



Designation: D7328 – 23

Standard Test Method for Determination of Existent and Potential Inorganic Sulfate and Total Inorganic Chloride in Fuel Ethanol by Ion Chromatography Using Aqueous Sample Injection¹

This standard is issued under the fixed designation D7328; 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 an ion chromatographic procedure for the determination of the existent inorganic and potential sulfate and total inorganic chloride content in hydrous and anhydrous denatured ethanol to be used in motor fuel applications. It is intended for the analysis of ethanol samples containing between 0.55 mg/kg and 20 mg/kg of existent inorganic sulfate, 4.0 mg/kg to 20 mg/kg of potential inorganic sulfate, and 0.75 mg/kg to 50 mg/kg of total inorganic chloride.

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, health, and environmental 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

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 ASTM Standards:²

D1193 Specification for Reagent Water

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.

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 Products, Liquid Fuels, and Lubricants

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D5827 Test Method for Analysis of Engine Coolant for Chloride and Other Anions by Ion Chromatography

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

D6792 Practice for Quality Management Systems in Petroleum Products, Liquid Fuels, and Lubricants Testing Laboratories

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

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

2.2 Other Standard:

ISO/CEN15492 Ethanol as a Blending Component for Petrol—Determination of Inorganic Chloride—Ion Chromatographic Method³

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in this test method, refer to Terminology **D4175**.

3.2 Definitions of Terms Specific to This Standard:

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

3.2.2 *inorganic chloride, n*—chloride present as hydrochloric acid, ionic salts of this acid, or mixtures of these.

³ Available from International Organization for Standardization (ISO), 1 rue de Varembe, Case postale 56, CH-1211, Geneva 20, Switzerland, <http://www.iso.ch>.

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

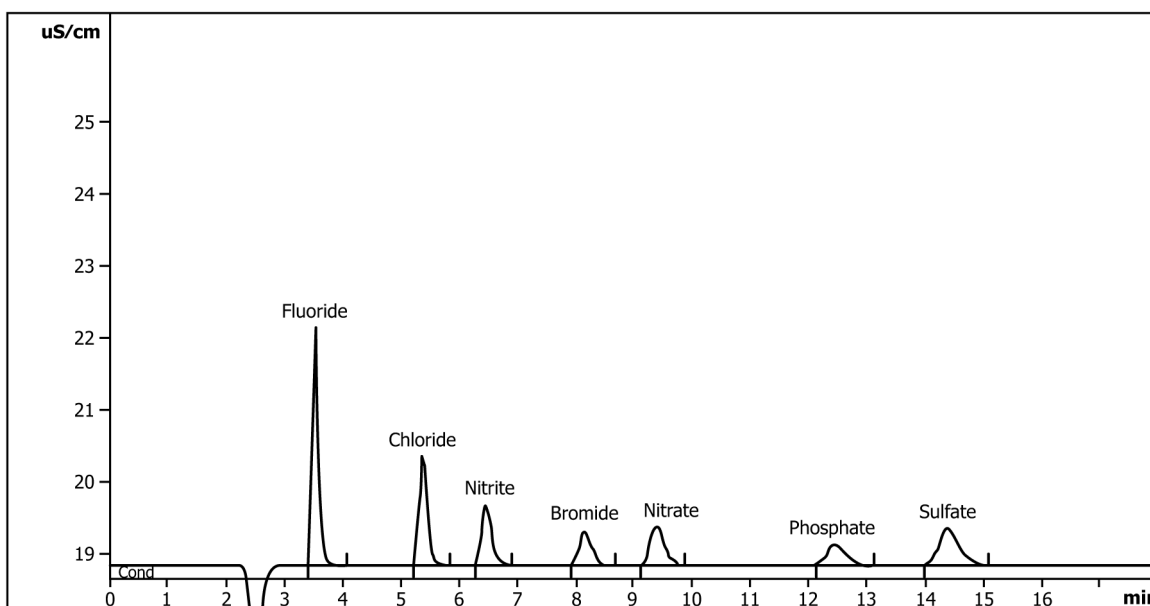


FIG. 1 Typical Ion Chromatogram of a Solution Containing 1 mg/kg of Various Anions in Water

3.2.3 *inorganic sulfate, n*—sulfate species present as sulfuric acid, ionic salts of this acid, or mixtures of these.

3.2.4 *potential sulfate, n*—total inorganic sulfate species present after the sample has been reacted with an oxidizing agent.

4. Summary of Test Method

4.1 For existent inorganic sulfate and chloride, a small volume of a sample is evaporated to dryness and reconstituted to the initial sample volume with deionized water, and injected into an ion chromatograph consisting of appropriate ion exchange columns, suppressor and a conductivity detector. For potential sulfate, a small volume of a sample is evaporated to dryness and reconstituted to the initial sample volume with 0.90 % hydrogen peroxide solution in water, and injected into an ion chromatograph. Ions are separated based on their affinity for exchange sites of the resin with respect to the resin's affinity for the eluent. The suppressor increases the sensitivity of the method by both increasing the conductivity of the analytes and decreasing the conductivity of the eluent. The suppressor converts the eluent and the analytes to the corresponding hydrogen form acids. Anions in the aqueous sample are quantified by integration of their responses compared with an external calibration curve, calculated as mg/L for each ion. The calibration standards are prepared from suitable salts dissolved in water solutions. Existent or potential inorganic sulfate and chloride concentrations may be calculated as mg/kg by measuring the density of the original sample.

4.2 Similar methods for chloride and sulfate determinations can be found in Test Method D5827 for engine coolant, and for ethanol in ISO/CEN15492, Test Method D7319 by ion chromatography with direct sample injection, and for sulfate only in Test Method D7318 by potentiometric lead titration.

5. Significance and Use

5.1 Sulfates and chlorides may be found in filter plugging deposits and fuel injector deposits. The acceptability for use of the fuel components and the finished fuels depends on the sulfate and chloride content.

5.2 Existent and potential inorganic sulfate and total chloride content, as measured by this test method, can be used as one measure of the acceptability of gasoline components for automotive spark-ignition engine fuel use.

6. Interferences

6.1 Interferences can be caused by substances with similar ion chromatographic retention times, especially if they are in high concentration compared to the analyte of interest. Sample dilution or standard addition can be used to minimize or resolve most interference problems.

6.2 A water dip (system void, negative peak as shown in Fig. 1) may cause interference with some integrators. Usually, for chloride and sulfate determinations, the water dip should not be a problem since the chloride and sulfate peaks are far enough away from the water dip.

6.3 Given the trace amounts of chloride and sulfate determined by this method, interferences can be caused by contamination of glassware, eluents, reagents, etc. Great care must be taken to ensure that contamination is kept at the lowest possible levels. The use of powder-free gloves is highly recommended to prevent sample contamination.

7. Apparatus

7.1 *Analytical Balance*, at least 2000 g capacity, capable of weighing accurately to 0.01 g.

7.1.1 *Analytical Balance*, at least 100 g capacity, capable of weighing accurately to 0.0001 g.