



Standard Specification for Fuel Ethanol (Ed75-Ed85) for Automotive Spark-Ignition Engines¹

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1. Scope

1.1 This specification covers a fuel blend, nominally 75 to 85 volume % denatured fuel ethanol and 25 to 15 additional volume % hydrocarbons for use in ground vehicles with automotive spark-ignition engines. Appendix X1 discusses the significance of the properties specified.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 The following precautionary caveat pertains only to the test method portion, Annex A1 of this specification. *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents²

2.1 ASTM Standards:

- D 86 Test Method for Distillation of Petroleum Products³
- D 130 Test Method for Detection of Copper Corrosion from Petroleum Products by the Copper Strip Tarnish Test³
- D 381 Test Method for Existent Gum in Fuels by Jet Evaporation³
- D 512 Test Methods for Chloride Ion in Water⁴
- D 525 Test Method for Oxidation Stability of Gasoline (Induction Period Method)³
- D 1266 Test Method for Sulfur in Petroleum Products (Lamp Method)³
- D 1613 Test Method for Acidity in Volatile Solvents and Chemical Intermediates Used in Paint, Varnish, Lacquer, and Related Products⁵
- D 1688 Test Methods for Copper in Water⁴

- D 2622 Test Method for Sulfur in Petroleum Products by X-Ray Spectrometry Method⁶
- D 2988 Test Method for Water-Soluble Halide Ion in Halogenated Organic Solvents and Their Admixtures⁷
- D 3120 Test Method for Trace Quantities of Sulfur in Light Liquid Petroleum Hydrocarbons by Oxidative Microcoulometry⁶
- D 3231 Test Method for Phosphorus in Gasoline⁶
- D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products⁶
- D 4806 Specification for Denatured Fuel Ethanol for Blending with Gasolines for Use as Automotive Spark-Ignition Engine Fuel⁸
- D 4814 Specification for Automotive Spark-Ignition Engine Fuel⁸
- D 4815 Test Method for Determination of MTBE, ETBE, TAME, DIPE, tertiary-Amyl Alcohol and C₁ to C₄ Alcohols in Gasoline by Gas Chromatography⁸
- D 4929 Test Methods for Determination of Organic Chloride Content in Crude Oil⁸
- D 4953 Test Method for Vapor Pressure of Gasoline and Gasoline-Oxygenate Blends (Dry Method)⁸
- D 5059 Test Method for Lead in Gasoline by X-ray Spectroscopy⁸
- D 5190 Test Method for Vapor Pressure of Petroleum Products (Automatic Method)⁸
- D 5191 Test Method for Vapor Pressure of Petroleum Products (Mini Method)⁸
- D 5453 Test Method for the Determination of Total Sulfur in Light Hydrocarbons, Motor Fuels and Oils by Ultraviolet Fluorescence⁸
- D 5501 Test Method for the Determination of Ethanol Content of Denatured Fuel Ethanol by Gas Chromatography⁸
- D 6423 Test method for Determination of pH_e of Ethanol, Denatured Fuel Ethanol, and Fuel Ethanol (Ed75-Ed85)⁹
- E 203 Test Method for Water Using Karl Fischer Titration⁷
- E 1064 Test Method for Water in Organic Liquids by

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² Reference to the following documents is to be the latest issue unless otherwise specified.

³ *Annual Book of ASTM Standards*, Vol 05.01.

⁴ *Annual Book of ASTM Standards*, Vol 11.01.

⁵ *Annual Book of ASTM Standards*, Vol 06.04.

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

⁷ *Annual Book of ASTM Standards*, Vol 15.05.

⁸ *Annual Book of ASTM Standards*, Vol 05.03.

⁹ *Annual Book of ASTM Standards*, Vol 05.04.

Coulometric Karl Fischer Titration⁷

3. Terminology

3.1 Definitions:

3.1.1 *ethanol, n*—ethyl alcohol, the chemical compound C₂H₅OH.

3.1.2 *methanol, n*—methyl alcohol, the chemical compound CH₃OH.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *aliphatic ether*—an oxygen-containing, ashless, organic compound in which the oxygen atom is interposed between two carbon atoms (organic groups), has the general formula C_nH_{2n+2}O with *n* being 5 to 8, and in which the carbon atoms are connected in open chains and not closed rings.

3.2.1.1 *Discussion*—Aliphatic compounds can be straight or branched chains and saturated or unsaturated. The term aliphatic ether, as used in this specification, refers only to the saturated compounds.

3.2.2 *denaturants*—natural gasoline, gasoline components, unleaded gasoline, or toxic or noxious materials added to fuel ethanol to make it unsuitable for beverage use but not unsuitable for automotive use.

3.2.3 *denatured fuel ethanol*—fuel ethanol made unfit for beverage use by the addition of denaturants.

3.2.4 *fuel ethanol*—ethanol with impurities common to its production (including water but excluding denaturants).

3.2.5 *fuel ethanol (Ed75-Ed85)*—blend of ethanol and hydrocarbon of which the ethanol portion is nominally 70 to 85 volume % denatured fuel ethanol.

3.2.6 *higher alcohols*—aliphatic alcohols of general formula C_nH_{2n+1}OH with *N* being 3 to 8.

3.2.7 *hydrocarbon*—those components in an ethanol-hydrocarbon blend containing only hydrogen and carbon.

3.2.8 *pH_e*—a measure of the acid strength of alcohol fuels.

4. Fuel Ethanol (Ed75-Ed85) Performance Requirements

4.1 Fuel ethanol (Ed75-Ed85) shall conform to the requirements of Table 1.

NOTE 1—Most of the requirements cited are based on the best technical information currently available. Requirements for sulfur, phosphorus, and lead are based on the use of gasoline defined in Specification D 4814 and the understanding that control of these elements will affect catalyst lifetime. The lead maximum is limited for Class 1 and Class 2 fuels to the lower limit of the test method. As greater experience is gained from field use of Ed75-Ed85 vehicles and further vehicle hardware developments for

TABLE 1 Requirements for Fuel Ethanol (Ed75-Ed85)

| Properties | Class 1 ^A | Class 2 | Class 3 |
|--|---|-----------------|------------------|
| Ethanol + higher alcohols, min, volume % | 79 | 74 | 70 |
| Hydrocarbon/aliphatic ether, volume % | 17–21 | 17–26 | 17–30 |
| Vapor pressure, kPa (psi) | 38–59 (5.5–8.5) | 48–65 (7.0–9.5) | 66–83 (9.5–12.0) |
| Lead, max, mg/L | 2.6 | 2.6 | 3.9 |
| Phosphorus, max, mg/L | 0.2 | 0.3 | 0.4 |
| Sulfur, max, mg/kg | 210 | 260 | 300 |
| All Classes | | | |
| Methanol, volume %, max | 0.5 | | |
| Higher alcohols (C ₃ –C ₈), max, volume % | 2 | | |
| Acidity, (as acetic acid CH ₃ COOH), mass % (mg/L), max | 0.005 (40) | | |
| Solvent-washed gum content, max, mg/100 mL | 5 | | |
| pH _e | 6.5 to 9.0 | | |
| Unwashed gum content, max, mg/100 mL | 20 | | |
| Total chlorine as chlorides, max, mg/kg | 2 | | |
| Inorganic chloride, max, mg/kg | 1 | | |
| Copper, max, mg/L | 0.07 | | |
| Water, max, mass % | 1.0 | | |
| Appearance | This product shall be visibly free of suspended or precipitated contaminants (clear and bright). This shall be determined at ambient temperature or 21°C (70°F), whichever is higher. | | |

^ASee 4.1.1 for volatility class criteria.

the use of ethanol content fuels occurs, it is expected that many of these requirements will change.

4.1.1 Vapor pressure is varied for seasonal and climatic changes by providing three vapor pressure classes for fuel ethanol (Ed75-Ed85). The seasonal and geographical distribution for three vapor pressure classes is shown in Table 2. Class 1 encompasses geographical areas with 6-h tenth percentile minimum ambient temperature of greater than 5°C (41°F). Class 2 encompasses geographical areas with 6-h tenth percentile minimum ambient temperature of greater than –5°C (23°F) but less than +5°C (41°F). Class 3 encompasses geographical areas with 6-h tenth percentile minimum ambient temperature less than or equal to –5°C (23°F).

TABLE 2 Seasonal and Geographical Volatility Specifications for Fuel Ethanol (Ed75-Ed85)

NOTE 1—This schedule, subject to agreement between the purchaser and the seller, denotes the vapor pressure class of the fuel at the time and place of bulk delivery to fuel-dispensing facilities for the end user. Shipments should anticipate this schedule.

| State | Jan | Feb | March | Apr | May | June | July | Aug | Sep | Oct | Nov | Dec |
|-------------------------|-----|-----|-------|-----|-----|------|------|-----|-----|-----|-----|-----|
| Alabama | 2 | 2 | 2 | 2 | 2/1 | 1 | 1 | 1 | 1 | 1/2 | 2 | 2 |
| Alaska | | | | | | | | | | | | |
| Southern Region | 3 | 3 | 3 | 3 | 3/2 | 2/1 | 1 | 1/2 | 2/3 | 3 | 3 | 3 |
| South Mainland | 3 | 3 | 3 | 3 | 3/2 | 2/1 | 1/2 | 2 | 2/3 | 3 | 3 | 3 |
| Arizona | | | | | | | | | | | | |
| N of 34° Latitude | 3 | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1 | 1/2 | 2/3 | 3 | 3 |
| S of 34° Latitude | 2 | 2 | 2 | 2/1 | 1 | 1 | 1 | 1 | 1 | 1/2 | 2 | 2 |
| Arkansas | 3 | 3 | 3/2 | 2/1 | 1 | 1 | 1 | 1 | 1/2 | 2 | 2/3 | 3 |
| California ^A | | | | | | | | | | | | |
| North Coast | 2 | 2 | 2 | 2 | 2 | 2/1 | 1 | 1 | 1 | 1/2 | 2 | 2 |

TABLE 2 *Continued*

| State | Jan | Feb | March | Apr | May | June | July | Aug | Sep | Oct | Nov | Dec |
|----------------------|-----|-----|-------|-----|-----|------|------|-----|-----|-----|-----|-----|
| South Coast | 3/2 | 2 | 2 | 2 | 2/1 | 1 | 1 | 1 | 1 | 1/2 | 2/3 | 3 |
| Southeast | 3 | 3/2 | 2 | 2 | 2/1 | 1 | 1 | 1 | 1/2 | 2 | 2/3 | 3 |
| Interior | 2 | 2 | 2 | 2 | 2 | 2/1 | 1 | 1 | 1 | 1/2 | 2 | 2 |
| Colorado | | | | | | | | | | | | |
| E of 105° Longitude | 3 | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1 | 1/2 | 2/3 | 3 | 3 |
| W of 105° Longitude | 3 | 3 | 3 | 3 | 3/2 | 2 | 2/1 | 1/2 | 2/3 | 3 | 3 | 3 |
| Connecticut | 3 | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1 | 1/2 | 2 | 2/3 | 3 |
| Delaware | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1 | 1 | 1/2 | 2 | 2/3 | 3 |
| District of Columbia | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1 | 1 | 1/2 | 2 | 2/3 | 3 |
| Florida | | | | | | | | | | | | |
| N of 29° Latitude | 2 | 2 | 2 | 2/1 | 1 | 1 | 1 | 1 | 1 | 1/2 | 2 | 2 |
| S of 29° Latitude | 2 | 2/1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1/2 | 2 |
| Georgia | 3 | 3/2 | 2 | 2/1 | 1 | 1 | 1 | 1 | 1 | 1/2 | 2 | 2/3 |
| Hawaii | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 |
| Idaho | 3 | 3 | 3 | 3/2 | 2 | 2 | 2/1 | 1/2 | 2 | 2/3 | 3 | 3 |
| Illinois | | | | | | | | | | | | |
| N of 40° Latitude | 3 | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1 | 1/2 | 2/3 | 3 | 3 |
| S of 40° Latitude | 3 | 3 | 3 | 3/2 | 2/1 | 1 | 1 | 1 | 1/2 | 2/3 | 3 | 3 |
| Indiana | 3 | 3 | 3 | 3/2 | 2/1 | 1 | 1 | 1 | 1/2 | 2/3 | 3 | 3 |
| Iowa | 3 | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1 | 1/2 | 2/3 | 3 | 3 |
| Kansas | 3 | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1 | 1/2 | 2/3 | 3 | 3 |
| Kentucky | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1 | 1 | 1/2 | 2 | 2/3 | 3 |
| Louisiana | 2 | 2 | 2 | 2/1 | 1 | 1 | 1 | 1 | 1 | 1/2 | 2 | 2 |
| Maine | 3 | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1/2 | 2 | 2/3 | 3 | 3 |
| Maryland | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1 | 1 | 1/2 | 2 | 2/3 | 3 |
| Massachusetts | 3 | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1 | 1/2 | 2 | 2/3 | 3 |
| Michigan | | | | | | | | | | | | |
| Lower Michigan | 3 | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1/2 | 2 | 2/3 | 3 | 3 |
| Upper Michigan | 3 | 3 | 3 | 3 | 3/2 | 2/1 | 1 | 1/2 | 2 | 2/3 | 3 | 3 |
| Minnesota | 3 | 3 | 3 | 3 | 3/2 | 2/1 | 1 | 1/2 | 2 | 2/3 | 3 | 3 |
| Mississippi | 2 | 2 | 2 | 2/1 | 1 | 1 | 1 | 1 | 1 | 1/2 | 2 | 2 |
| Missouri | 3 | 3 | 3 | 3/2 | 2/1 | 1 | 1 | 1 | 1/2 | 2/3 | 3 | 3 |
| Montana | 3 | 3 | 3 | 3 | 3/2 | 2 | 2/1 | 1/2 | 2/3 | 3 | 3 | 3 |
| Nebraska | 3 | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1/2 | 2 | 2/3 | 3 | 3 |
| Nevada | | | | | | | | | | | | |
| N of 38° Latitude | 3 | 3 | 3 | 3/2 | 2 | 2 | 2/1 | 1/2 | 2 | 2/3 | 3 | 3 |
| S of 38° Latitude | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1 | 1 | 1/2 | 2 | 2/3 | 3 |
| New Hampshire | 3 | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1/2 | 2 | 2/3 | 3 | 3 |
| New Jersey | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1 | 1 | 1/2 | 2 | 2/3 | 3 |
| New Mexico | | | | | | | | | | | | |
| N of 34° Latitude | 3 | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1 | 1/2 | 2/3 | 3 | 3 |
| S of 34° Latitude | 3 | 3 | 3/2 | 2/1 | 1 | 1 | 1 | 1 | 1/2 | 2/3 | 3 | 3 |
| New York | | | | | | | | | | | | |
| N of 42° Latitude | 3 | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1/2 | 2 | 2/3 | 3 | 3 |
| S of 42° Latitude | 3 | 3 | 3 | 3/2 | 2/1 | 1 | 1 | 1 | 1/2 | 2 | 2/3 | 3 |
| North Carolina | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1 | 1 | 1/2 | 2/3 | 3 | 3 |
| North Dakota | 3 | 3 | 3 | 3 | 3/2 | 2/1 | 1 | 1/2 | 2 | 2/3 | 3 | 3 |
| Ohio | 3 | 3 | 3 | 3/2 | 2/1 | 1 | 1 | 1 | 1/2 | 2/3 | 3 | 3 |
| Oklahoma | 3 | 3 | 3 | 3/2 | 2/1 | 1 | 1 | 1 | 1/2 | 2 | 2/3 | 3 |
| Oregon | | | | | | | | | | | | |
| E of 122° Longitude | 3 | 3 | 3 | 3/2 | 2 | 2 | 2/1 | 1/2 | 2 | 2/3 | 3 | 3 |
| W of 122° Longitude | 3 | 3/2 | 2 | 2 | 2 | 2/1 | 1 | 1 | 1/2 | 2 | 2 | 2/3 |
| Pennsylvania | | | | | | | | | | | | |
| N of 41° Latitude | 3 | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1/2 | 2 | 2/3 | 3 | 3 |
| S of 41° Latitude | 3 | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1 | 1/2 | 2 | 2/3 | 3 |
| Rhode Island | 3 | 3 | 3 | 3/2 | 2/1 | 1 | 1 | 1 | 1/2 | 2 | 2/3 | 3 |
| South Carolina | 2 | 2 | 2 | 2/1 | 1 | 1 | 1 | 1 | 1 | 1/2 | 2 | 2 |
| South Dakota | 3 | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1/2 | 2 | 2/3 | 3 | 3 |
| Tennessee | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1 | 1 | 1/2 | 2 | 2/3 | 3 |
| Texas | | | | | | | | | | | | |
| N of 31° Latitude | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1 | 1 | 1/2 | 2 | 2/3 | 3 |
| S of 31° Latitude | 2 | 2 | 2 | 2/1 | 1 | 1 | 1 | 1 | 1 | 1/2 | 2 | 2 |
| Utah | 3 | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1 | 1/2 | 2/3 | 3 | 3 |
| Vermont | 3 | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1/2 | 2 | 2/3 | 3 | 3 |
| Virginia | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1 | 1 | 1/2 | 2 | 2/3 | 3 |
| Washington | | | | | | | | | | | | |
| E of 122° Longitude | 3 | 3 | 3/2 | 2 | 2 | 2/1 | 1 | 1 | 1/2 | 2/3 | 3 | 3 |
| W of 122° Longitude | 3 | 3/2 | 2 | 2 | 2 | 2/1 | 1 | 1 | 1/2 | 2 | 2 | 2/3 |
| West Virginia | 3 | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1/2 | 2 | 2/3 | 3 | 3 |
| Wisconsin | 3 | 3 | 3 | 3/2 | 2 | 2/1 | 1 | 1/2 | 2 | 2/3 | 3 | 3 |
| Wyoming | 3 | 3 | 3 | 3 | 3/2 | 2 | 2/1 | 1/2 | 2 | 2/3 | 3 | 3 |

⁴ Details of State Climatological Division by county as indicated:

California, North Coast—Alameda, Contra Costa, Del Norte, Humboldt, Lake, Marin, Mendocino, Monterey, Napa, San Benito, San Francisco, San Mateo, Santa Clara, Santa Cruz, Solano, Sonoma, Trinity
 California, Interior—Lassen, Modoc, Plumas, Sierra, Siskiyou, Alpine, Amador, Butte, Calaveras, Colusa, El Dorado, Fresno, Glenn, Kern (except that portion lying east

of Los Angeles County Aqueduct), Kings, Madera, Mariposa, Marced, Placer, Sacramento, San Joaquin, Shasta, Stanislaus, Sutter, Tehama, Tulare, Tuolumne, Yolo, Yuba, Nevada

California, South Coast—Orange, San Diego, San Luis Obispo, Santa Barbara, Ventura, Los Angeles (except that portion north of the San Gabriel Mountain range and east of the Los Angeles County Aqueduct)

California, Southeast—Imperial, Riverside, San Bernadino, Los Angeles (that portion north of the San Gabriel Mountain range and east of the Los Angeles County Aqueduct), Mono, Inyo, Kern (that portion lying east of the Los Angeles County Aqueduct)

4.1.2 The hydrocarbons blended with the denatured fuel ethanol shall have a maximum boiling point of 225°C (437°F) by Test Method D 86, oxidation stability of 240-min minimum by Test Method D 525, and No. 1 maximum copper strip corrosion by Test Method D 130. The hydrocarbons may contain aliphatic ethers as blending components as are customarily used for automotive spark-ignition engine fuel.

4.1.3 The denaturant for the denatured fuel ethanol used in making fuel ethanol (Ed75-Ed85) shall meet the requirements of Specification D 4806 (see Section 5).

4.1.4 Use of unprotected aluminum in fuel ethanol (Ed75-Ed85) distribution and dispensing equipment will introduce insoluble aluminum compounds into the fuel, causing plugged vehicle fuel filters. Furthermore, this effect can be exaggerated even with protected aluminum by elevated fuel conductivity caused by contact with nitrile rubber dispensing hose. Therefore, unprotected aluminum and unlined nitrile rubber dispensing hose should be avoided in fuel ethanol (Ed75-Ed85) fuel distribution and dispensing systems.¹⁰

5. Sampling

5.1 Sample in accordance with Practice D 4057, except that water displacement (10.3.1.8 of Practice D 4057) shall not be used.

5.2 Where practical, fuel ethanol (Ed75-Ed85) should be sampled in glass containers. If samples must be collected in metal containers, do not use soldered containers. This is because the soldering flux residues in the containers and lead in the solder can contaminate the sample. Plastic containers should be avoided.

5.3 A minimum sample size of about 1 L (1 US qt) is recommended.

6. Test Methods

6.1 Determine the requirements enumerated in this specification in accordance with the following test methods:

NOTE 2—The appropriateness of ASTM test methods cited has not been

¹⁰ American Automobile Manufacturers Association, "Fuel Methanol Compatibility Standards and Dispensing Equipment List for M85 Fueled Vehicles," October 1994.

demonstrated for use with fuel ethanol (Ed75-Ed85). In addition, the test method outlined in Annex A1 is in the developmental stage and lacks precision and bias determinations.

6.1.1 *Ethanol*—Test Method D 5501.

6.1.2 *Hydrocarbon/Aliphatic Ether Blend Content*—Use Test Method D 4815 to determine other alcohols, methyl *tertiary*-butyl ether (MTBE), and other ethers. Determine water by the Karl Fischer test method (see 6.1.11). Subtract the concentration of alcohols and water from 100 to get the percent hydrocarbon/aliphatic ether.

6.1.3 *Vapor Pressure*—Test Method D 4953, D 5190, or D 5191.

6.1.4 *Acidity*—Test Method D 1613.

6.1.5 *pH_e*—Test Method D 6423.

6.1.6 *Gum Content, Solvent Washed and Unwashed*—Test Method D 381.

6.1.7 *Total Chlorine as Chloride*—Test Methods D 4929, Method B.

6.1.8 *Inorganic Chloride*—Test Methods D 512 or Test Method D 2988. An alternate method for inorganic chloride is found in Annex A1.

6.1.9 *Lead*—Test Method D 5059. With Test Method D 5059, prepare the calibration standards using ethanol (reagent grade) as the solvent to prevent errors caused by large differences in carbon-hydrogen ratios.

6.1.10 *Phosphorus*—Test Method D 3231.

6.1.11 *Water*—Test Method E 203 or E 1064.

6.1.12 *Copper*—Modification of Test Method D 1688 as outlined in Specification D 4806.

6.1.13 *Sulfur*—Test Method D 1266, D 2622, D 3120, or D 5453. With Test Method D 2622, prepare the calibration standards using ethanol (reagent grade) as the solvent to prevent errors caused by large differences in carbon-hydrogen ratios.

7. Keywords

7.1 acidity; alcohol; automotive spark-ignition engine fuel; chloride; copper corrosion; ether; fuel ethanol (Ed75-Ed85) for automotive spark-ignition engines; hydrocarbon; inorganic chloride; lead; MTBE; oxidation stability; oxygenates; pH_e; phosphorus; solvent washed gum content; sulfur; total chlorine; vapor pressure; volatility; water

(Mandatory Information)

A1. TEST METHOD FOR DETERMINATION OF INORGANIC CHLORIDE IN FUEL ETHANOL (Ed75-Ed85)

A1.1 Scope

A1.1.1 This test method covers a procedure to determine the inorganic chloride in fuel ethanol (Ed75-Ed85) in the range of concentrations from 0.4 to 2.0 ppm.

A1.1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

A1.2 Summary of Test Method

A1.2.1 The sample is concentrated, acidified, and treated with silver nitrate. The turbidity is visually compared with standards.

A1.3 Significance and Use

A1.3.1 Because of the corrosive nature of inorganic chloride to the fuel system of internal combustion engines, a means to measure low levels of inorganic chloride in fuel ethanol (Ed75-Ed85) is required.

A1.4 Apparatus

A1.4.1 *Distillation Apparatus*, 500-mL distillation flask, condenser, and 250-mL graduated cylinder as collector.

A1.4.2 *Nessler Tubes*, 100 mL, matched, tall form.

A1.5 Reagents and Materials

A1.5.1 *Demineralized Water*, halide- and sulfide-free.

A1.5.2 *Ethanol*, halide- and sulfide-free by distillation. (**Warning**—Flammable. Health hazard.)

A1.5.3 *Nitric Acid (HNO₃) Solution*, 1 part 15.7 M to 1 part demineralized water. (**Warning**—Corrosive. Health hazard.)

A1.5.4 *Silver Nitrate Solution*, 0.1 M. (**Warning**—Health hazard.)

A1.5.5 *Sodium Chloride*—(NaCl).

A1.6 Standards

A1.6.1 Dissolve 0.845 g of dry sodium chloride (NaCl) in halide- and sulfide-free water and dilute to 1 L in a volumetric flask. Mix thoroughly and label Solution A (0.5 mg Cl/mL).

A1.6.2 Pipet 10 mL of Solution A into a 1-L volumetric flask. Dilute to volume with halide- and sulfide-free water. Mix thoroughly and label Solution B (0.005 mg Cl/mL).

A1.6.3 In matching Nessler tubes, prepare the following standards:

| | Blank | 1 | 2 | 3 | 4 | 5 |
|------------------------|-------|-----|-----|-----|-----|-----|
| Solution B, mL (pipet) | 0.0 | 1.0 | 2.0 | 3.0 | 4.0 | 5.0 |
| Ethanol, mL (Cl-free) | 80 | 80 | 80 | 80 | 80 | 80 |

A1.6.4 For each standard follow A1.7.4-A1.7.8.

A1.6.5 The turbidity standards are affected by light and are not stable. Prepare fresh standards (from Solution B) for each group of samples.

A1.7 Procedure

A1.7.1 Clean all glassware with 1 M HNO₃, and rinse with demineralized water and halide- and sulfide-free ethanol.

A1.7.2 Measure 320 mL of sample in a graduated cylinder and put into the distillation flask, and add boiling beads. Distill the sample into a graduated cylinder until 240 mL of the distillate is obtained. Use 4 mL of the concentrated residue from the distillation flask as follows.

A1.7.3 Add a 4 mL sample to a 100 mL Nessler tube.

A1.7.4 Add 80 mL of halide- and sulfide-free ethanol to the sample in the Nessler tube.

A1.7.5 Dilute the contents of all tubes (sample and standards) to the 100-mL mark with halide- and sulfide-free water.

A1.7.6 Pipet 2 mL of nitric acid solution (1 part 15.7 M acid to 1 part demineralized water) into each tube.

A1.7.7 Pipet 1 mL of 0.1 M silver nitrate solution into each tube.

A1.7.8 Stopper and mix thoroughly by inverting.

A1.7.9 Allow the tubes to stand in the dark for 5 min. Visually compare the sample to the standard solutions while looking vertically against a black background. Record the millilitres of standard Solution B that match the sample.

A1.8 Calculation

A1.8.1 Calculate the results as follows:

$$(A)(B)(0.001)(1\ 000\ 000)/(320/80)(4)(0.789) = \text{ppm Cl} \quad (\text{A1.1})$$

where:

A = mL of Solution B that matched sample,

B = mg Cl/mL of Solution B,

0.001 = mg to g,

320/80 = concentration factor,

4 = mL of sample, and

0.789 = relative density of ethanol at 20°C (68°F) compared to water at 4°C (39°F).

A1.9 Precision and Bias

A1.9.1 *Precision*—The precision of this test method for measuring inorganic chloride in fuel ethanol (Ed75-Ed85) is being determined.

A1.9.2 *Bias*—Since there is no accepted reference material for determining bias for the procedure in this test method for measuring inorganic chloride in fuel ethanol (Ed75-Ed85), bias has not been determined.

APPENDIX**(Nonmandatory Information)****X1. SIGNIFICANCE OF SPECIFICATION FOR FUEL ETHANOL (Ed75-Ed85) FOR AUTOMOTIVE SPARK-IGNITION ENGINES****X1.1 Ethanol**

X1.1.1 The ethanol content of fuel ethanol (Ed75-Ed85) is a critical parameter as it affects the capability of the fuel metering system of the dedicated Ed75-Ed85 vehicle to establish the proper air/fuel ratio for optimum vehicle operation. This is much less of a concern for multifuel-capable vehicles than for dedicated Ed75-Ed85 vehicles. Ethanol content may also affect the lubricating properties of the fuel, the water tolerance of the fuel, and the ability to meet cold and cool area volatility requirements.

X1.1.2 The inclusion of impurities, some denaturants, and contaminants, except for the deliberately added hydrocarbons or additives, or both, can impact adversely on the properties and performance of fuel ethanol (Ed75-Ed85) as an automotive spark-ignition engine fuel. The quantities of some of these materials are controlled by specified property limits. The limits on water, higher molecular weight alcohols, and methanol and on types of denaturants as well as minimums on the amount of ethanol and hydrocarbons limit, but do not prevent, the presence of trace materials.

X1.2 Hydrocarbon

X1.2.1 Hydrocarbons are deliberately added to provide improved cold startability and warm up driveability. The addition of hydrocarbon to fuel ethanol changes its volatility and can affect the flammability of fuel tank vapors.

X1.2.2 This specification does not control the composition of the hydrocarbons added to the denatured fuel ethanol. However the hydrocarbons shall be stable and noncorrosive and be in the boiling range of automotive spark-ignition engine fuel as specified in Specification D 4814.

X1.3 Vapor Pressure

X1.3.1 The addition of volatile hydrocarbons is required for adequate cold startability. The addition of hydrocarbons that are too volatile can contribute to hot fuel handling problems. Higher vapor pressures are required at colder ambient temperatures while lower volatility fuels are less prone to hot fuel handling problems at higher (summertime) ambient temperatures. Excessive vapor pressure contributes to evaporative emissions. Lower and upper limits on vapor pressure for the three volatility classes are used to define the acceptable range of volatile components to ensure adequate vehicle performance.

X1.4 Acidity

X1.4.1 Very dilute aqueous solutions of organic acids such as acetic acid are highly corrosive to a wide range of metals and alloys. It is therefore necessary to keep such acids at a very low level.

X1.5 pH_e

X1.5.1 When the pH_e of ethanol used as a fuel for automo-

tive spark-ignition engines is below 6.5, fuel pumps can malfunction as a result of a film forming between the brushes and commutator, fuel injectors can fail from corrosive wear, and excessive engine cylinder wear can occur. When the pH_e is above 9.0, fuel pump plastic parts can fail.

X1.6 Gum Content, Solvent Washed and Unwashed

X1.6.1 The test for solvent washed gum content measures the amount of residue after the evaporation of the fuel and following a heptane wash. The heptane wash removes the heptane-soluble, nonvolatile material, such as additives, carrier oils used with the additives, and diesel fuel. Unwashed gum content consists of fuel-insoluble and fuel-soluble gum. The fuel-insoluble portion can clog fuel filters. Both can be deposited on surfaces when the fuel evaporates.

X1.6.2 Solvent washed gum can contribute to deposits on the surface of carburetors, fuel injectors, and intake manifolds, ports, valves, and valve guides. The impact of solvent washed gum on malfunctions of modern engines that can operate on fuel ethanol (Ed75-Ed85) has not been fully established but is based on limited experience gained with M70-M85 fuels in field tests and from historic gasoline limits. Performance effects depend on where the deposits form; the presence of other deposit precursors, such as airborne debris, blowby and exhaust gas recirculation gases; oxidized engine oil; and the amount of deposit.

X1.6.3 The difference between the unwashed and solvent washed and gum content values can be used to assess the presence and amount of nonvolatile material in the fuel. Additional analytical testing is required to determine if the material is additive, carrier oil, diesel fuel, and so forth.

X1.6.4 The unwashed gum content limit is intended to limit high-boiling contaminants, like diesel fuel, that can affect engine performance, yet allow the use of appropriate levels of deposit control additives with carrier oils in fuel ethanol (Ed75-Ed85).

X1.6.5 Because the precision statements for Test Method D 381 were developed using only data on hydrocarbons, they may not be applicable to fuel ethanol (Ed75-Ed85).

X1.7 Total Chlorine

X1.7.1 Ionic (inorganic) and organic chlorine are corrosive to many metals, and it is desirable to minimize organic and ionic chlorine compounds in fuel ethanol (Ed75-Ed85).

X1.7.2 A total chlorine limit of 2 mg/kg, maximum, has been found to be inadequate in protecting some fuel system components. An inorganic chloride limit of 1 mg/kg, maximum, is specified to provide additional protection.

X1.8 Lead

X1.8.1 Most vehicles equipped to operate on fuel ethanol (Ed75-Ed85) are equipped with exhaust catalysts that control

emissions of aldehydes (formaldehyde and acetaldehyde) as well as regulated emissions. Lead compounds deactivate the catalyst and are therefore limited to trace amounts.

X1.9 Phosphorus

X1.9.1 Like lead, phosphorus deactivates exhaust catalysts and is limited to trace amounts.

X1.10 Appearance

X1.10.1 Turbidity, phase separation, or evidence of precipitation normally indicates contamination.

X1.11 Water

X1.11.1 The solubility of hydrocarbon in fuel ethanol (Ed75-Ed85) and blends with gasoline as may occur in multifuel-capable vehicles decreases with lowering temperature and increasing water content. Separation of the hydrocarbon from the fuel will adversely affect cold starting and driveability and denaturing. Water may affect the calibration of

some types of composition sensors of multifuel-capable vehicles. Water also reduces the energy content of the fuel and thus adversely affects fuel economy and power. Because some degree of water contamination is practically unavoidable in transport and handling, and because the fuel ethanol (Ed75-Ed85) is miscible with water, the water content of fuel ethanol (Ed75-Ed85) is limited to reduce the potential for problems.

X1.12 Copper

X1.12.1 Copper is a very active catalyst for low-temperature oxidation of hydrocarbons. Experimental work has shown that copper concentrations higher than 0.012 mg/kg in commercial gasolines may significantly increase the rate of gum formation.

X1.13 Sulfur

X1.13.1 The limit on sulfur content is included to protect against engine wear, deterioration of engine oil, corrosion of exhaust system parts, and exhaust catalyst deactivation.

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