



Standard Test Method for Existent Inorganic Sulfate in Ethanol by Potentiometric Titration¹

This standard is issued under the fixed designation D7318; 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 test method covers a potentiometric titration procedure for determining the existent inorganic sulfate content of hydrous, anhydrous ethanol, and anhydrous denatured ethanol, which is added as a blending agent with spark ignition fuels. It is intended for the analysis of denatured ethanol samples containing between 1.0–20 mg/kg existent inorganic sulfate.

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

1.3 *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.* Material Safety Data Sheets are available for reagents and materials. Review them for hazards prior to usage.

2. Referenced Documents

2.1 ASTM Standards:²

D1193 Specification for Reagent Water

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

D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance

3. Terminology

3.1 Definitions:

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.03 on Elemental Analysis.

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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.

3.1.1 *existent inorganic sulfate, n*—inorganic sulfate species actually present in the sample at the time of analysis with no oxidation treatment.

3.1.1.1 *Discussion*—Specifically in this test method, inorganic sulfate is present as sulfate in ethanol.

3.1.2 *inorganic sulfate, n*—sulfate (SO_4^{2-}) species present as sulfuric acid, ionic salts of this acid, or mixtures of these.

4. Summary of Test Method

4.1 An ethanol sample containing inorganic sulfate is titrated in ethanolic medium with a standard lead nitrate solution. Lead sulfate precipitate is formed during the titration. Perchloric acid is added to remove possible interference from carbonate. The endpoint is signaled by an increase in lead ion activity, as measured by a lead-selective electrode.

5. Significance and Use

5.1 Ethanol is used as a blending agent added to gasoline. Sulfates are indicated in filter plugging deposits and fuel injector deposits. When fuel ethanol is burned, sulfates may contribute to sulfuric acid emissions. Ethanol acceptability for use depends on the sulfate content. Sulfate content, as measured by this test method, can be used as one measure of determination of the acceptability of ethanol for automotive spark-ignition engine fuel use.

6. Apparatus

6.1 *Potentiometric Titration Assembly*—A titration assembly consisting of an automatic titrator fitted with a lead ion-selective electrode, a double-junction reference electrode, buret, and stirring is used. Stirring may be accomplished by means of magnetic or propeller type stirrer mechanisms. The buret size should ideally be 10 or 20 mL.

6.2 *Reference Electrode*—A double junction reference electrode with the inner electrode composed of silver/silver chloride with a potassium chloride solution as internal electrolyte. The external solution is composed of 1 M lithium chloride in ethanol. This configuration is used to prevent silver ion, a lead electrode poison, from leaching into the analyte solution during titration. Preferred electrolytes for use in double junction electrodes may vary with the manufacturer; use the manufacturer's recommended electrolytes for the application. Other

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