Gl_i = mass percentage of individual glycerides in sample,
A_{gli} = peak area of individual glyceride,
A_{is2} = peak area of Internal Standard 2,
W_{is2} = weight of Internal Standard 2 , mg,
W = weight of sample, mg,
b_{ol} = slope of the calibration function for mono, di-, or triolein, and
a_{ol} = intercept of the calibration function for mono, di, or triolein.

A1.11.1.3 *Calculation of Total Glycerin:*

$$\text{total glycerin} = \text{free glycerin} + \text{bound glycerin} \tag{A1.9}$$

where:
free glycerin = glycerin determined in Eq A1.7,
bound glycerin = Σ (Gl_M, Gl_D, Gl_T)

where:
Gl_M = 0.2591 × Σ monoglyceride, mass % determined in Eq A1.8,
Gl_D = 0.1488 × Σ diglyceride, mass % determined in Eq A1.8, and
Gl_T = 0.1044 × Σ triglyceride, mass % determined in Eq A1.8.

A1.11.2 Report the free and total glycerin to the nearest 0.001 mass %.

TABLE A1.4 Approximate Relative Retention Times

Component	Use Internal Standard	Relative Retention Time
Glycerin	1	0.85
1,2,4 Butanetriol Internal Standard 1		1.00
Monopalmitin	2	0.76
Monoolein, monolinolein monolinolenin, and monostearin	2	0.83-0.86
Tricaprin Internal Standard 2		1.00
Diglycerides	2	1.05-1.09
Triglycerides	2	1.16-1.31

A1.12 Precision and Bias

A1.12.1 *Precision*—The precision of this test method is being determined.

A1.12.2 *Bias*—Since there is no accepted reference material suitable for determining bias for the procedure in this test method, bias has not been determined.

A1.13 Keywords

A1.13.1 biodiesel; free glycerin; Gas Chromatography 4; methyl esters; total glycerin

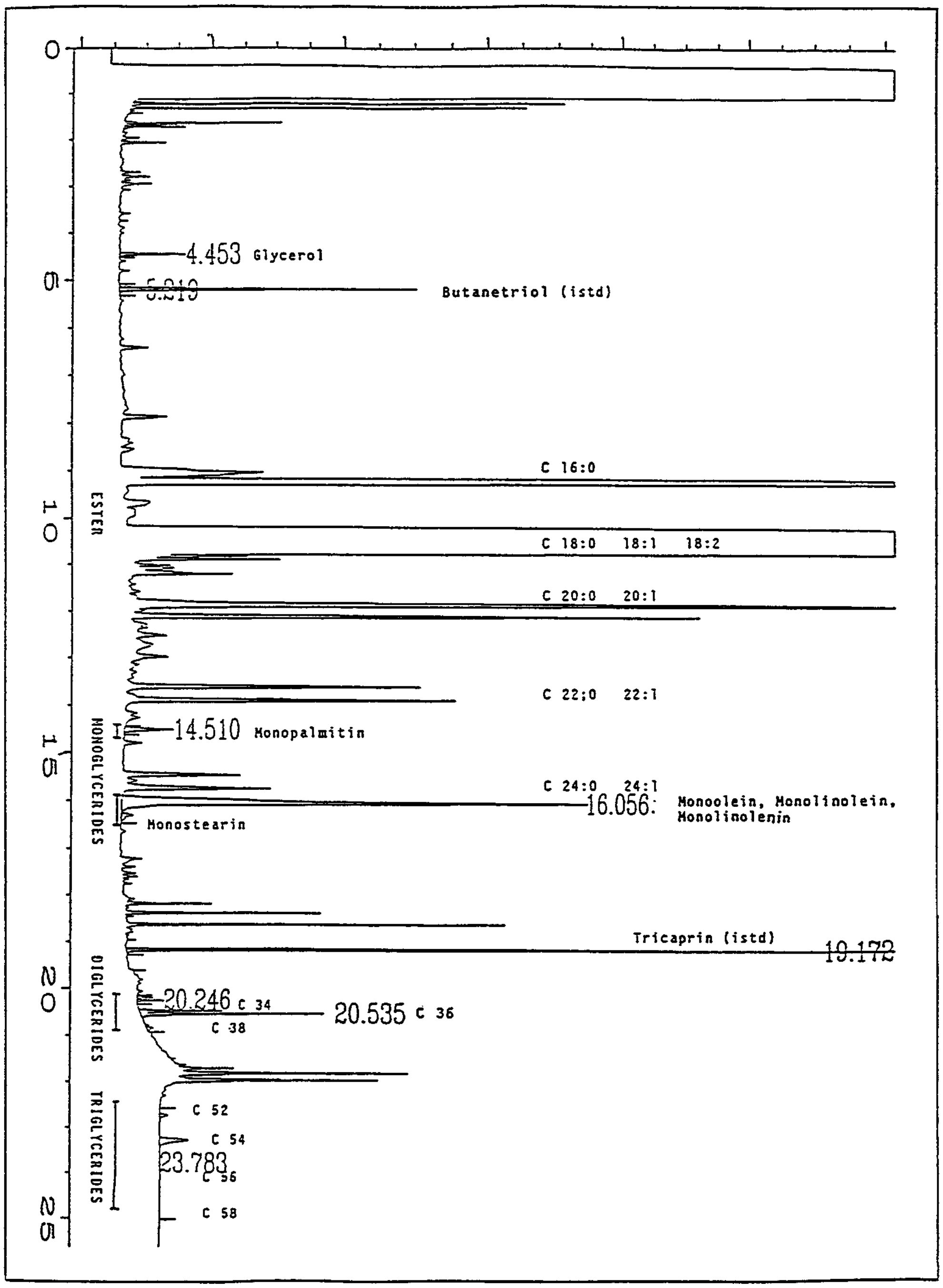

FIG. A1.1 Reference Chromatograms

APPENDIXES

(Nonmandatory Information)

X1. SIGNIFICANT OF PROPERTIES SPECIFIED FOR BIODIESEL FUEL

X1.1 Introduction

X1.1.1 The properties of commercial biodiesel fuel depends upon the refining practices employed and the nature of the renewable lipids from which it is produced. Biodiesel, for example, can be produced from a variety of vegetable oils or animal fats which produce similar volatility characteristics and combustion emissions with varying cold flow properties.

X1.2 Flash Point

X1.2.1 The flash point for biodiesel is used as the mechanism to limit the level of unreacted alcohol remaining in the finished fuel.

X1.2.2 The flash point is also of importance in connection with legal requirements and safety precautions involved in fuel handling and storage, and is normally specified to meet insurance and fire regulations.

X1.3 Viscosity

X1.3.1 For some engines it may be advantageous to specify a minimum viscosity because of power loss due to injection pump and injector leakage. Maximum allowable viscosity, on the other hand, is limited by considerations involved in engine design and size, and the characteristics of the injection system. The upper limit for the viscosity of biodiesel (6.0 mm^2/s at 40°C) is higher than the maximum allowable viscosity in Specification D 975 Grade 2-D and 2-D low sulfur (4.1 mm/s at 40°C). Blending biodiesel with diesel fuel close to its upper limit could result in a biodiesel blend with viscosity above the upper limits contained in Specification D 975.

X1.4 Sulfated Ash

X1.4.1 Ash-forming materials may be present in biodiesel in three forms: (*1*) abrasive solids, (*2*) soluble metallic soaps, and (*3*) unremoved catalysts. Abrasive solids and unremoved catalysts can contribute to injector, fuel pump, piston and ring wear, and also to engine deposits. Soluble metallic soaps have little effect on wear but may contribute to filter plugging and engine deposits.

X1.5 Sulfur

X1.5.1 The effect of sulfur content on engine wear and deposits appears to vary considerably in importance and depends largely on operating conditions. Fuel sulfur can also affect emissions control systems performance and various limits on sulfur have been imposed for environmental reasons.

X1.6 Copper Strip Corrosion

X1.6.1 This test serves as a measure of possible difficulties with copper and brass or bronze parts of the fuel system. The presence of acids or sulfur-containing compounds can tarnish the copper strip, thus indicating the possibility for corrosion.

X1.7 Cetane Number

X1.7.1 Cetane number is a measure of the ignition quality of the fuel and influences white smoke and combustion roughness. The cetane number requirements depend on engine design, size, nature of speed and load variations, and on starting and atmospheric conditions.

X1.7.2 The calculated cetane index, Test Methods D 976 or D 4737, may not be used to approximate the cetane number with biodiesel or its blends. There is no substantiating data to support the calculation of cetane index with biodiesel or biodiesel blends.

X1.8 Cloud Point

X1.8.1 Cloud point is of importance in that it defines the temperature at which a cloud or haze of crystals appears in the fuel under prescribed test conditions which generally relates to the temperature at which crystals begin to precipitate from the fuel in use. The cloud point of biodiesel and its impact on the cold flow properties of the resulting blend should be monitored by the user to ensure trouble free operation in cold climates. For further information, consult Appendix X4 of Specification D 975.

X1.9 Carbon Residue

X1.9.1 Carbon residue gives a measure of the carbon depositing tendencies of a fuel oil. While not directly correlating with engine deposits, this property is considered an approximation. Although biodiesel is in the distillate boiling range, most biodiesel boils at approximately the same temperature and it is difficult to leave a 10 % residual upon distillation. Thus, a 100 % sample is used to replace the 10 % residual sample, with the calculation executed as if it were the 10 % residual. Parameter E (final weight flask charge/original weight flask charge) in 8.1.2 of D 4530-93 is a constant 20/200.

X1.10 Acid Number

X1.10.1 The acid number is used to determine the level of free fatty acids or processing acids that may be present in biodiesel. Biodiesel with a high acid number has been shown to increase fueling system deposits and may increase the likelihood for corrosion.

NOTE X1.1—Acid number measures a different phenomenon for biodiesel than petrodiesel. Increased recycle temperatures in new fuel system designs may increase filter plugging potential.

X1.11 Free Glycerin

X1.11.1 The free glycerin method is used to determine the level of unbonded glycerin in the fuel. High levels of free glycerin can cause injector deposits, as well as clogged fueling systems, and result in a buildup of free glycerin in the bottom of storage and fueling systems.

X1.12 Total Glycerin

X1.12.1 The total glycerin method is used to determine the level of unbonded and bonded glycerin in the fuel. Low levels of total glycerin ensure that high conversion of the oil or fat into its mono-alkyl esters has taken place. High levels of mono-, di-, and triglycerides can cause injector deposits and may adversely affect cold weather operation and filter plugging.

X2. STORAGE STABILITY

X2.1 Oxidation stability is a procedure used to determine the likely storage life and potential degradation of a fuel in storage. Appropriate test methods for measuring the oxidative stability for biodiesel, and its correlation to long term storage as well as engine and fueling system performance are currently being evaluated.

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