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Carbonaceous materials for the production of aluminium — Petroleum coke — Determination of trace metals by inductively coupled plasma atomic emission spectrometry

Produits carbonés pour la production de l'aluminium — Coke de pétrole — Détermination des métaux à l'état de trace par spectrométrie d'émission atomique avec plasma induit par haute fréquence

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 14435 was prepared by Technical Committee ISO/TC 226, *Materials for the production of primary aluminium*.

Introduction

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. Users of petroleum coke require a standard procedure to determine the concentrations of these metallic elements in a coke sample. This International Standard describes such a procedure.

This International Standard is based on ASTM method D5600-98, published under the jurisdiction of ASTM Committee D2 on Petroleum Products and Lubricants and Subcommittee DO2.05.01 on Petroleum Coke Sampling and Procedures.

The repeatability and reproducibility information is based on an interlaboratory trial, which is reported in Research Report D02-1007 available from ASTM Headquarters.

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WARNING — This International 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 Scope

This International Standard applies to carbonaceous materials for the production of aluminium.

This International Standard describes a test method which covers the analysis for commonly determined trace metals in test specimens of raw and calcined petroleum coke by inductively coupled plasma atomic emission spectroscopy. It can also be applied to other heat-treated carbonaceous materials e.g. coal-tar pitch coke, anthracite.

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

This test method is applicable only to samples containing less than a mass fraction of 1 % ash.

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

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 6375, *Carbonaceous materials for the production of aluminium — Coke for electrodes — Sampling*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
petroleum coke
solid, carbonaceous residue produced by thermal decomposition of heavy petroleum fractions and cracked stocks

3.2
coal-tar pitch coke
solid, carbonaceous residue produced by decomposition of coal-tar pitch

3.3
gross sample
original, uncrushed, representative portion taken from a shipment or lot of coke

4 Principle

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 nitric acid (HNO_3), and the resultant solution is analyzed by inductively coupled plasma atomic emission spectrometry (ICP-AES) using simultaneous, or sequential, multi-elemental 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 Interferences

For spectral interferences, 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.

NOTE Spectral interferences are caused by

a) overlap of a spectral line from another element,

b) unresolved overlap of molecular band spectra,

c) background contribution from continuous or recombination phenomena, and

d) 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.

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 amounts of 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 amounts of dissolved solids is build-up of salts 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 by using a tip washer, or diluting the sample.

Table 1 — Elements determined and suggested wavelengths

Element	Wavelengths nm[a,b]	Concentration range µg/g[c]
Aluminium	237,313 256,799 308,215 396,152	15 to 110
Barium	455,403 493,410	1 to 65
Calcium	317,933 393,367 396,847	10 to 140
Iron	259,940	40 to 700
Magnesium	279,079 279,553	5 to 50
Manganese	257,610 294,920	1 to 7
Nickel	231,604 341,476 352,454	3 to 220
Silicon	212,412 251,611 288,159	60 to 290
Sodium	588,995 589,592	30 to 160
Titanium	334,941 337,280	1 to 7
Vanadium	292,402	2 to 480
Zinc	202,548 206,200 213,856	1 to 20

[a] The wavelengths listed were utilized in the interlaboratory trial because of their sensitivity. Other wavelengths can be substituted if they can provide the required sensitivity and are treated with the same corrective techniques for spectral Interference (see Clause 5). 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 interlaboratory trial. This method can be applicable to other elements or concentration ranges, but precision data are not available.

6 Apparatus

6.1 Balance, top loading, with automatic tare, capable of weighing to 0,000 1 g, of capacity 150 g.

6.2 Ceramic cooling plate, desiccator plates have been found to be effective.

6.3 Crucible support, nichrome wire triangles.

6.4 Furnaces, electric, capable of temperature regulation at 700 °C ± 10 °C and 1 000 °C ± 10 °C, with allowances for exchange of combustion gases and air.

6.5 Inductively coupled plasma atomic emission spectrometer, either a sequential or simultaneous spectrometer is suitable, equipped with a quartz ICP torch and radio frequency (RF) generator to form and sustain the plasma.

6.6 Magnetic stirring bars, polytetrafluoroethylene (PTFE) coated, approximately 12 mm in length.

6.7 Magnetic stirring hotplate.

6.8 Meker-type forced air burner.

6.9 Nebulizer, a high-solids nebulizer is recommended.

6.10 Peristaltic pump, a peristaltic pump is recommended.