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Standard Specification for Butanol for Blending with Gasoline for Use as Automotive Spark-Ignition Engine Fuel¹

This standard is issued under the fixed designation D7862; 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-Scope*

1.1 This specification covers butanol intended to be blended with gasoline at 1 to 12.5 volume % for use as an automotive spark-ignition engine fuel.

1.1.1 Butanol contains 22 mass % oxygen. The mass % of oxygen of a butanol blend with gasoline depends on the volume % of butanol blended, the density of the butanol isomer and the density of the base blendstock.

1.1.2 The maximum limit on blending is not a performance limit but a current regulatory limit in the United States.

1.2 This specification covers three butanol isomers:1-butanol, 2-butanol, and 2-methyl-1-propanol. This specification specifically excludes 2-methyl-2-propanol (that is, *tert*-butyl alcohol).

1.2.1 Tert-butyl alcohol has different physical properties (melting point, water miscibility) than the other three isomers.

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

1.4 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 Documents

2.1 ASTM Standards:²

- D381 Test Method for Gum Content in Fuels by Jet Evaporation
- D1298 Test Method for Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method
- D1613 Test Method for Acidity in Volatile Solvents and Chemical Intermediates Used in Paint, Varnish, Lacquer, and Related Products
- 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
- 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
- D4175 Terminology Relating to Petroleum, Petroleum Products, and Lubricants
- 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
- D5854 Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products
- D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance
- D7319 Test Method for Determination of Existent and Potential Sulfate and Inorganic Chloride in Fuel Ethanol and Butanol by Direct Injection Suppressed Ion Chromatography

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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.



D7875 Test Method for Determination of Butanol and Acetone Content of Butanol for Blending with Gasoline by Gas Chromatography

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with SpecificationsE203 Test Method for Water Using Volumetric Karl Fischer TitrationE300 Practice for Sampling Industrial ChemicalsE1064 Test Method for Water in Organic Liquids by Coulometric Karl Fischer Titration

3. Terminology

3.1 For general terminology, refer to Terminology D4175.

NOTE 1—The user is advised that the definitions used by various industries, marketers, and regulatory bodies can differ from those specific to this specification. It is the responsibility of the user to ensure that the terms used in a particular context are clearly understood.

3.2 Definitions:

3.2.1 *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. D4814

3.2.2 *oxygenate, n*—an oxygen-containing, ashless, organic compound, such as an alcohol or ether, which may be used as a fuel or fuel supplement. D4814

3.3 Definitions of Terms Specific to This Standard:

3.3.1 *butanol, n*—butanol or butyl alcohol refers to 1-butanol, 2-butanol, and 2-methyl 1-propanol, three isomeric alcohols with the molecular formula C_4H_9OH , either individually or as mixtures.

4. Performance Requirements

4.1 *Butanol*—Butanol shall conform to the requirements shown in Table 1 at the time of blending with a gasoline. (See Note 2.)

NOTE 2—Commercial processes used to manufacture butanol from biological feedstock typically yield some fusel oil or alcohols such as pentanol and other higher alcohols.

4.1.1 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 Table 1 and will not be observed in the remainder of this specification.

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. Workmanship

5.1 The butanol 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.

5.2 The product shall be free of any adulterant or contaminant that can render the material unacceptable for its commonly used applications.

IADLE I Requirements		
Property	Limit	Method
Butanol, volume %, min	96.0	Annex A1
Butanol, volume %, min	<u>96.0</u>	D7875
1-butanol, volume %	Report	Annex A1
1-butanol, volume %	Report	<u>D7875</u>
2-butanol, volume %	Report	Annex A1
2-butanol, volume %	Report	D7875
2-methyl 1-propanol, volume %	Report	Annex A1
2-methyl 1-propanol, volume %	Report	D7875
Methanol, volume % max	0.4	Annex A1
Methanol, volume % max	0.4	<u>D7875</u>
Water content, volume %, max	1.0	E203 or E1064
Acidity (as acetic acid CH ₃ COOH), mass % (mg/L), max	0.007 (56)	D1613
Inorganic Chloride, mg/kg (mg/L), max	8 (6)	D7319
Solvent-washed gum, mg/100 mL, max	5.0	D381
Sulfur, mg/kg, max	30.	D2622, D5453
Existent sulfate, mg/kg, max	4.	D7319

TABLE 1 Requirements



5.2.1 Manufacturers and importers of butanol shall avoid butanol contaminated by silicon-containing materials. Silicon contamination of gasoline-oxygenate blends has led to fouled vehicle components (for example, spark plugs, exhaust oxygen sensors, catalytic converters) requiring parts replacement and repairs.

5.2.2 Manufacturers and importers of butanol shall avoid butanol contaminated by acetone. Acetone contamination of gasoline-oxygenate blends can degrade elastomers used in fuel system components as well as paint and/or clearcoat finishes used on vehicles.

6. Sampling, Containers, and Sample Handling

6.1 The user 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.

6.2 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 Practice D4177 for automatic method sampling, as applicable.

6.3 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 butanol prior to use. Butanol may be sampled in glass containers; however sodium leaching from glass containers has been shown to interfere with sulfate analysis. HDPE (high density polyethylene) containers may be used in place of glass to avoid sodium leaching. If samples must be collected in metal containers, do not use soldered metal containers. Soldering flux in the containers and lead in the solder can contaminate the sample.

6.4 Sample Size-A minimum of about 1 L or 1 U.S. qt is recommended.

6.5 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 shall be agreed upon between the supplier and purchaser. Note 3—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.

7. Test Methods

7.1 The scope of some of the test methods listed below do not include butanol. The precisions of those test methods can differ from the reported precisions when testing butanol.

7.2 Water Content—Test Methods E203 or E1064.

7.3 Solvent-Washed Gum Content-Test Method D381, air jet apparatus.

7.4 *Acidity*—Test Method D1613.

7.5 *Sulfur Content*—In the United States, US EPA allows Test Methods D3120 or D5453 for measuring sulfur in gasoline as long as these alternative test method results are correlated to the US EPA designated Test Method D2622 when determining compliance with US Federal EPA sulfur standards.

7.6 Inorganic Chloride—Test Method D7319.

7.7 Butanol (1-butanol, 2-butanol, 2-methyl 1-propanol)—SeeTest Annex A1Method D7875for a test method.

7.8 Methanol—SeeTest Annex A1Method D7875for a test method.

7.9 Total Sulfate—Test Method D7319.

8. Keywords

8.1 acidity; automotive spark-ignition engine fuel; base gasoline; bio-butanol; butanol; chloride ion content; corrosion inhibitors; fuel; gasoline; gasoline-butanol blend; impurities; oxygenate; solvent-washed gum; sulfate ion content; sulfur content; water content

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ANNEX

(Mandatory Information)

A1. TEST METHOD FOR DETERMINATION OF BUTANOL AND ACETONE CONTENT OF BUTANOL FOR BLENDING WITH GASOLINE BY GAS CHROMATOGRAPHY

A1.1. Scope

A1.1.1 This test method covers the gas chromatographic determination of the butanol content of butanol for blending with gasoline.

A1.1.2 Butanol is determined from 95 to 99.9 mass %, acetone is determined from 0.02 to 1.5 mass %, ethanol is determined from 0.02 to 1.5 mass %. Equations used to convert these individual components from mass % to volume % are provided.

A1.1.3 This test method does identify and quantify acetone but does not purport to identify all individual components that can be present in butanol for gasoline blending.

A1.1.4 Water is determined by the test method referenced in Table 1.

A1.1.5 This test method is inappropriate for impurities that boil at temperatures higher than 225°C or for impurities that cause poor or no response in a flame ionization detector, such as water.

A1.1.6 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

A1.1.7 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.

A1.2. Referenced Documents

ASTM D7862-15

A1.2.1 ASTM Standards:³ D1364 Test Method for Water in Volatile Solvents (Karl Fischer Reagent Titration Method)

D1304 Test Method for Water In Volatic Solvents (Karr Fischer Reagent Fination Method D4307 Practice for Preparation of Liquid Blends for Use as Analytical Standards

D4626 Practice for Calculation of Gas Chromatographic Response Factors

E355 Practice for Gas Chromatography Terms and Relationships

E594 Practice for Testing Flame Ionization Detectors Used in Gas or Supercritical Fluid Chromatography

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

A1.3. Terminology

A1.3.1 *Definitions*—This test method makes reference to many common gas chromatographic procedures, terms, and relationships. Detailed definitions can be found in Practices E355 and E594.

A1.3.2 Definitions of Terms Specific to This Standard:

A1.3.2.1 *butanol,* n—for the purposes of this method, butanol or butyl alcohol refers to 3 of the structural isomers of butanol = 1-butanol, 2-butanol, and 2-methyl-1-propanol.

A1.3.2.1.1 Discussion—

This method has not been evaluated for use with the butanol isomer 2-methyl-2-propanol.



A1.4. Summary of Test Method

A1.4.1 A representative aliquot of the butanol sample is introduced into a gas chromatograph equipped with a polydimethylsiloxane bonded phase capillary column. Helium carrier gas transports the vaporized aliquot through the column where the components are separated by the chromatographic process. Components are sensed by a flame ionization detector as they elute from the column. The detector signal is processed by an electronic data acquisition system. The butanol, acetone, ethanol, and methanol components are identified by comparing their retention times to the ones identified by analyzing standards under identical conditions. The concentrations of all components are determined in mass percent area by normalization of the peak areas.

A1.5. Significance and Use

A1.5.1 Butanol is a potential blendstock for blending with gasoline provided that it meets a purity specification of >96.0 % butanol in accordance with Specification D7862. This test method provides a method of determining the percentage of butanol (purity) of the butanol for blending with gasoline.

A1.6. Apparatus

A1.6.1 *Gas Chromatograph*, capable of operating at the conditions listed in Table A1.1. A heated flash vaporizing injector designed to provide a linear sample split injection (for example, 200:1) is required for proper sample introduction. Carrier gas controls shall be of adequate precision to provide reproducible column flows and split ratios in order to maintain analytical integrity. Pressure control devices and gauges shall be designed to attain the linear velocity required in the column used. A hydrogen flame ionization detector with associated gas controls and electronics, designed for optimum response with open tubular columns, is required.

iTeh Standards

A1.6.2 Sample Introduction, Manual or automatic liquid syringe sample injection to the splitting injector is employed. Devices capable of 1.0 µL injections are suitable. It should be noted that inadequate splitter design, poor injection technique, and overloading the column can result in poor resolution. Avoid overloading, particularly of the butanol peak(s), and eliminate this condition during analysis.

A1.6.3 Column, This test method utilizes a fused silica open tubular column with non-polar polydimethylsiloxane bonded (cross-linked) phase internal coating. Any column with equivalent or better chromatographic efficiency and selectivity to those described in A1.6.3.1 may be used.

A1.6.3.1 Open tubular column with a non-polar polydimethylsiloxane bonded (cross-linked) phase internal coating 150 m long by 0.25 mm internal diameter with a 1.0 µm film thickness has been found to be suitable.

A1.6.4 *Electronic Data Acquisition System*, Any data acquisition and integration device used for quantification of these analyses shall meet or exceed these minimum requirements:

A1.6.4.1 Capacity for at least 80 peaks/analysis,

A1.6.4.2 Normalized percent calculation based on peak area and using response factors,

A1.6.4.3 Identification of individual components based on retention time,

A1.6.4.4 Noise and spike rejection capability,

A1.6.4.5 Sampling rate for narrow (<1 s) peaks,

A1.6.4.6 Positive and negative sloping baseline correction,

A1.6.4.7 Peak detection sensitivity compensation for narrow and broad peaks, and

A1.6.4.8 Non-resolved peaks separated by perpendicular drop or tangential skimming as needed.

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A1.7. Reagents and Materials

A1.7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.⁴

A1.7.2 *Carrier Gas*, helium, with a minimum purity of 99.95 mol %. Oxygen removal systems and gas purifiers should be used. (Warning—Helium, compressed gas under high pressure.)

A1.7.3 Detector Gases, hydrogen, air, and nitrogen. The minimum purity of the gases used should be 99.95 % for the hydrogen and nitrogen. The air should be hydrocarbon-free grade. Gas purifiers are recommended for the detector gases. (Warning—Hydrogen, extremely flammable gas under high pressure.) (Warning—Air and nitrogen, compressed gases under high pressure.)

A1.7.4 *Standards for Calibration and Identification*—Standards of all components to be analyzed are required for establishing identification by retention time as well as calibration for quantitative measurements. These materials shall be of known purity and free of the other components to be analyzed.

A1.7.4.1 2-Methyl-1-propanol or isobutanol (Warning-Flammable and may be harmful or fatal, if ingested or inhaled.)

A1.7.4.2 1-Butanol or normal butanol (Warning-Flammable and may be harmful or fatal, if ingested or inhaled.)

A1.7.4.3 2-Butanol or secondary butanol (Warning-Flammable and may be harmful or fatal, if ingested or inhaled.)

A1.7.4.4 Acetone (Warning-Flammable and may be harmful or fatal, if ingested or inhaled.)

A1.7.4.5 3-Methyl-1-butanol (Warning-Flammable and may be harmful or fatal, if ingested or inhaled.)

A1.7.4.6 2-Propanol (Warning—Flammable and may be harmful or fatal, if ingested or inhaled.)

A1.7.4.7 2-Butanone (Warning-Flammable and may be harmful or fatal, if ingested or inhaled.)

A1.7.4.8 Methanol (Warning-Flammable and may be harmful or fatal, if ingested or inhaled.)

A1.7.4.9 Ethanol (Warning-Flammable and may be harmful or fatal, if ingested or inhaled.) 9a9ec4b/astm-d7862-15

Note A1.1-Two grades of ethanol are available. Only absolute ethanol 99.5 minimum percent meets the requirements of this test method.

A1.8. Sampling

A1.8.1 Butanol may be sampled into an open container since a vapor pressure of less than 21 kPa is expected. Refer to Practice D4057 for instruction on manual sampling from bulk storage into open containers. Stopper container immediately after drawing the sample.

A1.8.2 Transfer an aliquot of the sample into a septum vial and seal. Obtain the test sample for analysis directly from the sealed septum vial, for either manual or automatic syringe injection.

A1.9. Preparation of Apparatus

A1.9.1 Install and condition the column in accordance with manufacturer's or supplier's instructions. After conditioning, attach column outlet to flame ionization detector inlet and check for leaks throughout the system. When leaks are found, tighten or replace fittings before proceeding.

A1.9.2 Adjust the carrier gas flow rate so that the average linear gas velocity, at the initial temperature of the run, is between 18 and 20 cm/s, as determined by the following equation:

$$\frac{L}{t_m}$$
 (A1.1)

 $\bar{\mu} =$