



Designation: D5184 – 22

Standard Test Methods for Determination of Aluminum and Silicon in Fuel Oils by Ashing, Fusion, Inductively Coupled Plasma Atomic Emission Spectrometry, and Atomic Absorption Spectrometry¹

This standard is issued under the fixed designation D5184; 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 These test methods cover the determination of aluminum and silicon in fuel oils at concentrations between 5 mg/kg and 150 mg/kg for aluminum and 10 mg/kg and 250 mg/kg for silicon.

1.2 *Test Method A*—Inductively coupled plasma atomic emission spectrometry is used in this test method to quantitatively determine aluminum and silicon.

1.3 *Test Method B*—Flame atomic absorption spectrometry is used in this test method to quantitatively determine aluminum and silicon.

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

1.5 *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.* Specific warning statements are given in Sections 7.6, 10.1, and 11.5.

1.6 *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.*

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

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2. Referenced Documents

2.1 ASTM Standards:²

- D1193 Specification for Reagent Water
- 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
- 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
- D7260 Practice for Optimization, Calibration, and Validation of Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) for Elemental Analysis of Petroleum Products and Lubricants
- D7740 Practice for Optimization, Calibration, and Validation of Atomic Absorption Spectrometry for Metal Analysis of Petroleum Products and Lubricants

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 *calibration, n*—the process by which the relationship between signal intensity and elemental concentration is determined for a specific element analysis.

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.

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

3.2.2 *check standard, n*—in calibration, an artifact measured periodically, the results of which typically are plotted on a control chart to evaluate the measurement process.

4. Summary of Test Methods

4.1 A weighed quantity of homogenized sample is heated in a clean platinum dish, the combustible material is removed by burning and the carbon finally removed by heating in a muffle furnace at a temperature of $550\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$. The residue is fused with a lithium tetraborate/lithium fluoride flux. The fused mixture is digested in a solution of tartaric acid and hydrochloric acid and diluted to volume with water. The resulting solution is aspirated into an inductively-coupled plasma and the emission intensities of aluminum and silicon lines are measured. Standard calibration solutions are also aspirated and aluminum and silicon intensities are measured for comparison. Alternatively, the resulting solution is aspirated into the flame of an atomic absorption spectrometer and the absorptions of the resonance radiation of aluminum and silicon are measured. Standard calibration solutions are also aspirated and aluminum and silicon absorption intensities are measured for comparison.

4.2 Information on proper protocols for conducting atomic absorption spectrometry can be found in Practice **D7740**.

4.3 Information on Proper protocols for conducting inductively coupled plasma-atomic absorption spectrometry can be found in Practice **D7260**.

5. Significance and Use

5.1 Catalyst fines in fuel oils can cause abnormal engine wear. These test methods provide a means of determining silicon and aluminum, the major constituents of the catalysts.

6. Apparatus

6.1 *Balance*, capable of weighing to 0.1 g, capacity of 150 g.

6.2 *Choice of Instrument*:

6.2.1 *Inductively-Coupled Plasma Atomic Emission Spectrometer*—Either a sequential or simultaneous spectrometer is suitable, if equipped with an ICP torch and RF generator to form and sustain the plasma.

6.2.2 *Atomic Absorption Spectrometer*—A suitable instrument will consist of modulated hollow cathode lamps or other sources of resonance radiation of aluminum and silicon, a nitrous oxide/acetylene burner, and a spectrometer with a suitable detection and read-out system.

6.3 *Homogenizer*, non-aerating, high-speed shear mixer to homogenize the sample.

NOTE 1—Ultrasonic bath and ultrasonic probe type homogenizers were not evaluated in the development of these test methods.

6.4 *Electric Muffle Furnace*, capable of being maintained at temperatures of $550\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$ and $925\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$. The furnace preferably having suitable apertures at front and rear to allow a slow, natural draft of air to pass through.

6.5 *Electric Hot Plate*, with or without magnetic stirring capability.

6.6 *Electric Oven*, maintained at a temperature of $50\text{ }^{\circ}\text{C}$ to $60\text{ }^{\circ}\text{C}$.

6.7 *Graduated Cylinders*, 10 mL, 25 mL, 50 mL, and 100 mL.

6.8 *Pipettes*, 1 mL, 2 mL, 5 mL, 10 mL, 20 mL, and 25 mL.

6.9 *Platinum Dish*, 100 mL capacity, cleaned with fused potassium hydrogen sulfate.

6.10 *Volumetric Flasks*, 100 mL and 1000 mL.

6.11 All glassware must be carefully cleaned with 1 + 1 hydrochloric acid and rinsed thoroughly with water to minimize contamination. The use of chromic acid cleaning solution is not recommended.

6.12 Zirconium crucible with close fitting zirconium lid, 30 mL to 50 mL capacity.

7. Reagents

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.³ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Type II of Specification **D1193**.

7.3 *Flux*—Mixture of 90 % lithium tetraborate and 10 % lithium fluoride.

NOTE 2—Lithium fluoride is necessary to prevent heavy metal corrosion of the platinum dish and to lower the fusion temperature.

7.4 *Hydrochloric acid (36 % (m/m))*—concentrated hydrochloric acid.

7.5 *Potassium Hydrogen Sulfate*, fused solid.

7.6 *2-Propanol (Isopropyl Alcohol)* (**Warning**—Flammable; can be explosive when evaporated to or near dryness.)

7.7 *Aqueous Standard Solutions*.

7.7.1 *Aluminum Standard Solutions*—Obtain a ready made, aqueous standard or prepare a standard from aluminum wire.

7.7.1.1 *Aluminum Solution (1000 mg/L)*—Aqueous, ready made commercial standard.

7.7.1.2 *Aluminum Solution (1000 mg/L)*—Cut an arbitrary length of 99.99 % minimum purity aluminum wire (2 mm diameter aluminum wire has been found satisfactory). Measure the length to the nearest 0.1 cm and weigh the aluminum wire to the nearest 0.001 g. Determine the mass/cm for the aluminum wire and cut a length of aluminum wire that is calculated

³ *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.