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Standard Specification for Denatured Fuel Ethanol for Blending with Gasolines for Use as Automotive Spark-Ignition Engine Fuel¹

This standard is issued under the fixed designation D4806; 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 (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

- 1.1 This specification covers nominally anhydrous denatured fuel ethanol intended to be blended with unleaded or leaded gasolines at 1 to 10 volume % for use as a sparkignition automotive engine fuel. The significance of this specification is shown in Appendix X1.
- 1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.
- 1.2.1 *Exception*—Federal regulations use the inch-pound units that appear in Note 2, 5.1, and X1.2.1.

2. Referenced Documents

2.1 ASTM Standards:²

D86 Test Method for Distillation of Petroleum Products at Atmospheric Pressure

D381 Test Method for Gum Content in Fuels by Jet Evaporation

D891 Test Methods for Specific Gravity, Apparent, of Liquid Industrial Chemicals

D1152 Specification for Methanol (Methyl Alcohol)

D1613 Test Method for Acidity in Volatile Solvents and Chemical Intermediates Used in Paint, Varnish, Lacquer, and Related Products

D1688 Test Methods for Copper in Water

D2622 Test Method for Sulfur in Petroleum Products by Wavelength Dispersive X-ray Fluorescence Spectrometry

D3120 Test Method for Trace Quantities of Sulfur in Light Liquid Petroleum Hydrocarbons by Oxidative Microcoulometry D3505 Test Method for Density or Relative Density of Pure Liquid Chemicals

D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D4306 Practice for Aviation Fuel Sample Containers for Tests Affected by Trace Contamination

D4814 Specification for Automotive Spark-Ignition Engine Fuel

D5453 Test Method for Determination of Total Sulfur in Light Hydrocarbons, Spark Ignition Engine Fuel, Diesel Engine Fuel, and Engine Oil by Ultraviolet Fluorescence

D5501 Test Method for Determination of Ethanol Content of Denatured Fuel Ethanol by Gas Chromatography

D5580 Test Method for Determination of Benzene, Toluene, Ethylbenzene, *p/m*-Xylene, *o*-Xylene, C₉ and Heavier Aromatics, and Total Aromatics in Finished Gasoline by Gas Chromatography

D5854 Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products

D6423 Test Method for Determination of pHe of Ethanol, Denatured Fuel Ethanol, and Fuel Ethanol (Ed75-Ed85)

D6550 Test Method for Determination of Olefin Content of Gasolines by Supercritical-Fluid Chromatography

D7318 Test Method for Total Inorganic Sulfate in Ethanol by Potentiometric Titration

D7319 Test Method for Determination of Total and Potential Sulfate and Inorganic Chloride in Fuel Ethanol by Direct Injection Suppressed Ion Chromatography

D7328 Test Method for Determination of Total and Potential Inorganic Sulfate and Total Inorganic Chloride in Fuel Ethanol by Ion Chromatography Using Aqueous Sample Injection

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

E203 Test Method for Water Using Volumetric Karl Fischer Titration

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² 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.



E300 Practice for Sampling Industrial ChemicalsE1064 Test Method for Water in Organic Liquids by Coulometric Karl Fischer Titration

2.2 Other Standards:

United States Code of Federal Regulations, Title 27, Parts 19, 20, and 21³

3. Terminology

- 3.1 Definitions:
- 3.1.1 *ethanol*, n—ethyl alcohol, the chemical compound C_2H_5OH .
- 3.1.2 *gasoline*, *n*—a volatile mixture of liquid hydrocarbons, generally containing small amounts of additives, suitable for use as a fuel in spark-ignition, internal combustion engines.

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- 3.1.3 *gasoline-ethanol blend*, *n*—a fuel consisting primarily of gasoline along with a substantial amount (more than 0.35 mass % oxygen) of denatured fuel ethanol.

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- 3.1.4 *oxygenate*, *n*—an oxygen-containing, ashless, organic compound, such as an alcohol or ether, which may be used as a fuel or fuel supplement.

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 - 3.2 Definitions of Terms Specific to This Standard:
- 3.2.1 *denaturants*, *n*—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.
- 3.2.2 *denatured fuel ethanol*, *n*—fuel ethanol made unfit for beverage use by the addition of denaturants.
- 3.2.3 *fuel ethanol*, *n*—ethanol with impurities common to its production (including water but excluding denaturants).
- 3.2.4 *impurities*, *n*—in commercially produced fuel ethanol, compounds other than ethanol or denaturants present, such as methanol and fusel oil (for example, amyl and isoamyl alcohols).
- 3.2.5 *pHe*, *n*—a measure of the acid strength of alcohol fuels.

4. Performance Requirements

4.1 *Denatured Fuel Ethanol*—When fuel ethanol is denatured as specified in Section 5, it shall conform to the following requirements at the time of blending with a gasoline. (See Note 1 and Note 2.)

92.1 Ethanol, volume %, min Methanol, volume %, max 0.5 Solvent-washed gum, 5.0 mg/100 mL, max Water content, volume %, max 1.0 (Note 3) 1.96 Denaturant content, volume %, min volume %, max 5.0 Inorganic Chloride content, mass 10. (8) ppm (mg/L), max Copper content, mg/kg, max Acidity (as acetic acid CH₃COOH), 0.007 (56) (Note 4) mass % (mg/L), max рHe 65 to 90 Sulfur, mass ppm, max 30. Total sulfate, mass ppm, max Visibly free of suspended or precipi-Appearance tated contaminants (clear and bright)

Note 1—For purposes of determining conformance with these specification limits, an observed value or a calculated value shall be rounded "to the nearest unit" in the right-most significant digit used in expressing the specification limit, in accordance with the rounding method of Practice E29. For a specification limit expressed as an integer, a trailing zero is significant only if the decimal point is specified. For a specified limit expressed as an integer, and the right-most digit is non-zero, the right-most digit is significant without a decimal point being specified. This convention applies to specified limits in this table (4.1) and will not be observed in the remainder of this specification.

Note 2—If denatured fuel ethanol is prepared by the addition of denaturants to undenatured fuel ethanol after it has been produced rather than during the dehydration process, the 15.56/15.56°C (60/60°F) specific gravity in air of the undenatured fuel ethanol shall be in the range from 0.7937–0.7977.

Note 3—In some cases, a lower water content may be necessary to avoid phase separation of a gasoline-ethanol blend at very low temperatures. This reduced water content, measured at the time of delivery, shall be agreed upon between the supplier and purchaser.

Note 4—Denatured fuel ethanol may contain additives, such as corrosion inhibitors and detergents, that may affect the titratable acidity (acidity as acetic acid) of the finished fuel ethanol. Although the base fuel ethanol may meet the acidity specification, the effect of these additives may produce an apparent high titratable acidity of the finished product. Contact the ethanol supplier if there is a question regarding the titratable acidity of your denatured fuel ethanol to verify that the base ethanol meets the acidity requirements of 4.1.

4.2 Other Properties—Limits more restrictive than those specified above, or the specification of additional properties such as color, may be agreed upon between the supplier and the purchaser.

5. Denaturants

5.1 The only denaturants used for fuel ethanol shall be natural gasoline, gasoline components, or unleaded gasoline at a minimum concentration of two parts by volume per 100 parts by volume of fuel ethanol. One denatured formula specifically designed for fuel use by the Alcohol and Tobacco Tax and Trade Bureau (TTB) of the U.S. Treasury Department is Formula C.D.A. 20. It requires that for every 100 gal of ethanol of not less than 195 proof, a total of 2.0 gal of denaturant be added. Another fuel alcohol rendered unfit for beverage use

³ Order as Code of Federal Regulations Title 27 Parts 1–39: from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401 or the online store at www.gpo.gov.

and manufactured at an alcohol fuel plant (AFP) requires the addition of 2 gal or more of materials listed by the director to each 100 gal of ethanol. The fuel ethanol formulas approved by the U.S. Treasury Department include materials, which are not allowed by this ASTM specification. This specification prohibits the use of hydrocarbons with an end boiling point higher than 225°C (437°F) as determined by Test Method D86, although they may be permitted by TTB regulations. Some kerosines, for instance, promote piston scuff in automotive engines. The denaturants permitted by this specification may be included as part of the 10 volume % denatured fuel ethanol blended with a gasoline if they do not exceed five volume % of fuel ethanol. Any part of these denaturants that are present at concentrations higher than five volume % of fuel ethanol are considered as part of the base gasoline. The maximum denaturant limits are specified by United States Internal Revenue Service (IRS) regulations.

Note 5—TTB regulations concerning the preparation, use, and handling of denatured ethanols are published in the United States Code of Federal Regulations, Title 27, Parts 19, 20, and 21. 27 CFR 19.1005 contains regulations for rendering fuel alcohol unfit for beverage use by an AFP. 27 CFR 21.24 contains the formula for manufacturing completely denatured alcohol, C.D.A. 20.

5.2 Prohibited Denaturants—Although this specification permits only hydrocarbons in the gasoline boiling range to be used as denaturants, specific mention must be made of some materials that have extremely adverse effects on fuel stability, automotive engines, and fuel systems. These materials shall not be used as denaturants for fuel ethanol under any circumstances. They are as follows: methanol which does not meet Specification D1152, pyrroles, turpentine, ketones, and tars (high-molecular weight pyrolysis products of fossil or nonfossil vegetable matter). While any significant amount of methanol will lower the water tolerance and increase the vapor pressure of a gasoline-ethanol blend, these effects become more serious when methanol is present at more than 2.5 parts by volume per 100 parts by volume of fuel ethanol. Also, methanol, which does not meet Specification D1152, frequently contains impurities, such as turpentine and tars. Similarly, ketone denaturants tend to degrade fuel stability or increase the tendency of a gasoline-ethanol blend to corrode metals and attack elastomers. These effects become more serious if the concentration of a ketone such as 4-methyl pentanone (methyl isobutyl ketone) exceeds one part by volume per 100 parts by volume of fuel ethanol. There is no information available on the effects of denaturants other than those mentioned above; but unless a denaturant, such as a higher aliphatic alcohol or ether, is known to have no adverse effect on a gasoline-ethanol blend or on automotive engines or fuel systems, it shall not be used.

6. Workmanship

- 6.1 The fuel ethanol shall be visually free of sediment and suspended matter. It shall be clear and bright at the ambient temperature or 21°C, whichever is higher.
- 6.2 The specification defines only a basic purity for this product. The product shall be free of any adulterant or contaminant that may render the material unacceptable for its commonly used applications.

7. Sampling, Containers, and Sample Handling

- 7.1 The reader is strongly advised to review all intended test methods prior to sampling to understand the importance and effects of sampling technique, proper containers, and special handling required for each test method.
- 7.2 Correct sampling procedures are critical to obtain a sample representative of the lot intended to be tested. Use appropriate procedures in Practice D4057 or Practice E300 for manual method sampling and in PracticeD4177 for automatic method sampling, as applicable.
- 7.3 The correct sample volume and appropriate container selection are important decisions that can impact test results. Refer to Practice D4306 for aviation fuel container selection for tests sensitive to trace contamination. Refer to Practice D5854 for procedures on container selection and sample mixing and handling. All sampling and storage containers should be evaluated for durability and contamination of fuel ethanol prior to use. If samples must be collected in metal containers, do not use soldered metal containers. Soldering flux in the containers and the lead in the solder can contaminate the sample.
- 7.4 Sample Size—A minimum of about 1 L is recommended. If specific gravity is to be determined by a hydrometer method, additional volume may be required. This depends on the size of the hydrometer.
- 7.5 Lot Size—A lot shall normally consist of the amount contained in a tanker compartment or other bulk container in which it is delivered. If this definition does not apply, the definition of a lot must be agreed upon between the supplier and purchaser.

Note 6—See Sections 5, 6, and 7 on Significance, Safety, and Statistical Considerations, respectively, of Practice E300 for a detailed discussion of the statistics of sampling.

8. Test Methods

- 8.1 The scope of some of the test methods specified in 8.2-8.10 do not include denatured fuel ethanol. The precisions of those test methods may differ from the reported precisions when testing denatured fuel ethanol.
 - 8.2 Water Content—Test Methods E203 or E1064.
- 8.3 Solvent-Washed Gum Content—Test Method D381, air jet apparatus.
 - 8.4 *Acidity*—Test Method D1613.
 - 8.5 pHe—Test Method D6423.
- 8.6 Appearance—The product shall be visibly free of suspended or precipitated contaminants (clear and bright). This shall be determined at indoor ambient temperature unless otherwise agreed upon between the supplier and the purchaser.
- 8.7 Specific Gravity—Test Methods D891, Procedure B or Test Method D4052. For Test Methods D891, Procedure B (hydrometer), no formal precision statement is available, but practical experience indicates that precision is no better than 0.0005. Test Methods D891 Procedure C (pycnometer), with an interlaboratory precision (reproducibility) of 0.0002, should be used as a referee method.
- 8.8 *Inorganic Chloride Content*—Test Methods D7319 and D7328.



- 8.9 *Copper Content*—Modification of Test Methods D1688, Test Method A.
- 8.9.1 The modifications of Test Methods D1688, Test Method A (atomic absorption, direct) consists of mixing reagent-grade ethanol (which may be denatured in accordance with TTB Formula 3A or 30) in place of water as the solvent or diluent for the preparation of reagents and standard solutions. However, this must not be done to prepare the stock copper solution described in the section on Copper Solution, Stock in Test Method D1688. Because a violent reaction may occur between the acid and the ethanol, use water, as specified, in the acid solution part of the procedure to prepare the stock copper solution. Use ethanol for the rinse and final dilution only.
- 8.9.2 The precision of this modified method has not been determined, but it is expected to be similar to the precision of Test Method D1688, Test Method A.
 - 8.10 Ethanol and Methanol Contents—Test Method D5501.
- 8.11 *Sulfur Content*—Test Methods D2622, D3120, or D5453. California specifies that compliance with the California sulfur standard for denatured ethanol shall be determined using Test Method D5453–93. EPA allows Test Methods D3120 or

- D5453 for measuring sulfur in gasoline as long as these alternative test method results are correlated to the EPA designated Test Method D2622 when determining compliance with Federal EPA sulfur standards.
- 8.12 *Total Sulfate Content*—Test Methods D7318, D7319, and D7328.
- 8.13 Denaturant Content—Denaturant is added in the specified range to comply with federal regulations. The content is set by volumetric addition during the denaturing process. There is no standardized test procedure to directly determine the denaturant content in fuel ethanol. Current analytical procedures only provide a calculated estimate of the denaturant content, which is not sufficiently accurate for determining compliance.

9. Keywords

9.1 acidity; automotive spark-ignition engine fuel; base gasoline; chloride ion content; copper content; corrosion inhibitors; denaturants; denatured fuel ethanol; ethanol; ethanol content; ethanol purity; fuel; fuel ethanol; gasoline; gasoline-ethanol blend; impurities; oxygenate; solvent-washed gum; sulfate ion content; sulfur content; water content

APPENDIXES

(Nonmandatory Information)

X1. SIGNIFICANCE OF SPECIFIED PROPERTIES

X1.1 Denatured Fuel Alcohol

X1.1.1 Water Content—Karl Fischer analysis is generally the only consistently reliable procedure for the determination of water in denatured ethanol. Test Method E203 describes the modifications required to run the test in the presence of alcohols. Specific gravity methods such as Test Methods D891 and D3505, are generally unsuitable for the reasons given in X1.2.1. Blends of fuel ethanol and gasoline have a limited solvency for water. This solvency will vary with the ethanol content, the temperature of the blend, and the aromatic content of the base gasoline. A fuel made by blending 10 volume % fuel ethanol with a gasoline containing 14 volume % aromatics and 0.6 mass % dissolved water (about 0.5 volume %), will separate into a lower alcohol-rich aqueous phase and an upper hydrocarbon phase if cooled to about 7°C. As normal sparkignition engines will not run on the aqueous phase material, such a separation is likely to cause serious operating problems. Because some degree of water contamination is practically unavoidable in transport and handling, and because gasolineethanol blends are hygroscopic, the water content of the denatured fuel ethanol must be limited when it is blended with gasoline to reduce the risk of phase separation.

X1.1.2 Solvent-Washed Gum Content:

X1.1.2.1 The test for solvent-washed gum content measures the amount of residue after 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 additives, and diesel fuels. Solvent-washed gum

consists of fuel-insoluble gum and fuel-soluble gum. The fuel-insoluble portion can clog fuel filters. Both can be deposited on surfaces when the fuel evaporates.

X1.1.2.2 Solvent-washed gum can contribute to deposits on the surfaces of carburetors, fuel injectors, and intake manifolds, ports, valves, and valve guides. The impact of solvent-washed gum from pure alcohols such as ethanol on malfunctions of modern engines is not known. The test method is used essentially to detect the presence of high boiling, heptane-insoluble impurities.

X1.1.2.3 Because the precision statements for Test Method D381 were developed using only data on hydrocarbons, they may not be applicable to denatured fuel ethanol.

X1.1.3 *Chloride Ion Content*—Low concentrations of chloride ions are corrosive to many metals.

X1.1.4 Copper Content—Copper is a very active catalyst for the low-temperature oxidation of hydrocarbons. Experimental work has shown that copper concentrations higher than 0.012 mass ppm in commercial gasolines may significantly increase the rate of gum formation.

X1.1.5 *Acidity*—Very dilute aqueous solutions of low-molecular weight organic acids such as acetic (CH₃COOH) are highly corrosive to many metals. It is therefore necessary to keep such acids at a very low level.

X1.1.6 *pHe*—When the pHe of ethanol used as a fuel for automotive spark-ignition engines is below 6.5, fuel pumps can malfunction as a result of film forming between the brushes and commutator, fuel injectors can fail from corrosive wear,

and excessive engine cylinder wear can occur. When the pHe is above 9.0, fuel pump plastic parts can fail. The adverse effects are less when ethanol is used at 10 volume % in a blend with gasoline.

X1.1.7 *Appearance*—Turbidity or evidence of precipitation normally indicates major contamination.

X1.1.8 Ethanol Purity—The presence of even small quantities of some organic oxygen compounds other than ethanol may adversely affect the properties of fuel ethanol-gasoline blends.

X1.1.9 *Total Sulfate Content*—The presence of small amounts of inorganic sulfates in denatured ethanol under the right conditions can contribute to turbine meter deposits and the premature plugging of fuel dispensing pump filters in the fuel distribution system. The sulfates also have been shown to cause fuel injector sticking resulting in engine misfiring and poor driveability in automobiles.

X1.1.10 *Denaturant*—Hydrocarbon denaturant (see Section 5) is required by federal regulations for denatured fuel ethanol. The regulations limit both the minimum and maximum amount that can be used. Denatured ethanol producers often use meters

to control the amount of denaturant present. Current analytical procedures only provide an estimate of the denaturant content.

X1.2 Undenatured Ethanol

X1.2.1 Specific Gravity—The density of a water-ethanol mixture is primarily a function of its water content. Normal U.S. industry practice and Federal regulations call for the use of the 15.56/15.56°C (60/60°F) specific gravity in air as the control method for water content of undenatured ethanol. Because the addition of denaturants will normally affect specific gravity, specific gravity methods are generally not suitable for determining the water content of denatured ethanols

X1.2.2 Sulfur Content—The Federal Tier 2 Motor Vehicle and Emissions Standards and Gasoline Sulfur Control Requirements establish sulfur standards for refineries and importers producing reformulated gasoline, Reformulated Blendstock for Oxygenate Blending (RBOB), and conventional gasoline. EPA has established gasoline sulfur controls to support vehicle emission standards. Sulfur contaminates the catalytic converter necessary for reducing emissions of HC, CO, and NOx.

X2. CALIFORNIA ETHANOL REQUIREMENTS

X2.1 California Ethanol Requirements

X2.1.1 The California Air Resources Board has approved standards for denatured ethanol to be field-blended with California Reformulated Gasoline Blendstock for Oxygenate Blending (CARBOB) to make California Phase 3 Reformulated Gasoline (CaRFG3). Standards also have been specified for the denaturant. These California standards for denatured ethanol and denaturant became effective Dec. 31, 2003 and were amended on Aug. 29, 2008.

X2.1.2 The California standards for denatured ethanol set maximum limits on sulfur, benzene, olefins, and aromatics contents as shown in Table X2.1, and also require the denatured ethanol to comply with the performance requirements in Specification D4806-99.

X2.1.3 California specifies that compliance with the sulfur standard shall be determined by testing the denatured ethanol using Test Method D5453-93. California specifies that compliance with the standards for benzene, olefins, and aromatics contents shall be determined by testing a sample of the denaturant using the test methods specified for CARB gasoline, and then calculating the content of those compounds in the denatured ethanol, multiplying the test value by 0.0500 (except where it is demonstrated that the denatured ethanol contains less than 5.00 % denaturant, then the test results are multiplied by the decimal fraction representing the percent denaturant).

X2.1.4 California allows an exception to the limits shown in Table X2.1 where the denatured ethanol supplier takes reasonably prudent precautions to ensure the denatured ethanol that exceeds these limits will only be added to a specially designed CARBOB which has been designated to be blended with such denatured ethanol. Documentation is required to support the

transfer of denatured ethanol. All CaRFG3 requirements for the final blend shall be met.

X2.1.5 California specifies the standards for the denaturant used in denatured ethanol as shown in Table X2.2. Also shown are the test methods required to determine compliance.

X2.1.6 California standards allow higher amounts of benzene, olefins, and aromatics in the denaturant if the supplier takes necessary precautions to ensure that when added to the ethanol, the level is less than 5.00 vol % and the limits in Table X2.1 are met.

TABLE X2.1 California Denatured Ethanol Standards (In Addition to the Performance Requirements in ASTM D4806-99)

Property	Specification Limit	n Test Method
Sulfur, mass ppm, max	(10	D5453-93
Benzene, vol % max	0.06	D5580-00 test results of a sample of the denaturant multiplied by 0.0500 (see X2.1.3 for exceptions)
Olefins, vol % max	0.5	D6550-00 (modified) test results of a sample of the denaturant multiplied by 0.0500 (see X2.1.3 for exceptions)
Aromatics, vol % max	1.7	D5580-00 test results of a sample of the denaturant multiplied by 0.0500 (see X2.1.3 for exceptions)

TABLE X2.2 California Denaturant Standards

Property	Specification Limit	Test Method
Benzene, vol % max	1.10	D5580-00
Olefins, vol % max	10.0	D6550-00 (modified)
Aromatics, vol % max	35.0	D5580-00

SUMMARY OF CHANGES

Subcommittee D02.A0 has identified the location of selected changes to this standard since the last issue (D4806–08a) that may impact the use of this standard. (Approved June 1, 2009.)

- (1) Revised 2.2 to update references to Code of Federal Regulations.
- (2) Revised 2.2 to delete reference to Federal Specification O-E-760b and corresponding footnote.
- (3) Added parts of speech to terms in 3.2.
- (4) Revised 8.10 to include test method for methanol.
- (5) Updated 4.1, 8.12, and X1.1.9 to add "total" before sulfate.
- (6) Updated 8.9.1 to change the name of federal agency.

Subcommittee D02.A0 has identified the location of selected changes to this standard since the last issue (D4806–08) that may impact the use of this standard. (Approved Dec. 1, 2008.)

- (1) Incorporated lowered inorganic chloride content in 4.1.
- 2008) for CARB ethanol specifications in Appendix X2.
- (2) Incorporated recently approved updates (from August

Subcommittee D02.A0 has identified the location of selected changes to this standard since the last issue (D4806–07a) that may impact the use of this standard. (Approved July 1, 2008.)

(1) Revised 7.3.

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