Designation: D5600 – 17

Standard Test Method for
Trace Metals in Petroleum Coke by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)[1]

This standard is issued under the fixed designation D5600; 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 commonly determined trace metals in test specimens of raw and calcined petroleum coke by inductively coupled plasma atomic emission spectroscopy.

1.2 Elements for which this test method is applicable are listed in Table 1. Detection limits, sensitivity, and optimum ranges of the metals will vary with the matrices and model of spectrometer.

1.3 This test method is applicable only to samples containing less than one mass % ash.

1.4 Elements present at concentrations above the upper limit of the working ranges can be determined with additional, appropriate dilutions.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 *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.*

1.7 *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
D7260 Practice for Optimization, Calibration, and Validation of Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) for Elemental Analysis of Petroleum Products and Lubricants
E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *gross sample*—the original, uncrushed, representative portion taken from a shipment or lot of coke.

3.1.2 *ICP-AES*—Inductively Coupled Plasma—Atomic Emission Spectrometry.

3.1.3 *petroleum coke*—a solid, carbonaceous residue produced by thermal decomposition of heavy petroleum fractions and cracked stocks.

4. Summary of Test Method

4.1 A test sample of the petroleum coke is ashed at 700 °C. The ash is fused with lithium borate. The melt is dissolved in dilute hydrochloric acid (HCl), and the resultant solution is analyzed by inductively coupled plasma atomic emission spectrometry (ICP-AES) using simultaneous, or sequential multielemental determination of elements. The solution is introduced to the ICP instrument by free aspiration or by an optional peristaltic pump. The concentrations of the trace metals are then calculated by comparing the emission intensities from the sample with the emission intensities of the standards used in calibration.

5. Significance and Use

5.1 The presence and concentration of various metallic elements in a petroleum coke are major factors in determining the suitability of the coke for various end uses. This test method provides a means of determining the concentrations of these metallic elements in a coke sample.

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

TABLE 1 Elements Determined and Suggested Wavelengths

Element	Wavelengths, nm[A],[B]	Concentration Range, mg/kg[C]
Aluminum	237.313, 256.799, 308.216, 396.152	15–110
Barium	455.403, 493.410	1–65
Calcium	317.933, 393.367, 396.847	10–140
Iron	259.940	40–700
Magnesium	279.079, 279.553	5–50
Manganese	257.610, 294.920	1–7
Nickel	231.604, 241.476, 352.454	3–220
Silicon	212.412, 251.611, 288.159	60–290
Sodium	588.995, 589.3, 589.592	30–160
Titanium	334.941, 337.280	1–7
Vanadium	292.402	2–480
Zinc	202.548, 206.200, 213.856	1–20

[A] The wavelengths listed were utilized in the round robin because of their sensitivity. Other wavelengths can be substituted if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference (see 6.1). In time, other elements may be added as more information becomes available and as required.
[B] Alternative wavelengths can be found in references such as "Inductively Coupled Plasma Atomic Emission Spectroscopy," Winge, R. K., Fassel, V. A., Peterson, V. J., and Floyd, M. A., Elsevier, 1985.
[C] Based on this round-robin study. This test method can be applicable to other elements or concentration ranges but precision data is not available.

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

6. Interferences

6.1 *Spectral*—Follow the instrument manufacturer's operating guide to develop and apply correction factors to compensate for the interferences. To apply interference corrections, all concentrations shall be within the previously established linear response range of each element.

6.2 Spectral interferences are caused by: *(1)* overlap of a spectral line from another element; *(2)* unresolved overlap of molecular band spectra; *(3)* background contribution from continuous or recombination phenomena; and *(4)* stray light from the line emission of high-concentration elements. Spectral overlap can be compensated for by computer-correcting the raw data after monitoring and measuring the interfering element. Unresolved overlap requires selection of an alternate wavelength. Background contribution and stray light can usually be compensated for by a background correction adjacent to the analyte line.

6.3 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they shall be reduced by diluting the sample, by using a peristaltic pump, or by using the standard additions method. Another problem that can occur with high dissolved solids is salts buildup at the tip of the nebulizer, which can affect aerosol flow rate and cause instrumental drift. This problem can be controlled by wetting the argon prior to nebulization, using a tip washer, or diluting the sample.

6.4 See Practice D7260 for explanation of ICP-AES interferences and other operational details.

7. Apparatus

7.1 *Balance,* top loading, with automatic tare, capable of weighing to 0.0001 g, 150 g capacity.

7.2 *Ceramic Cooling Plate,* desiccator plates have been found effective.

7.3 *Crucible Support,* nichrome wire triangles.

7.4 *Furnaces,* electric, capable of regulation of temperature at 700 °C ± 10 °C and 1000 °C ± 10 °C, with allowances for exchange of combustion gases and air.

7.5 *Inductively Coupled Plasma Atomic Emission Spectrometer*—Either sequential or simultaneous spectrometer is suitable.

7.6 *Magnetic Stirring Bars,* polytetrafluoroethylene (PTFE) coated, approximately 12 mm (½ in.) in length.

7.7 *Magnetic Stirring Hot Plate.*

7.8 *Meker-Type Forced Air Burner.*

7.9 *Nebulizer*—A high-solids nebulizer is strongly recommended. This type of nebulizer reduces the possibility of clogging and minimizes aerosol particle effects.

7.10 *Peristaltic Pump*—A peristaltic pump is strongly recommended to provide a constant flow of solution.

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

7.12 *Platinum Dish,* 100 mL to 200 mL capacity.

7.13 *Platinum-tipped Tongs.*

7.14 *Ring Stand,* with crucible support.

7.15 *Sieves,* 0.250 mm (No. 60) and 0.075 mm (No. 200), conforming to Specification E11.

7.16 *Tungsten Carbide Mill,* laboratory size.

7.17 *Vacuum Filtration Apparatus.*

7.18 *Filter Paper,* sized to fit vacuum filtration apparatus, fine porosity, slow flow rate, 2.5 micron particle retention.

8. Reagents

8.1 *Purity of Reagents*—Reagent-grade chemicals shall be used in all tests. 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.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II reagent water as defined in Specification D1193.

8.3 *Argon Gas Supply,* welding grade.

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[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.