



Standard Specification for Biodiesel Fuel (B100) Blend Stock for Distillate Fuels¹

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1. Scope

1.1 This specification 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.

1.2 Biodiesel may be blended with fuel oils whose sulfur or aromatic levels are outside Specification D 975 Grades 1-D, 2-D, and low sulfur 1-D and 2-D, provided the finished mixture meets pertinent national and local specifications and requirements for these properties.

1.3 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.

1.4 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 Tester²
- D 130 Test Method for Detection of Copper Corrosion from Petroleum Products by the Copper Strip Tarnish Test²
- D 189 Test Method for Conradson Carbon Residue of Petroleum Products²
- D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (the Calculation of Dynamic Viscosity)²
- D 524 Test Method for Ramsbottom Carbon Residue of Petroleum Products²
- D 613 Test Method for Cetane Number of Diesel Fuel Oil³
- D 664 Test Method for Acid Number of Petroleum Products

- by Potentiometric Titration²
- D 874 Test Method for Sulfated Ash from Lubricating Oils and Additives²
- D 974 Test Method for Acid and Base Number by Color-Indicator Titration²
- D 975 Specification for Diesel Fuel Oils²
- D 976 Test Methods for Calculated Cetane Index of Distillate Fuels²
- D 1160 Test Method for Distillation of Petroleum Products at Reduced Pressure²
- D 1266 Test Method for Sulfur in Petroleum Products (Lamp Method)²
- D 1796 Test Method for Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure)²
- D 2274 Test Method for Oxidation Stability of Distillate Fuel Oil (Accelerated Method)²
- D 2500 Test Method for Cloud Point of Petroleum Products²
- D 2622 Test Method for Sulfur in Petroleum Products by Wavelength Dispersive X-ray Fluorescence Spectrometry²
- D 2709 Test Method for Water and Sediment in Middle Distillate Fuels by Centrifuge²
- D 2880 Specification for Gas Turbine Fuel Oils²
- D 3117 Test Method for Wax Appearance Point of Distillate Fuels²
- D 3120 Test Method for Trace Quantities of Sulfur in Light Liquid Petroleum Hydrocarbons by Oxidative Microcoulometry²
- D 3242 Test Method for Acidity in Aviation Turbine Fuel⁴
- D 3828 Test Method for Flash Point by Small Scale Closed Tester⁴
- D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products⁴
- D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products⁴
- D 4294 Test Method for Sulfur in Petroleum and Petroleum Products by Energy Dispersive X-ray Fluorescence Spectrometry⁴
- D 4530 Test Method for Determination of Carbon Residue (Micro Method)⁴
- D 4737 Test Method for Calculated Cetane Index by Four Variable Equation⁴

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² *Annual Book of ASTM Standards*, Vol 05.01.

³ *Annual Book of ASTM Standards*, Vol 05.05.

⁴ *Annual Book of ASTM Standards*, Vol 05.02.

- D 4865 Guide for Generation and Dissipation of Static Electricity in Petroleum Fuel Systems⁴
- D 4951 Test Method for Determination of Additive Elements in Lubricating Oils by Inductively Coupled Plasma Atomic Emission Spectrometry⁴
- D 5453 Test Method for Determination of Total Sulfur in Light Hydrocarbons, Motor Fuels and Oils by Ultraviolet Fluorescence⁵
- D 6217 Test Method for Particulate Contamination in Middle Distillate Fuels by Laboratory Filtration⁵
- D 6450 Test Method for Flash Point by Continuously Closed Cup (CCCFP) Tester⁶
- D 6469 Guide for Microbial Contamination in Fuels and Fuel Systems⁶
- D 6584 Test Method for Determination of Free and Total Glycerine in B-100 Biodiesel Methyl Esters by Gas Chromatography⁶

2.2 Government Standard:

- 40 CFR Part 79 Registration of Fuels and Fuel Additives Section 211(b) Clean Air Act⁷

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.

3.1.1.1 Discussion—Biodiesel, as defined above, is registered with the U.S. 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 marketplace. Due to its EPA registration and the widespread commercial use of the term biodiesel in the U.S. marketplace, 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, which is removed. 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, 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 volume percentage of biodiesel fuel in the blend.

3.1.3 biodiesel fuel, n—synonym 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 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 Unless otherwise specified, samples for analysis shall be taken by the procedure described in Practices D 4057 or D 4177.

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 U.S. 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—The user should consult the equipment manufacturer or owner’s manual regarding the suitability of using biodiesel or biodiesel blends in a particular engine or application.

5. Test Methods

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

5.1.1 Flash Point—Test Methods D 93, except where other methods are prescribed by law. Test Methods D 3828 or D 6450 can also be used. The precision and bias of Test Methods D 3828 and D 6450 with biodiesel is not known and is currently under investigation. Test Method D 93 shall be the referee method.

5.1.2 Water and Sediment—Test Method D 2709. Test Method D 1796 may also be used. Test Method D 2709 shall be the referee method. The precision and bias of these test methods with biodiesel is not known and is currently under investigation.

5.1.3 Viscosity—Test Method D 445.

5.1.4 Sulfated Ash—Test Method D 874.

TABLE 1 Detailed Requirements for Biodiesel (B100)^A

Property	Test Method ^B	Limits	Units
Flash point (closed cup)	D 93	130.0 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 5453	0.05 max	% mass
Copper strip corrosion	D 130	No. 3 max	
Cetane number	D 613	47 min	
Cloud point	D 2500	Report ^E	°C
Carbon residue ^F	D 4530	0.050 max	% mass
Acid number	D 664	0.80 max	mg KOH/g
Free glycerin	D 6584	0.020	% mass
Total glycerin	D 6584	0.240	% mass
Phosphorus content	D 4951	0.001 max	% mass
Distillation temperature, Atmospheric equivalent temperature, 90 % recovered	D 1160	360 max	°C

^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 5.1.

^C See X1.3.1. The 6.0 mm²/s upper viscosity limit is higher than petrodiesel and should be taken into consideration when blending.

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

^E The cloud point of biodiesel is generally higher than petrodiesel and should be taken into consideration when blending.

^F Carbon residue shall be run on the 100 % sample (see 5.1.10).

⁵ Annual Book of ASTM Standards, Vol 05.03.

⁶ Annual Book of ASTM Standards, Vol 05.04.

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

5.1.5 *Sulfur*—Test Method D 5453. Other test methods may also be suitable for determining up to 0.05 % sulfur in biodiesel fuels such as Test Methods D 1266, D 2622, D 3120 and D 4294 but may provide falsely high results (see X1.5) although their precision and bias with biodiesel is unknown. Test Method D 5453 shall be the referee test method.

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

5.1.7 *Cetane Number*—Test Method D 613.

5.1.8 *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. The precision and bias of these test methods for biodiesel is not known and is currently under investigation.

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

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

5.1.11 *Total Glycerin*—Test Method D 6584.

5.1.12 *Free Glycerin*—Test Method D 6584.

5.1.13 *Phosphorus Content*—Test Method D 4951.

5.1.14 *Distillation Temperature, Reduced Pressure*—Test Method D 1160.

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

APPENDIXES

(Nonmandatory Information)

X1. SIGNIFICANCE 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.

X1.2.3 The flash point specification for biodiesel is intended to be 100°C minimum. Typical values are over 160°C. Due to high variability with Test Method D 93 as the flash point approaches 100°C, the flash point specification has been set at 130°C minimum to ensure an actual value of 100°C minimum. Improvements and alternatives to Test Method D 93 are being investigated. Once complete, the specification of 100°C minimum may be reevaluated.

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 mm²/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. B100 is essentially sulfur-free.

NOTE X1.1—Test Method D 5453 should be used with biodiesel. Use of other test methods may provide falsely high results when analyzing B100 with extremely low sulfur levels (less than 5 ppm). Biodiesel sulfur analysis from RR D02-1480⁸, *Biodiesel Fuel Cetane Number Testing Program, January-April, 1999*, using Test Method D 2622 yielded falsely high results due to the presence of the oxygen in the biodiesel. Sulfur results using Test Method D 2622 were more accurate with B20 than with B100 due to the lower oxygen content of B20. Potential improvements to Test Method D 2622 may provide more accurate values in the future.

⁸ Supporting data are available from ASTM International Headquarters. Request RR: D02-1480.



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. Biodiesel generally has a higher cloud point than petrodiesel. 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 Test Method 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 X1.2—Acid number measures a different phenomenon for biodiesel than petrodiesel. The acid number for biodiesel measures free fatty acids or degradation by-products not found in petrodiesel. Increased recycle temperatures in new fuel system designs may accelerate fuel degradation which could result in high acid values and increased filter plugging potential.

X1.11 Free Glycerin

X1.11.1 The free glycerin method is used to determine the level of 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 glycerin in the fuel and includes the free glycerin and the glycerine portion of any unreacted or partially reacted oil or fat. 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.

X1.13 Phosphorus Content

X1.13.1 Phosphorus can damage catalytic converters used in emissions control systems and its level must be kept low. Catalytic converters are becoming more common on diesel-powered equipment as emissions standards are tightened, so low phosphorus levels will be of increasing importance. Biodiesel produced from U.S. sources has been shown to have low phosphorus content (below 1 ppm) and the specification value of 10 ppm maximum is not problematic. Biodiesel from other sources may or may not contain higher levels of phosphorus and this specification was added to ensure that all biodiesel, regardless of the source, has low phosphorus content.

X1.14 Reduced Pressure Distillation

X1.14.1 Biodiesel exhibits a boiling point rather than a distillation curve. The fatty acids chains in the raw oils and fats from which biodiesel is produced are mainly comprised of straight chain hydrocarbons with 16 to 18 carbons that have similar boiling temperatures. The atmospheric boiling point of biodiesel generally ranges from 330 to 357°C, thus the specification value of 360°C is not problematic. This specification was incorporated as an added precaution to ensure the fuel has not been adulterated with high boiling contaminants.

NOTE X1.3—The density of biodiesel meeting the specifications in Table 1 falls between 0.86 and 0.90, with typical values falling between 0.88 and 0.89. Since biodiesel density falls between 0.86 and 0.90, a separate specification is not needed. The density of raw oils and fats is similar to biodiesel, therefore use of density as an expedient check of fuel quality may not be as useful for biodiesel as it is for petrodiesel. This section has been added to provide users and engine interests with this information.

NOTE X1.4—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 petroleum based compression-ignition fuel typically improves fuel lubricity.

X2. LONG-TERM STORAGE OF BIODIESEL

X2.1 Scope

X2.1.1 This appendix provides guidance for consumers of biodiesel (B100) who may wish to store quantities of fuels for extended periods. Consistently successful long-term fuel storage requires attention to fuel selection, storage conditions, and monitoring of properties prior to and during storage. This appendix is directed toward biodiesel (B100) and may be more or less applicable to blends of biodiesel with petrodiesel.

X2.1.2 Normally produced biodiesel has adequate stability properties to withstand normal storage without the formation of troublesome amounts of insoluble degradation products, although data suggests some biodiesel may degrade faster than petrodiesel. Biodiesel that is to be stored for prolonged periods should be selected to avoid formation of sediments, high acid numbers, and high viscosities that can clog filters, affect fuel pump operation or plug combustor nozzles or injectors. The selection of biodiesel should result from supplier-user discussions.

X2.1.3 These suggested practices are general in nature and should not be considered substitutes for any requirement imposed by the warranty of the distillate fuel equipment manufacturers or by federal, state, or local government regulations. Although they cannot replace knowledge of local conditions or good engineering and scientific judgment, these suggested practices do provide guidance in developing an individual fuel management system for the biodiesel fuel user. They include suggestions in the operation and maintenance of existing fuel storage and handling facilities and for identifying where, when, and how fuel quality should be monitored.

X2.2 Terminology

X2.2.1 *bulk fuel*—fuel in the storage facility in quantities over 50 gallons.

X2.2.2 *combustor fuel*—fuel entering the combustion zone of the burner or engine after filtration or other treatment of bulk fuel.

X2.2.3 *fuel contaminants*—foreign materials that make fuel less suitable or unsuitable for the intended use. Fuel contaminants include materials introduced subsequent to the manufacture of fuel and fuel degradation products.

X2.2.4 *fuel-degradation products*—those materials formed in fuel after it is produced. Insoluble degradation products may combine with other fuel contaminants to reinforce deleterious effects. Soluble degradation products (acids and gums) may be more or less volatile than the fuel and may cause an increase in injector and nozzle deposits. The formation of degradation products may be catalyzed by contact with metals, especially those containing copper and, to a lesser extent, iron.

X2.2.5 *long-term storage*—storage of fuel for longer than 6 months after it is received by the user.

X2.3 Fuel Selection

X2.3.1 The stability properties of biodiesel are not fully understood and appear to depend on the vegetable oil and animal fat sources, severity of processing, and whether addi-

tional production plant treatment has been carried out or stability additives are present.

X2.3.2 The composition and stability properties of biodiesel produced at specific production plants may be different. Any special requirements of the user, such as long-term storage, should be discussed with the supplier.

X2.4 Fuel Additives

X2.4.1 Available fuel additives appear to improve the long term storage of biodiesel. Most additives should be added as close to the production site as possible to obtain maximum benefits.

X2.4.2 Biocides or biostats destroy or inhibit the growth of fungi and bacteria which can grow at fuel-water interfaces to give high particulate concentrations in the fuel. Available biocides are soluble in the fuel phase or the water phase, or both. Refer to Guide D 6469 for a more complete discussion.

X2.5 Tests for Fuel Quality

X2.5.1 Test methods for estimating the storage stability of biodiesel (B100) are being developed. Modifications of Test Method D 2274 to use glass fiber filters, varying times and temperatures, and the measurement of pre-test and post-test acid number and viscosity appear promising. However, correlation of this test with actual storage stability is unknown, and may depend upon field conditions and fuel composition.

X2.5.2 Performance criteria for accelerated stability tests that ensure satisfactory long-term storage of biodiesel (B100) have not been established.

X2.6 Fuel Monitoring

X2.6.1 A plan for monitoring the quality of bulk fuel during prolonged storage is an integral part of a successful monitoring program. A plan to replace aged fuel with fresh product at established intervals is also desirable.

X2.6.2 Stored fuel should be periodically sampled and its quality assessed. Practice D 4057 provides guidance for sampling. Fuel contaminants and degradation products may settle to the bottom of a quiescent tank although detrimental changes to biodiesel can occur (rising acid value) without causing sediment formation. A *Bottom* or *Clearance* sample, as defined in Practice D 4057, should be included in the evaluation along with an *All Level* sample.

X2.6.3 The quantity of insoluble fuel contaminants present in biodiesel can be determined using Test Method D 6217 with glass fiber filters and abundant washing although no precision or bias testing has been performed with biodiesel using Test Method D 6217.

X2.6.4 The acid value of biodiesel appears to exceed its specified maximum before other deleterious fuel property changes occur. A conscientious program of measuring the acid value of biodiesel may be sufficient for monitoring biodiesel stability.



X2.7 Fuel Storage Conditions

X2.7.1 Contamination levels in fuel can be reduced by storage in tanks kept free of water, and tankage should have provisions for water draining on a scheduled basis. Water promotes corrosion, and microbiological growth may occur at a fuel-water interface. Refer to Guide D 6469 for a more complete discussion. Underground or isothermal storage is preferred to avoid temperature extremes; above-ground storage tanks should be sheltered or painted with reflective paint. High storage temperatures accelerate fuel degradation. Fixed roof tanks should be kept full to limit oxygen supply and tank

breathing. The use of airtight sealed containers, such as drums or totes, can enhance the storage life of biodiesel.

X2.7.2 Copper and copper-containing alloys should be avoided with biodiesel due to increased sediment and deposit formation. Contact with lead, tin, and zinc can also cause increased sediment levels that can rapidly plug filters and should be avoided.

X2.7.3 Appendix X3 of Specification D 2880 discusses fuel contaminants as a general topic. The discussion in Specification D 2880 pertains to gas turbine combustion which may or may not be applicable to diesel engine combustion.

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