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Designation: <del>D7467 – 10</del> <u>D7467 – 13</u>

# Standard Specification for Diesel Fuel Oil, Biodiesel Blend (B6 to B20)<sup>1</sup>

This standard is issued under the fixed designation D7467; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope\*

1.1 This specification covers fuel blend grades of 6 to 20 volume percent (%) biodiesel with the remainder being a light middle or middle distillate diesel fuel, collectively designated as B6 to B20. These grades are suitable for various types of diesel engines.

1.1.1 The biodiesel component of the blend shall conform to the requirements of Specification D6751. The remainder of the fuel shall be a light middle or middle distillate grade diesel fuel conforming to Specification D975 grades No. 1-D and No. 2-D of any sulfur level specified with the following exceptions. The light middle or middle distillate grade diesel fuel whose sulfur level, aromatic level, cetane, or lubricity falls outside of Specification D975 may be blended with biodiesel meeting Specification D6751, provided the finished mixtures meets this specification.

1.1.2 The fuel sulfur grades are described as follows:

1.1.2.1 Grade B6 to B20 S15-A fuel with a maximum of 15 ppm sulfur.

1.1.2.2 Grade B6 to B20 S500-A fuel with a maximum of 500 ppm sulfur.

1.1.2.3 Grade B6 to B20 S5000-A fuel with a maximum of 5000 ppm sulfur.

1.2 This specification prescribes the required properties of B6 to B20 biodiesel blends at the time and place of delivery. The specification requirements may be applied at other points in the production and distribution system when provided by agreement between the purchaser and the supplier.

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

NOTE 1—The generation and dissipation of static electricity can create problems in the handling of distillate diesel fuel oils. For more information on this subject, see Guide D4865.

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

### 2. Referenced Documents

2.1 ASTM Standards:<sup>2</sup>

D56 Test Method for Flash Point by Tag Closed Cup Tester

D86 Test Method for Distillation of Petroleum Products at Atmospheric Pressure

D93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester

D129 Test Method for Sulfur in Petroleum Products (General High Pressure Decomposition Device Method)

D130 Test Method for Corrosiveness to Copper from Petroleum Products by Copper Strip Test

D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)

D482 Test Method for Ash from Petroleum Products

D524 Test Method for Ramsbottom Carbon Residue of Petroleum Products

D613 Test Method for Cetane Number of Diesel Fuel Oil

D664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration

D975 Specification for Diesel Fuel Oils

D976 Test Method for Calculated Cetane Index of Distillate Fuels

D1266 Test Method for Sulfur in Petroleum Products (Lamp Method)

D1319 Test Method for Hydrocarbon Types in Liquid Petroleum Products by Fluorescent Indicator Adsorption

D1552 Test Method for Sulfur in Petroleum Products (High-Temperature Method)

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

<sup>&</sup>lt;sup>1</sup> This specification is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.E0 on Burner, Diesel, Non-Aviation Gas Turbine, and Marine Fuels.

<sup>&</sup>lt;sup>2</sup> 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.

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D2500 Test Method for Cloud Point of Petroleum Products D2622 Test Method for Sulfur in Petroleum Products by Wavelength Dispersive X-ray Fluorescence Spectrometry D2709 Test Method for Water and Sediment in Middle Distillate Fuels by Centrifuge D2880 Specification for Gas Turbine Fuel Oils D3117 Test Method for Wax Appearance Point of Distillate Fuels (Withdrawn 2010)<sup>3</sup> D3120 Test Method for Trace Quantities of Sulfur in Light Liquid Petroleum Hydrocarbons by Oxidative Microcoulometry D3828 Test Methods for Flash Point by Small Scale Closed Cup Tester D4057 Practice for Manual Sampling of Petroleum and Petroleum Products D4294 Test Method for Sulfur in Petroleum and Petroleum Products by Energy Dispersive X-ray Fluorescence Spectrometry D4539 Test Method for Filterability of Diesel Fuels by Low-Temperature Flow Test (LTFT) D4737 Test Method for Calculated Cetane Index by Four Variable Equation D4865 Guide for Generation and Dissipation of Static Electricity in Petroleum Fuel Systems D5453 Test Method for Determination of Total Sulfur in Light Hydrocarbons, Spark Ignition Engine Fuel, Diesel Engine Fuel, and Engine Oil by Ultraviolet Fluorescence D5771 Test Method for Cloud Point of Petroleum Products (Optical Detection Stepped Cooling Method) D5772 Test Method for Cloud Point of Petroleum Products (Linear Cooling Rate Method) D5773 Test Method for Cloud Point of Petroleum Products (Constant Cooling Rate Method) D6079 Test Method for Evaluating Lubricity of Diesel Fuels by the High-Frequency Reciprocating Rig (HFRR) D6217 Test Method for Particulate Contamination in Middle Distillate Fuels by Laboratory Filtration D6371 Test Method for Cold Filter Plugging Point of Diesel and Heating Fuels D6468 Test Method for High Temperature Stability of Middle Distillate Fuels D6469 Guide for Microbial Contamination in Fuels and Fuel Systems D6751 Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels D6890 Test Method for Determination of Ignition Delay and Derived Cetane Number (DCN) of Diesel Fuel Oils by Combustion in a Constant Volume Chamber D7220 Test Method for Sulfur in Automotive, Heating, and Jet Fuels by Monochromatic Energy Dispersive X-ray Fluorescence Spectrometry

D7371 Test Method for Determination of Biodiesel (Fatty Acid Methyl Esters) Content in Diesel Fuel Oil Using Mid Infrared Spectroscopy (FTIR-ATR-PLS Method)

D7397 Test Method for Cloud Point of Petroleum Products (Miniaturized Optical Method)

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

2.2 Other Standards:

26 CFR Part 48 Manufacturers and Retailers Excise Taxes<sup>4</sup>

40 CFR Part 80 Regulation of Fuels and Fuel Additives<sup>4</sup> D7467-13

EN 14078 Liquid petroleum products - Determination of fatty acid methyl ester (FAME) content in middle distillates - Infrared spectrometry method<sup>5</sup>

EN 14112 Fat and oil derivatives - Fatty acid methyl esters (FAME) - Determination of oxidation stability (Accelerated oxidation test)<sup>5</sup>

EN 15751 Automotive fuels - Fatty acid methyl ester (FAME) fuel and blends with diesel fuel - Determination of oxidation stability by accelerated oxidation method<sup>5</sup>

# 3. Terminology

3.1

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

3.3 *B6 to B20, n*—fuel blend consisting of 6 to 20 volume percent biodiesel conforming to the requirements of Specification D6751 with the remainder being a light middle or middle distillate grade diesel fuel and meeting the requirements of this specification.

3.3.1 Discussion—

The abbreviation BXX represents a specific blend concentration in the range B6 to B20, where XX is the percent volume of biodiesel in the fuel blend.

<sup>&</sup>lt;sup>3</sup> The last approved version of this historical standard is referenced on www.astm.org.

<sup>&</sup>lt;sup>4</sup> Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401.

<sup>&</sup>lt;sup>5</sup> Available from the National CEN members listed on the CEN website (www.cenorm.be.) or from the CEN/TC 19 Secretariat (astm@nen.nl).

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TABLE 1 Detailed	Requirements for B6 to B20 Biodiesel Blends
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Property	Test Method	Grade		
		B6 to B20 S15	B6 to B20 S500 <sup>A</sup>	B6 to B20 S5000 <sup>B</sup>
Acid Number, mg KOH/g, max	D664	0.3	0.3	0.3
Viscosity, mm <sup>2</sup> /s at 40°C	D445	1.9-4.1 <sup><i>C</i></sup>	1.9-4.1 <sup><i>C</i></sup>	1.9- 4.1 <sup><i>C</i></sup>
Flash Point, °C, min	D93	52 <sup>D</sup>	52 <sup>D</sup>	52 <sup>D</sup>
Cloud Point, °C, max or LTFT/ CFPP, °C, max	D2500, D4539, D6371	E	E	E
Sulfur Content, (µg/g)	D5453	15		
mass %, max	D2622		0.05	
mass %, max	D129			0.50
Distillation Temperature, °C,	D86	343	343	343
90% vol recovered, max				
Ramsbottom Carbon Residue	D524	0.35	0.35	0.35
on 10% bottoms, mass %, max				
Cetane Number, min	D613 <sup>F</sup>	40 <sup>G</sup>	40 <sup>G</sup>	40 <sup>G</sup>
One of the following must be	D976-80 <sup>H</sup>	40	40	40
met:				
<ol><li>Cetane index, min.</li></ol>				
(2) Aromaticity, %vol, max	D1319-03 <sup>H</sup>	35	35	
Ash Content, mass%, max	D482	0.01	0.01	0.01
Water and Sediment, volume%, max	D2709	0.05	0.05	0.05
Copper Corrosion, 3 h at 50°C,	D130	No. 3	No. 3	No. 3
max	D7074	0 00	0 00	0 00
Biodiesel Content, % (V/V)	D7371	6 20.	6 20.	6 20.
Oxidation Stability, hours, min	EN 15751	6	6	6
Lubricity, HFRR at 60°C, mi- cron (µm), max	D6079	520'	520'	520'

<sup>4</sup> Under United States of America regulations, if Grades B6-20 S500 are sold for tax exempt purposes then, at, or beyond terminal storage tanks, they are required by 26 CFR Part 48 to contain the dye Solvent Red 164 at a concentration spectrally equivalent to 3.9 lb per thousand barrels of the solid dye standard Solvent Red 164, or the tax must be collected.

<sup>B</sup> Under United States of America regulations, Grades B6-20 S5000 are required by 40 CFR part 80 to contain a sufficient amount of the dye Solvent Red 164 so its presence is visually apparent. At or beyond terminal storage tanks, they are required by 26 CFR Part 48 to contain the dye Solvent Red 164 at a concentration spectrally equivalent to 3.9 lb per thousand barrels of the solid dye standard Solvent Red 26.

<sup>C</sup> If Grade No. 1-D or blends of Grade No. 1-D and Grade No. 2-D diesel fuel are used, the minimum viscosity shall be 1.3 mm<sup>2</sup>/s.

<sup>D</sup> If Grade No. 1-D or blends of Grade No. 1-D and Grade No. 2-D diesel fuel are used, or a cloud point of less than -12°C is specified, the minimum flash point shall be 38°C.

<sup>E</sup> It is unrealistic to specify low temperature properties that will ensure satisfactory operation at all ambient conditions. In general, cloud point (or wax appearance point) Low Temperature Flow Test, and Cold Filter Plugging Point Test may be useful to estimate vehicle low temperature operability limits but their use with B6 to B20 has not been validated. However, satisfactory operation below the cloud point (or wax appearance point) may be achieved depending on equipment design, operating conditions, and the use of flow-improver additives as described in X3.1.2. Appropriate low temperature operability properties should be agreed upon between the fuel supplier and purchaser for the intended use and expected ambient temperatures. Test Methods D4539 and D6371 may be especially useful to estimate vehicle low temperature operability limits when flow improvers are used but their use with B6 to B20 from a full range of biodiesel feedstock sources has not been validated. Due to fuel delivery system, engine design, and test method differences, low temperature operability tests may not provide the same degree of protection in various vehicle operating classes. Tenth percentile minimum air temperatures for U.S. locations are provided in Appendix X3 as a means of estimating expected regional temperatures. The tenth percentile minimum air temperatures may be used to estimate expected regional target temperatures for use with Test Methods D2500, D4539, and D6371. Refer to X3.1.3 for further general guidance on test application.

<sup>F</sup> Calculated cetane index approximation, Test Method D4737, is not applicable to biodiesel blends.

<sup>G</sup> Low ambient temperatures, as well as engine operation at high altitudes, may require the use of fuels with higher cetane ratings. If the diesel fuel is qualified under Table 1 of Specification D975 for cetane, it is not necessary to measure the cetane number of the blend. This is because the cetane number of the individual blend components will be at least 40, so the resulting blend will also be at least 40 cetane number.

<sup>H</sup> These test methods are specified in 40 CFR Part 80.

<sup>1</sup> If the diesel fuel is qualified under Table 1 of Specification D975 for lubricity, it is not necessary to measure the lubricity of the blend. This is because the lubricity of the individual blend components will be less than 520 micron (µm) so the resulting blend will also be less than 520 (µm).

<sup>J</sup> Other sulfur limits can apply in selected areas in the United States and in other countries.

3.4 *S*(*numerical specification maximum*)—indicates the maximum sulfur content, in weight ppm ( $\mu$ g/g), allowed by this specification.

#### 4. Test Methods

4.1 The requirements enumerated in this specification shall be determined in accordance with the following methods:

4.1.1 Acid Number—Test Method D664.

4.1.2 *Flash Point*—Test Method D93, except where other methods are prescribed by law. Test Method D3828 may be used as an alternate with the same limits. Test Method D56 may be used as an alternate with the same limits, provided the flash point is below 93°C. This test method will give slightly lower values. In cases of dispute, Test Method D93 shall be used as the referee method.

4.1.3 *Cloud Point*—Test Method D2500. For all B6 to B20 grades in Table 1, Test Method D7397 and the automatic Test Methods D5771, D5772, or D5773 may be used as alternates with the same limits. Test Method D3117 may also be used since it is closely related to Test Method D2500. In case of dispute, Test Method D2500 shall be the referee test method.

4.1.4 Cold Filter Plugging Point (CFPP)—Test Method D6371.



### TABLE 2 Sulfur Test Methods

Sulfur Test Method	Range	Grades	Units Used to Report Results <sup>A</sup>
D129	>0.1 mass %	S5000	mass %
(referee)			
D1266	0.0005 to 0.4 mass %	S500	mass %
	5 to 4000 mg/kg (wt ppm)		
D1552	>0.06 mass %	S5000	mass %
D2622	0.0003 to 5.3 mass %	all grades	mass %
(referee for S500 grades)	3 to 53 000 mg/kg (wt ppm)	0	
D3120	3.0 to 100 mg/kg (wt ppm)	S15, S500	ppm (µg/g)
		(S500 grades must be diluted before	
		testing)	
D4294	0.0150 to 5.00 mass %	S5000	mass %
	150 to 50 000 mg/kg (wt ppm)		
D5453	0.0001 to 0.8 mass %	all grades	ppm (µg/g)
(referee for S15 grades)	1.0 to 8000 mg/kg (wt ppm)	5	
D7220	3.0 to 942 mg/kg (mass ppm)	S15, S500	mass ppm (µg/g)

<sup>A</sup> Results reported in mg/kg and in ppm (µg/g) are numerically the same. The units used in Table 1 for the sulfur requirements are the units in which results for the referee test are reported.

4.1.5 Low Temperature Flow Test (LTFT)—Test Method D4539.

4.1.6 Water and Sediment—Test Method D2709.

4.1.7 Carbon Residue—Test Method D524.

4.1.8 Ash—Test Method D482.

4.1.9 Distillation—Test Method D86.

4.1.10 Viscosity—Test Method D445.

4.1.11 *Sulfur*—Table 2 shows the referee test methods and alternate test methods for sulfur, the range over which each test method applies and the corresponding fuel grades.

4.1.12 *Aromaticity*—Test Method D1319. This test method provides an indication of the aromatic content of fuels. For fuels with a maximum final boiling point of 315°C, this test method is a measurement of the aromatic content of the fuel. Grade S5000 does not have an aromatics content.

4.1.13 Cetane Index—Test Method D976.

4.1.14 Lubricity—Test Method D6079.

4.1.15 Copper Corrosion—Test Method D130, 3 h test at 50°C minimum.

4.1.16 *Cetane Number*—Test Method D613. Test Method D6890 may also be used. In cases of dispute, Test Method D613 shall be the referee test method.

4.1.17 Oxidation Stability—Test Method EN 15751. Test Method EN 14112 may also be used but has been shown to provide falsely low readings in some cases. See X1.16.2 for further information. In case of dispute, Test Method EN 15751 shall be the referee test method.

4.1.18 *Biodiesel Content*—Test Method D7371. Test Method EN 14078 may also be used. In cases of dispute, Test Method D7371 shall be the referee test method. See Practice E29 for guidance on significant digits.

### 5. Workmanship

5.1 The biodiesel blend (B6 to B20) shall be visually free of undissolved water, sediment, and suspended matter.

5.2 The biodiesel blend (B6 to B20) shall also be free of any adulterant or contaminant that may render the fuel unacceptable for its commonly used applications.

### 6. Requirements

6.1 The biodiesel blend (B6 to B20) specified shall conform to the detailed requirements shown in Table 1.

### 7. Keywords

7.1 biodiesel; biodiesel blend; diesel; fuel oil; petroleum and petroleum products

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### **APPENDIXES**

### (Nonmandatory Information)

### X1. SIGNIFICANCE OF ASTM SPECIFICATION FOR B6 to B20 BIODIESEL BLENDS

### **X1.1 Introduction**

X1.1.1 The properties of commercial B6 to B20 blends depend on the refining practices employed and the nature of the distillate fuel oils and biodiesel from which they are produced. Distillate fuel oils, for example, may be produced within the boiling range of 150 and 400°C having many possible combinations of various properties, such as volatility, ignition quality, viscosity, and other characteristics. Biodiesel, for example, can be produced from a variety of animal fats or vegetable oils that produce similar volatility characteristics and combustion emissions with varying cold flow properties.

### X1.2 Cetane Number

X1.2.1 Cetane number is a measure of the ignition quality of the fuel and influences combustion roughness. The cetane number requirements depend on engine design, size, nature of speed and load variations, and on starting and atmospheric conditions. Increase in cetane number over values actually required does not materially improve engine performance. Accordingly, the cetane number specified should be as low as possible to ensure maximum fuel availability.

### X1.3 Distillation

X1.3.1 The fuel volatility requirements depend on engine design, size, nature of speed and load variations, and starting and atmospheric conditions. For engines in services involving rapidly fluctuating loads and speeds, as in bus and truck operation, the more volatile fuels may provide best performance, particularly with respect to smoke and odor. The biodiesel portion of the B6 to B20 may also provide smoke and odor improvements. However, best fuel economy is generally obtained from the heavier types of fuels because of their higher heat content.

### X1.4 Viscosity

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X1.4.1 For some engines it is advantageous to specify a minimum viscosity because of power loss due to injection pump and injector leakage. Maximum viscosity, on the other hand, is limited by considerations involved in engine design and size, and the characteristics of the injection system.

### X1.5 Carbon Residue

X1.5.1 Carbon residue gives a measure of the carbon depositing tendencies of a fuel oil when heated in a bulb under prescribed conditions. While not directly correlating with engine deposits, this property is considered an approximation.

### X1.6 Sulfur

X1.6.1 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 affect emission control systems performance. To ensure maximum availability of fuels, the permissible sulfur content should be specified as high as is practicable, consistent with maintenance considerations and legal limits.

### X1.7 Flash Point

X1.7.1 The flash point as specified is not directly related to engine performance. It is, however, of importance in connection with legal requirements and safety precautions involved in fuel handling and storage, and it is normally specified to meet insurance and fire regulations.

# 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 wax crystals appears in the oil under prescribed test conditions that generally relates to the temperature at which wax crystals begin to precipitate from the oil in use.

### X1.9 Ash

X1.9.1 Ash-forming materials may be present in fuel oil in three forms: (1) abrasive solids, (2) soluble metallic soaps, and (3) unremoved biodiesel catalysts. Abrasive solids and unremoved biodiesel catalysts 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 engine deposits and filter clogging.

### X1.10 Copper Strip Corrosion

X1.10.1 This test serves as a measure of possible difficulties with copper and brass or bronze parts of the fuel system.

# X1.11 Aromaticity

X1.11.1 This test is used as an indication of the aromatics content of diesel fuel. Aromatics content is specified to prevent an increase in the average aromatics content in diesel fuels. Increases in aromatics content of fuels over current levels may have a negative impact on emissions. Use of Test Method D1319-03 or cetane index, Test Method D976-80, is required in the United States of America by 40 CFR Part 80. The precision and bias of Test Method D1319-03 with biodiesel blends is not known and is currently under investigation.

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### X1.12 Cetane Index

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X1.12.1 Cetane index is specified as a limitation on the amount of high aromatic components in S15 and S500 Grades. Use of Test Method D1319-03 or cetane index, Test Method D976-80, is required in the United States of America by 40 CFR Part 80. The precision and bias of Test Method D976-80 with biodiesel blends is not known.

# X1.13 Total and Free Glycerin

X1.13.1 High levels of total or free glycerin can cause injector deposits and may adversely affect cold weather operation and filter plugging and result in a buildup of material in the bottom of storage and fueling systems. The total and free glycerin levels are controlled by Specification D6751 to 0.24% mass maximum and 0.02% mass maximum, respectively. Diesel fuel contains no total or free glycerin, so the level of total and free glycerin in a biodiesel blend is solely derived from the biodiesel contribution and is extremely low and in direct proportion to the level of biodiesel added and its total and free glycerin values. In finished blends, the ability to measure total and free glycerin is compromised by interference with naturally occurring petroleum diesel fuel components and the extremely low values. No ASTM test methods for measuring total and free glycerin in blends currently exist, so no specification for the finished B6 to B20 blend is included. If test methods become available, the level of total and free glycerin should not exceed the maximum contribution derived from biodiesel based on the blend content and the maximum level allowed in Specification D6751.

### X1.14 Calcium and Magnesium, Sodium and Potassium, and Phosphorus Content

X1.14.1 Calcium and magnesium combined and sodium and potassium combined are controlled to 5 ppm maximum in Specification D6751. Phosphorus is controlled to 10 ppm maximum in Specification D6751. The presence of high levels of these elements could adversely affect exhaust catalysts and after-treatment systems. The concentration of these materials due to biodiesel in a B6 to B20 blends should be less than 1 or 2 ppm, making accurate measurement difficult. There are also no controls for these

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materials in Specification D975 at present and no available database for the potential contribution of these materials from petroleum based diesel fuel. Based on this, a specification for finished blends for these compounds has not been established. If measured, the level of these materials should not exceed the maximum contribution derived from biodiesel based on the blend content and the maximum level allowed in Specification D6751 and the contribution of the petroleum based diesel fuel.

# X1.15 Other

# X1.15.1 *Microbial Contamination:*

X1.15.1.1 Uncontrolled microbial contamination in fuel systems can cause or contribute to a variety of problems, including increased corrosivity and decreased stability, filterability, and caloric value. Microbial processes in fuel systems can also cause or contribute to system damage.

X1.15.1.2 Because the microbes contributing to the aforementioned problems are not necessarily present in the fuel itself, no microbial quality criterion for fuels is recommended. However, it is important that personnel responsible for fuel quality understand how uncontrolled microbial contamination can affect fuel quality.

X1.15.1.3 Guide D6469 provides personnel with limited microbiological background an understanding of the symptoms, occurrences, and consequences of microbial contamination. Guide D6469 also suggests means for detecting and controlling microbial contamination in fuels and fuel systems. Good housekeeping, especially keeping fuel dry, is critical.

# X1.16 Oxidation Stability

X1.16.1 If the biodiesel is qualified under Table 1 of Specification D6751 for oxidation stability, it may not be necessary to measure the oxidation stability of the blend. Existing data<sup>6</sup> indicates the oxidation stability of B6 to B20 should be over 6 h if the oxidation stability of the biodiesel is 3 h or higher at the time of blending.

X1.16.2 Special precautions may be necessary to eliminate falsely low readings using EN 14112 with biodiesel blends. The petroleum portion of the blend may affect tubing between the reaction vessel and the measuring vessel and the plastic seal on the top of the reaction vessel or condense in various parts of the test setup. Some of these parts may need to be replaced frequently, and all components should be thoroughly cleaned to prevent falsely low readings. Improvements to these parts and changes in the test method have been incorporated into a revised method, EN 15751, which is the referee method. It is recommended that EN 15751 be utilized for measurement of biodiesel blend oxidation stability, because EN 14112 may be withdrawn in the future as an option for testing biodiesel and biodiesel blends.

# X1.17 Acid Number

X1.17.1 The acid number is used to determine the level of free fatty acids or processing acids that may be present in the biodiesel or diesel fuel oil when produced, or those which form upon aging. Biodiesel blends with a high acid number have been shown to increase fueling system deposits and may increase the likelihood for corrosion.

### X2. STORAGE AND THERMAL STABILITY OF B6 TO B20 BLENDS

# X2.1 Scope

X2.1.1 This appendix provides guidance for consumers of B6 to B20 who may wish to store quantities of fuels for extended periods or use the fuel in severe service or high temperature applications. Fuels containing residual components are excluded. Consistently successful long-term fuel storage or use in severe applications requires attention to fuel selection, storage conditions, handling and monitoring of properties during storage and prior to use.

<sup>&</sup>lt;sup>6</sup> McCormick, R. L., and Westbrook, S. R., "Empirical Study of the Stability of Biodiesel and Biodiesel Blends, Milestone Report," NREL/TP-540-41619, National Renewable Energy Laboratory, Golden, Colorado, May 2007. http://www.nrel.gov/docs/fy07osti/41619.pdf.



X2.1.2 Normally produced fuels have adequate stability properties to withstand normal storage and use without the formation of troublesome amounts of insoluble degradation products although data suggests some B6 to B20 blends may degrade faster than petrodiesel. Fuels that are to be stored for prolonged periods or used in severe applications should be selected to avoid formation of sediments or gums, high acid numbers, or high viscosity which can overload filters or plug injectors. Selection of these fuels should result from supplier-user discussions.

X2.1.3 These suggested practices are general in nature and should not be considered substitutes for any requirements imposed by the warranty of the distillate fuel equipment manufacturer or by federal, state, or local government regulations. Although they cannot replace a 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 B6 to B20 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 or selected for storage or severe use.

# **X2.2** Definitions

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

X2.2.2 fuel contaminants-foreign materials that make fuel less suitable or unsuitable for the intended use.

### X2.2.2.1 Discussion

Fuel contaminants include materials introduced subsequent to the manufacture of fuel and fuel degradation products.

X2.2.3 *fuel-degradation products*—those materials that are formed in fuel during extended storage or exposure to high temperatures.

X2.2.3.1 Discussion—

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Insoluble degradation products may combine with other fuel contaminants to reinforce deleterious effects. Soluble degradation products (soluble gums) are less volatile than fuel and may carbonize to form deposits due to complex interactions and oxidation of small amounts of olefinic or sulfur-, oxygen-, or nitrogen-contaminating compounds present in fuels. The formation of degradation products may be catalyzed by dissolved metals, especially copper salts. When dissolved copper is present it can be deactivated with metal deactivator additives.

X2.2.4 long-term storage—storage of fuel for longer than 6 months after it is received by the user.

X2.2.5 *severe use*—use of the fuel in applications which may result in engines operating under high load conditions that may cause the fuel to be exposed to excessive heat.

### X2.3 Fuel Selection

X2.3.1 Certain distilled refinery and biodiesel products are generally more suitable for long-term storage and severe service than others. The stability properties of B6 to B20 blends are highly dependent on the crude oil sources, severity of processing, use of additives and whether additional refinery treatment has been carried out.

X2.3.2 The composition and stability properties of B6 to B20 produced at specific refineries or blending locations may be different. Any special requirements of the user, such as long-term storage or severe service, should be discussed with the supplier.

X2.3.3 Blends of fuels from various sources may interact to give stability properties worse than expected based on the characteristics of the individual fuels.

# X2.4 Fuel Additives

X2.4.1 Available fuel additives can improve the suitability of marginal fuels for long-term storage and thermal stability, but may be unsuccessful for fuels with markedly poor stability properties. Most additives should be added at the refinery or during the early weeks of storage 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 both the fuel and water or in the water phase only.

# X2.5 Tests for Fuel Quality

X2.5.1 At the time of manufacture, the storage stability of B6 to B20 may be assessed using Test Method EN 14112. Other tests methods are under development. However, these accelerated stability tests may not correlate well with field storage stability due to varying field conditions and to fuel composition.

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

X2.5.3 Test Method D6468 provides an indication of thermal oxidative stability of middle distillate fuels when heated to temperatures near 150°C.

### **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 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 D4057 provides guidance for sampling. Fuel contaminants and degradation products will usually settle to the bottom of a quiescent tank. A "Bottom" or "Clearance" sample, as defined in Practice D4057, should be included in the evaluation along with an "All Level" sample. 4/astm-d7467-13

X2.6.3 The quantity of insoluble fuel contaminants present in fuel can be determined using Test Method D6217 although no precision or bias testing has been performed with B6 to B20 blends.

X2.6.4 Test Method D6468 can be used for investigation of operational problems that might be related to fuel thermal stability. Testing samples from the fuel tank or from bulk storage may give an indication as to the cause of filter plugging. It is more difficult to monitor the quality of fuels in vehicle tanks since operation may be on fuels from multiple sources.

X2.6.5 Some additives exhibit effects on fuels tested in accordance with Test Method D6468 that may or may not be observed in the field. Data have not been developed that correlate results from the test method for various engine types and levels of operating severity.

X2.6.6 Ongoing monitoring of the acid number is a useful means of monitoring oxidation or degradation of biodiesel blends.

### **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 D6469 for a more complete discussion. Underground 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.



X2.7.2 Copper and copper-containing alloys should be avoided. Copper can promote fuel degradation and may produce mercaptide gels. Zinc coatings can react with water or organic acids in the fuel to form gels that rapidly plug filters.

X2.7.3 Appendix X2 of Specification D2880 discusses fuel contaminants as a general topic.

### **X2.8 Fuel Use Conditions**

X2.8.1 Many diesel engines are designed so that the diesel fuel is used for heat transfer. In modern heavy-duty diesel engines, for example, only a portion of the fuel that is circulated to the fuel injectors is actually delivered to the combustion chamber. The remainder of the fuel is circulated back to the fuel tank, carrying heat with it. Thus adequate high temperature stability can be a necessary requirement in some severe applications or types of service.

X2.8.2 Inadequate high temperature stability may result in the formation of insoluble degradation products.

### X2.9 Use of Degraded Fuels

X2.9.1 Fuels that have undergone mild-to-moderate degradation can sometimes be consumed in a normal way, depending on the fuel system requirements. Filters and other cleanup equipment can require special attention and increased maintenance. Burner nozzle or injector fouling can occur more rapidly.

X2.9.2 Fuels containing very large quantities of fuel degradation products and other contaminants or with runaway microbiological growth require special attention. Consultation with experts in this area is desirable. It can be possible to drain the sediment or draw off most of the fuel above the sediment layer and use it with the precautions described in X2.9.1. However, very high soluble gum levels or corrosion products from microbiological contamination can cause severe operational problems.

# **X2.10** Thermal Stability Guidelines **Document Preview**

X2.10.1 Results from truck fleet experience suggests that Test Method D6468 can be used to qualitatively indicate whether diesel fuels have satisfactory thermal stability performance properties.<sup>7,8</sup>

X2.10.2 Performance in engines has not been sufficiently correlated with results from Test Method D6468 to provide definitive specification requirements. However, the following guidelines are suggested.

X2.10.2.1 Fuels giving a Test Method D6468 reflectance value of 70 % or more in a 90 minute test at the time of manufacture should give satisfactory performance in normal use.

X2.10.2.2 Fuels giving a Test Method D6468 reflectance value of 80 % or more in a 180 minute test at the time of manufacture should give satisfactory performance in severe use.

X2.10.3 Thermal stability as determined by Test Method D6468 is known to degrade during storage.<sup>9</sup> The guidance under X2.10 is for fuels used within six months of manufacture.

<sup>&</sup>lt;sup>7</sup> Bacha, John D., and Lesnini, David G., "Diesel Fuel Thermal Stability at 300°F," *Proceedings of the 6th International Conference on Stability and Handling of Liquid Fuels*, Vancouver, B.C., October 1997.

<sup>&</sup>lt;sup>8</sup> Schwab, Scott D., Henly, Timothy J., Moxley, Joel F., and Miller, Keith, "Thermal Stability of Diesel Fuel," *Proceedings of the 7th International Conference on Stability and Handling of Liquid Fuels*, Graz, Austria, September 2000.

<sup>&</sup>lt;sup>9</sup> Henry, C. P., "The du Pont F21 149°C (300°F) Accelerated Stability Test," *Distillate Fuel Stability and Cleanliness, ASTM STP 751*, Stavinoha, L. L., Henry, C. P., editors, ASTM International, W. Conshohocken, PA, 1981, pp. 22-33.