



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

This standard is issued under the fixed designation D 5056; 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 hazard statements, see Sections 8, 9 and 10.

2. Referenced Documents

2.1 ASTM Standards:

D 346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis²

D 1193 Specification for Reagent Water³

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.

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.

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

4.2 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 $525^\circ\text{C} \pm 10^\circ\text{C}$.

6.2 *Magnetic Stirring Hot Plate*.

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

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

6.5 *Platinum-Tipped Tongs*.

6.6 *Furnace*, electric, capable of regulation of temperature at $950 \pm 10^\circ\text{C}$ 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

¹ This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.03.0B on Spectrometric Methods.

Current edition approved Apr. 10, 1996. Published June 1996. Originally published as D 5056 – 90. Last previous edition D 5056 – 95.

² *Annual Book of ASTM Standards*, Vol 05.05.

³ *Annual Book of ASTM Standards*, Vol 11.01.