



Designation: D 5184 – 00

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 D 5184; 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 and 150 mg/kg for aluminum and 10 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 given in SI (metric) units are to be regarded as the 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 and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in 10.1 and 11.5.

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products³

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products³

D 6299 Practice for Applying Statistical Quality Assurance Techniques to Evaluate Analytical Measurement System Performance⁴

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials⁵

3. Terminology

3.1 Definition:

3.1.1 *emission spectroscopy*—Refer to Terminology E 135.

3.2 Definitions of Terms Specific to This Standard:

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

3.2.2 *check standard*—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 $550 \pm 25^\circ\text{C}$. 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.

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*

¹ These test methods are under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and are the direct responsibility of Subcommittee D02.03.0B on Spectrometric Methods.

Current edition approved Nov. 10, 2000. Published December 2000. Originally published as D 5184 – 91. Last previous edition D 5184 – 91 (1995).

² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 05.02.

⁴ *Annual Book of ASTM Standards*, Vol 05.04.

⁵ *Annual Book of ASTM Standards*, Vol 03.05.