



Designation: D5056 – 17

Standard Test Method for Trace Metals in Petroleum Coke by Atomic Absorption¹

This standard is issued under the fixed designation D5056; 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 the analysis for the commonly determined trace metals (aluminum, calcium, iron, nickel, silicon, sodium, and vanadium) in laboratory analysis samples of raw and calcined petroleum coke by atomic absorption spectroscopy.

1.2 The elemental concentration ranges for which this test method is applicable and the limits of detection of this test method are listed in [Table 1](#).

1.3 The values stated in SI units are to be regarded as the 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.* For warning statements, see Sections 8 – 10.

1.5 *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

[D346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis](#)

[D1193 Specification for Reagent Water](#)

[D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance](#)

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.

Current edition approved May 1, 2017. Published May 2017. Originally approved in 1990. Last previous edition approved in 2015 as D5056 – 15. DOI: 10.1520/D5056-17.

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.

[D7740 Practice for Optimization, Calibration, and Validation of Atomic Absorption Spectrometry for Metal Analysis of Petroleum Products and Lubricants](#)

3. Summary of Test Method

3.1 A representative sample of the petroleum coke is ashed at 525 °C under specified conditions. The ash is fused with lithium tetraborate ($\text{Li}_2\text{B}_4\text{O}_7$), or lithium metaborate (LiBO_3). The melt is dissolved in dilute hydrochloric acid (HCl), and the resultant solution is analyzed by atomic absorption spectroscopy for the following elements: aluminum, calcium, iron, nickel, silicon, sodium, and vanadium.

3.2 Standard practice for using atomic absorption spectrometry for determination of metals in petroleum products and lubricants can be found in Practice [D7740](#).

4. Significance and Use

4.1 The presence and concentration of various metallic elements in a petroleum coke are major factors in the suitability of the coke for various uses. This test method provides a means of measuring the amounts of those metallic elements in the coke sample.

4.2 The concentration of these elements is one factor in determining the economic value of the coke. Coke used for production of electrodes will have different specification requirements dependent on what service the electrodes will be used in. Generally the fuel cokes are highest in metallic element concentration and have the least economic value.

4.3 The test method provides a standard procedure for use by the purchaser and seller in the commercial transfer of petroleum coke to determine whether the lot of coke meets the specifications of the purchasing party.

5. Interferences

5.1 Spectral interferences can occur when using other than the recommended wavelength for analysis or when using multi-elemental hollow cathode lamps.

6. Apparatus

6.1 *Furnace*, electric, capable of regulation of temperature at 525 °C \pm 10 °C.

6.2 *Magnetic Stirring Hot Plate*.

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

TABLE 1 Applicable Concentration Ranges and Limits of Detection on a Dried Original Sample Basis

Element	Concentration Range (mg/kg)	Limit Detection (mg/kg)
Aluminum	15 to 105	5.0
Calcium	20 to 225	1.0
Iron	150 to 500	1.5
Nickel	5 to 200	1.5
Silicon	90 to 420	20
Sodium	15 to 115	0.2
Vanadium	5 to 500	2.0

6.3 *Platinum Dish*, 50 mL to 58 mL capacity.

6.4 *Platinum Dish*, 150 mL to 200 mL capacity.

6.5 *Platinum-Tipped Tong*s.

6.6 *Furnace*, electric, capable of regulation of temperature at 950 °C ± 10 °C or a Meker-type forced air burner.

6.7 *Atomic Absorption Spectrophotometer (AAS)*, equipped as follows:

6.7.1 *Background Correction*, using either a deuterium (D_2) arc background corrector or other comparable simultaneous background correction system.

6.7.2 *Burner Head*, capable of supporting a nitrous oxide-acetylene flame.

6.7.3 *Burner Head*, single or multiple-slot, capable of supporting an air-acetylene flame.

6.7.4 *Hollow Cathode Lamps*, one for each of the elements to be analyzed: aluminum, calcium, iron, nickel, silicon, sodium, and vanadium.

NOTE 1—Multi-elemental lamps can also be used; however, spectral interferences are possible (see 5.1).

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 shall 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, references to water shall be understood to mean reagent water as defined by Type II of Specification **D1193**.

7.3 *Hydrochloric Acid, Solution 1*, 20 % by volume (20 mL of concentrated HCl diluted to 100 mL with Type II reagent water).

7.4 *Lanthanum Additive, Solution 2*, 100 g/L lanthanum (dissolve 175 g $LaCl_3$ in water and dilute to 1 L with water).

7.5 *Lanthanum Chloride ($LaCl_3$) powder* (high purity).

³ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual 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.

7.6 *Lithium Tetraborate ($Li_2B_4O_7$)*, powder (high purity), or *Lithium Metaborate ($LiBO_3$)*, powder (high purity).

7.7 *Standard and Sample Dilution Additive, Solution 3*—Weigh 40.0 g, to the nearest 0.1 g, of $Li_2B_4O_7$ into a 150 mL to 200 mL platinum dish, fuse with a Meker-type burner to form a liquid, and cool. Alternatively, heat in a furnace at 950 °C ± 10 °C for 10 min or until a liquid forms. Place the cooled platinum dish containing the fused recrystallized $Li_2B_4O_7$, and a magnetic stirring bar into a 2 L beaker. Add 1000 mL of Solution 1 (20 % HCl). Heat and stir the solution on a magnetic stirring hot plate until the melt completely dissolves. After dissolution, remove the platinum dish with a glass rod. Rinse the platinum dish and glass rod with water into the lithium borate solution. Immediately transfer the warm solution quantitatively to a 2 L flask. To avoid crystallization add about 100 mL of water; stir the solution and cool to room temperature. Add 400 mL of Solution 2 (lanthanum additive) and mix. Dilute to 2000 mL with water, mix thoroughly, and vacuum filter the entire solution through Dow filter paper.

NOTE 2—Fifty millilitres of Solution 3 contains 1 g $Li_2B_4O_7$, 25 mL of Solution 1, 20 % HCl, and 10 mL of Solution 2, lanthanum additive.

7.8 *Standard Stock Solutions*—Prepare standard stock solutions from high purity (99.9 % or better) metals, oxides, or salts. Stock solutions of 1000 ppm (mg/L) for each metal are needed for preparation of dilute standards in the range from <1.0 ppm to 50 ppm (mg/L). Working standards containing aluminum, calcium, iron, nickel, silicon, sodium, and vanadium in concentration ranges below 10 ppm (mg/kg) are to be prepared daily to ensure stability.

7.9 *Quality Control (QC) Samples*, preferably are portions of one or more petroleum coke samples that are stable and representative of the samples of interest. These QC samples can be used to check the validity of the testing process as described in Section 13.

7.9.1 NIST SRM 2718A green petroleum coke or 2719 calcined petroleum coke can be used as a QC material for this analysis.⁴

NOTE 3—Commercially available standards and other reagent solutions may be used in place of laboratory preparations.

8. Sample Preparation

8.1 Crush and divide the initial sample to obtain a laboratory analysis sample. Crush to pass a No. 60 (0.250 mm) sieve by the procedure in Practice **D346**, Section 10 on Preparation of Coke Sample for Laboratory Analysis.

8.2 Crush approximately a 30 g of representative portion of the minus No. 60 sieve analysis sample to pass a No. 200 (0.075 mm) sieve. Use a tungsten carbide mill to minimize metal contamination. Dry this sample to constant weight at 110 °C to 115 °C (approximately 8 h) and store in a desiccator until cool. (Note that preparation of the minus 200 mesh analysis samples, from the minus 60 mesh analysis samples,

⁴ Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 2300, Gaithersburg, MD 20899-2300, <http://www.nist.gov>.