



Designation: D7467 – 15

## 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope\*

1.1 This specification covers fuel blend grades of 6 volume percent 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.

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

Current edition approved Jan. 1, 2015. Published February 2015. Originally approved in 2008. Last previous edition approved in 2013 as D7467 – 14. DOI: 10.1520/D7467-15.

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)

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

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

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

- 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
- D6304** Test Method for Determination of Water in Petroleum Products, Lubricating Oils, and Additives by Coulometric Karl Fischer Titration
- 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
- D7094** Test Method for Flash Point by Modified Continuously Closed Cup (MCCCFP) Tester
- 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)
- D7619** Test Method for Sizing and Counting Particles in Light and Middle Distillate Fuels, by Automatic Particle Counter
- D7668** Test Method for Determination of Derived Cetane Number (DCN) of Diesel Fuel Oils—Ignition Delay and Combustion Delay Using a Constant Volume Combustion Chamber Method
- E29** Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
- E1064** Test Method for Water in Organic Liquids by Coulometric Karl Fischer Titration
- 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>
- 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>
- ISO 4406** Hydraulic Fluid Power—Fluids—Method for Coding the Level of Contamination by Solid Particles<sup>6</sup>

### 3. Terminology

#### 3.1 Definitions:

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

3.1.2 *B6 to B20, n*—fuel blend consisting of 6 volume percent 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.

3.1.2.1 *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.

3.1.3 *S(numerical specification maximum)*—indicates the maximum sulfur content, in weight ppm (µg/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. For all grades, Test Method **D3828** and Test Method **D7094** may be used as an alternate

<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>5</sup> Available from the National CEN members listed on the CEN website ([www.cenorm.be](http://www.cenorm.be)) or from the CEN/TC 19 Secretariat ([astm@nen.nl](mailto:astm@nen.nl)).

<sup>6</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

<sup>3</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

**TABLE 1 Detailed Requirements for B6 to B20 Biodiesel Blends**

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	<b>D664</b>	0.3	0.3	0.3
Viscosity, mm <sup>2</sup> /s at 40 °C	<b>D445</b>	1.9-4.1 <sup>C</sup>	1.9-4.1 <sup>C</sup>	1.9- 4.1 <sup>C</sup>
Flash Point, °C, min	<b>D93</b>	52 <sup>D</sup>	52 <sup>D</sup>	52 <sup>D</sup>
Cloud Point, °C, max or LTFT/CFPP, °C, max	<b>D2500, D4539, D6371</b>	<i>E</i>	<i>E</i>	<i>E</i>
Sulfur Content, (µg/g)	<b>D5453</b>	15	...	...
mass percent, max	<b>D2622</b>	...	0.05	...
mass percent, max	<b>D129</b>	...	...	0.50
Distillation Temperature, °C, 90 % vol recovered, max	<b>D86</b>	343	343	343
Ramsbottom Carbon Residue on 10 % bottoms, mass %, max	<b>D524</b>	0.35	0.35	0.35
Cetane Number, min	<b>D613<sup>F</sup></b>	40 <sup>G</sup>	40 <sup>G</sup>	40 <sup>G</sup>
One of the following must be met:	<b>D976-80<sup>H</sup></b>	40	40	40
(1) Cetane index, min.				
(2) Aromaticity, volume percent, max	<b>D1319-03<sup>H</sup></b>	35	35	...
Ash Content, mass percent, max	<b>D482</b>	0.01	0.01	0.01
Water and Sediment, volume percent, max	<b>D2709</b>	0.05	0.05	0.05
Copper Corrosion, 3 h at 50 °C, max	<b>D130</b>	No. 3	No. 3	No. 3
Biodiesel Content, % (V/V)	<b>D7371</b>	6. - 20.	6. - 20.	6. - 20.
Oxidation Stability, hours, min	EN 15751	6	6	6
Lubricity, HFRR at 60 °C, micron (µm), max	<b>D6079</b>	520 <sup>I</sup>	520 <sup>I</sup>	520 <sup>I</sup>

<sup>A</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>I</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 µm so the resulting blend will also be less than 520 m.

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**.

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

4.1.6 *Water and Sediment*—Test Method **D2709**. See **Appendix X4** for additional guidance on water and sediment in biodiesel blends.

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.

**TABLE 2 Sulfur Test Methods**

Sulfur Test Method	Range	Grades	Units Used to Report Results <sup>4</sup>
<b>D129</b> (referee)	>0.1 % mass	S5000	mass percent
<b>D1266</b>	0.0005 % to 0.4 % mass	S500	mass percent
<b>D1552</b>	5 mg/kg to 4000 mg/kg (wt ppm)	S5000	mass percent
<b>D2622</b> (referee for S500 grades)	>0.06 % mass	all grades	mass percent
<b>D3120</b>	0.0003 % to 5.3 % mass	S15, S500	ppm (µg/g)
	3 mg/kg to 53 000 mg/kg (wt ppm)	(S500 grades must be diluted before testing)	
<b>D4294</b>	3.0 mg/kg to 100 mg/kg (wt ppm)	S5000	mass percent
	0.0150 % to 5.00 % mass	S5000	mass percent
<b>D5453</b> (referee for S15 grades)	150 mg/kg to 50 000 mg/kg (wt ppm)	all grades	ppm (µg/g)
<b>D7220</b>	0.0001 % to 0.8 % mass	S15, S500	mass ppm (µg/g)
	1.0 mg/kg to 8000 mg/kg (wt ppm)		
	3.0 mg/kg to 942 mg/kg (mass ppm)		

<sup>4</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.

4.1.12 *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** or Test Method **D7668** (see [Note 2](#)) may also be used. In cases of dispute, Test Method **D613** shall be the referee test method.

NOTE 2—Precision from Test Method **D7668** were obtained from results produced by laboratories using externally obtained pre-blended calibration reference material.

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

## 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 °C 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

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.

### X1.12 Cetane Index

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 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>7</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.

<sup>7</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. 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.

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*—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 six 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.

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

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>8,9</sup>

<sup>8</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.

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 min 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 min 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>10</sup> The guidance under X2.10 is for fuels used within six months of manufacture.

<sup>9</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>10</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.

## X3. TENTH PERCENTILE MINIMUM AMBIENT AIR TEMPERATURES FOR THE UNITED STATES (EXCEPT HAWAII)

### X3.1 Introduction

X3.1.1 The tenth percentile minimum ambient air temperatures shown on the following maps (Figs. X3.1-X3.12) and in Table X3.1 were derived from an analysis of historical hourly temperature readings recorded over a period of 15 to 21 years from 345 weather stations in the United States. This study was conducted by the U.S. Army Mobility Equipment Research and Development Center (USAMERDC), Coating and Chemical Laboratory, Aberdeen Proving Ground, MD 21005. The tenth percentile minimum ambient air temperature is defined as the lowest ambient air temperature which will not go lower on average more than 10 % of the time. In other words, the daily minimum ambient air temperature would on average not be expected to go below the monthly tenth percentile minimum ambient air temperature more than 3 days for a 30-day month. See Table X3.1.

X3.1.2 These data may be used to estimate low temperature operability requirements. In establishing low temperature operability requirements, consideration should be given to the following. These factors, or any combination, may make low temperature operability more or less severe than normal. As

X3.1.2.1 through X3.1.2.12 indicate, field work suggests that cloud point (or wax appearance point) is a fair indication of the low temperature operability limit of fuels without cold flow additives in most vehicles with diesel fuel that contains no biodiesel, and its relevance with B6 to B20 blends has not been validated.

X3.1.2.1 Long term weather patterns (Average winter low temperatures will be exceeded on occasion).

X3.1.2.2 Short term local weather conditions (Unusual cold periods do occur).

X3.1.2.3 Elevation (High locations are usually colder than surrounding lower areas).

X3.1.2.4 Specific engine design.

X3.1.2.5 Fuel system design (Recycle rate, filter location, filter capacity, filter porosity, and so forth.)

X3.1.2.6 Fuel viscosity at low temperatures.

X3.1.2.7 Equipment add-ons (Engine heaters, radiator covers, fuel line and fuel filter heaters and so forth.)

X3.1.2.8 Types of operation (Extensive idling, engine shutdown, or unusual operation).

X3.1.2.9 Low temperature flow improver additives in fuel.



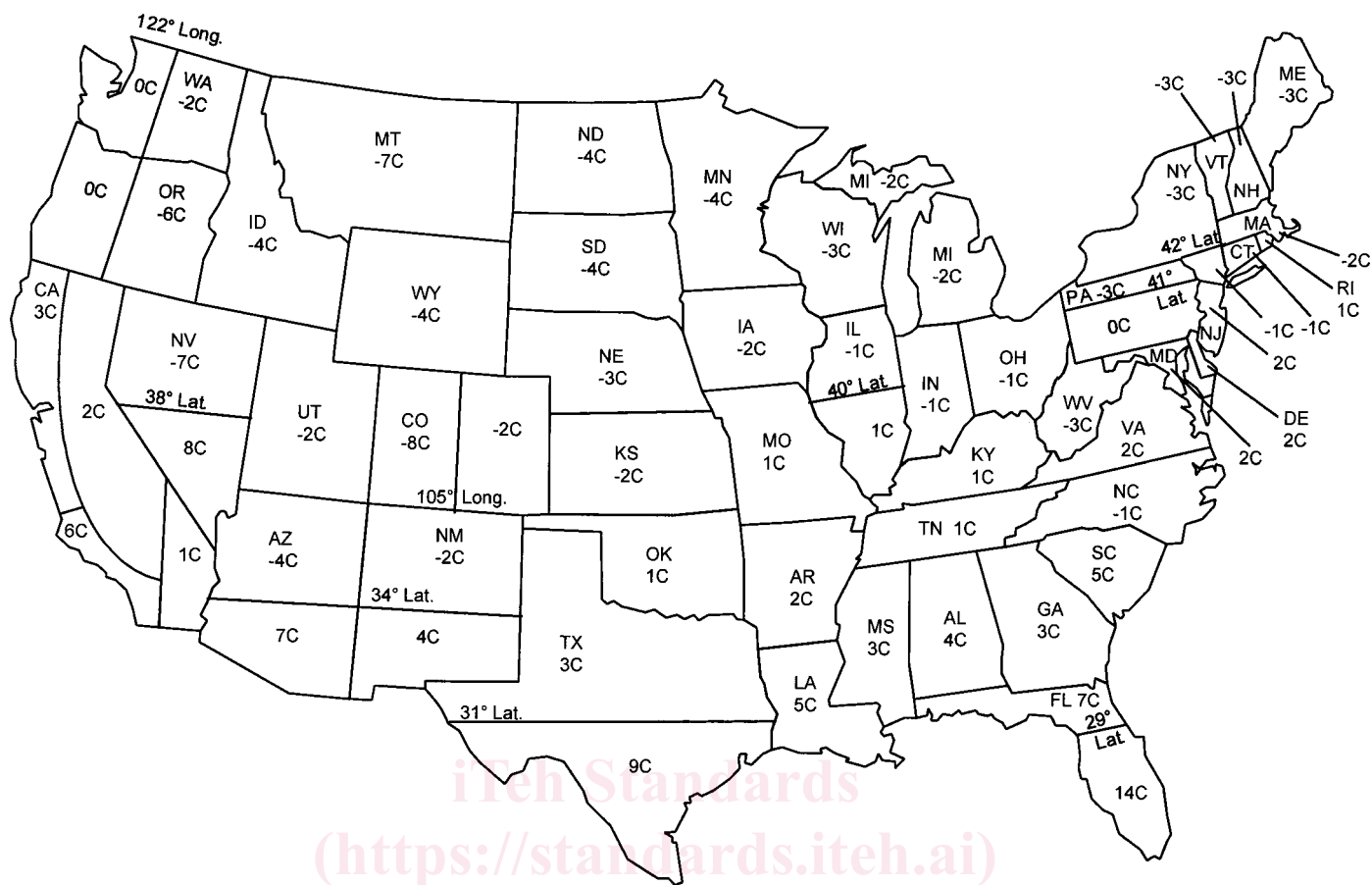


FIG. X3.1 October—10th Percentile Minimum Temperatures

X3.1.2.10 Geographic area for fuel use and movement between geographical areas.

X3.1.2.11 General housekeeping (Dirt or water, or both, in fuel or fuel supply system).

X3.1.2.12 Impact failure for engine to start or run (Critical vs. non-critical application).

X3.1.3 *Historical Background*—Three test methods have been widely used to estimate or correlate with low temperature vehicle operability with diesel fuel that contains no biodiesel. These test methods may be useful to estimate or correlate with lower temperature vehicle operability with B6 to B20, but their use with B6 to B20 has not been validated. Cloud point, Test Method **D2500**, is the oldest of the three and most conservative of the tests. The cloud point test indicates the earliest appearance of wax precipitation that might result in plugging of fuel filters or fuel lines under prescribed cooling conditions. Although not 100 % failsafe, it is the most appropriate test for applications that can not tolerate much risk. The Cold Filter Plugging Point (CFPP) test, Test Method **D6371**, was introduced in Europe in 1965. The CFPP was designed to correlate with the majority of European vehicles. Under rapid cooling conditions, 20 cc fuel is drawn through a 45 μm screen then allowed to flow back through the screen for further cooling. This process is continued every 1 °C until either the 20 cc fuel fails to be drawn through the screen in 60 s or it fails to return through the screen in 60 s. It was field tested many times in

Europe<sup>11</sup> before being widely accepted as a European specification. Field tests have also shown CFPP results more than 10 °C below the cloud point should be viewed with caution because those results did not necessarily reflect the true vehicle low temperature operability limits.<sup>12</sup> CFPP has been applied to many areas of the world where similar vehicle designs are used. The Low Temperature Flow Test (LTFT), Test Method **D4539**, was designed to correlate with the most severe and one of the most common fuel delivery systems used in North American Heavy Duty trucks. Under prescribed slow cool conditions (1 °C/h), similar to typical field conditions, several 200 cc fuel specimens in glass containers fitted with 17 μm screen assemblies are cooled. At 1 °C intervals one specimen is drawn through the screen under a 20 kPa vacuum. Approximately 90 % of the fuel must come over in 60 s or less for the result to be a pass. This process is continued at lower temperatures (1 °C increments) until the fuel fails to come over in the allotted 60 s. The lowest passing temperature is defined as the LTFT for that fuel. In 1981, a CRC program was conducted to evaluate the efficacy of cloud point, CFPP, pour point, and LTFT for protecting the diesel vehicle population in

<sup>11</sup> "Low Temperature Operability of Diesels. A Report by CEC Investigation Group IGF-3," CEC, P-171-82.

<sup>12</sup> "SFPP—A New Laboratory Test for Assessment of Low Temperature Operability of Modern Diesel Fuels," CEC/93/EF 15, May 1993, pp. 5-7.

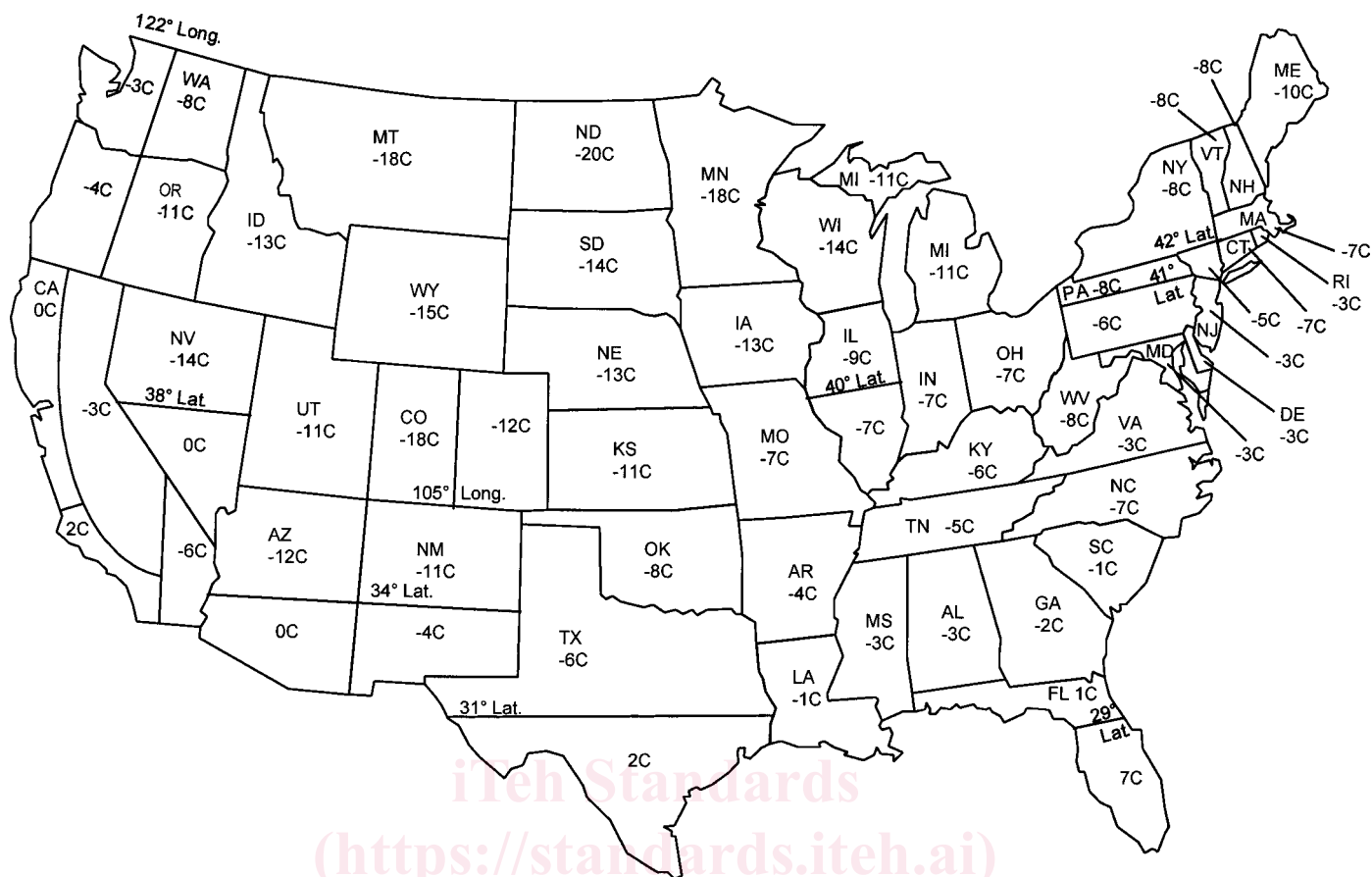


FIG. X3.2 November—10th Percentile Minimum Ambient Air Temperatures

North America and to determine what benefit flow-improvers could provide. The field test consisted of 3 non-flow improved diesel fuels, 5 flow improved diesel fuels, 4 light-duty passenger cars, and 3 heavy-duty trucks. The field trial resulted in two documents<sup>13,14</sup> that provide insight into correlating laboratory tests to North American vehicle performance in the field. The general conclusions of the study were:

- (1) In overnight cool down, 30 % of the vehicles tested had a final fuel tank temperature within 2 °C of the overnight minimum ambient temperature.
- (2) The use of flow-improved diesel fuel permits some vehicles to operate well below the fuel cloud point.
- (3) Significant differences exist in the severity of diesel vehicles in terms of low temperature operation.
- (4) No single laboratory test was found that adequately predicts the performance of all fuels in all vehicles.
- (5) CFPP was a better predictor than pour point, but both methods over-predicted, minimum operating temperatures in many vehicles. For this reason, these tests were judged inadequate predictors of low-temperature performance and dismissed from further consideration.

(6) Cloud point and LTFT showed varying degrees of predictive capability, and offered distinctively different advantages. Both predicted the performance of the base fuels well, but LTFT more accurately predicted the performance of the flow-improved fuels. On the other hand, cloud point came closest to a fail-safe predictor of vehicle performance for all vehicles.

Since the 1981 field test, non-independent studies<sup>15</sup> using newer vehicles verified the suitability of the LTFT for North American heavy-duty trucks. Users are advised to review these and any more recent publications when establishing low temperature operability requirements and deciding upon test methods.

**X3.1.3.1 Current Practices**—It is recognized that fuel distributors, producers, and end users in the United States use cloud point, wax appearance point, CFPP, and LTFT to estimate vehicle low temperature operability limits for diesel fuel. No independent data has been published in recent years to determine test applicability for today’s fuels and vehicles.

### X3.2 Maps

**X3.2.1** The maps in the following figures were derived from CCL Report No. 316, “A Predictive Study for Defining

<sup>13</sup> CRC Report No. 537, “The Relationship Between Vehicle Fuel Temperature and Ambient Temperature, 1981 CRC Kapuskasing Field Test,” December 1983.

<sup>14</sup> CRC Report No. 528, “1981 CRC Diesel Fuel Low-Temperature Operability Field Test,” September 1983.

<sup>15</sup> SAE 962197, SAE 982576, SAE 2000-01-2883.