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Solid mineral fuels — Determination of nine trace elements — Acid digestion and flame atomic absorption method

*Combustibles minéraux solides — Dosage de neuf éléments à l'état de
trace — Digestion par l'acide et spectrométrie d'absorption atomique
dans la flamme*

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Foreword

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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.

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ISO 8983 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 5, *Methods of analysis*.

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Solid mineral fuels — Determination of nine trace elements — Acid digestion and flame atomic absorption method

1 Scope

This International Standard covers the determination of beryllium, chromium, copper, manganese, nickel, lead, strontium, vanadium and zinc by atomic absorption spectrometry, following acid digestion of a laboratory-prepared ash.

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 687, *Solid mineral fuels — Coke — Determination of moisture in the general analysis test sample*

ISO 1171, *Solid mineral fuels — Determination of ash*

ISO 13909-4, *Hard coal and coke — Mechanical sampling — Part 4: Coal — Preparation of test samples*

ISO 13909-6, *Hard coal and coke — Mechanical sampling — Part 6: Coke — Preparation of test samples*

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

ISO 5068-2, *Brown coals and lignites — Determination of moisture content — Part 2: Indirect gravimetric method for moisture in the analysis sample*

ISO 5725-6, *Accuracy (trueness and precision) of measurement methods and results — Part 6: Use in practice of accuracy values*

ISO 11722, *Solid mineral fuels — Hard coal — Determination of moisture in the general analysis test sample by drying in nitrogen*

ISO 18283, *Hard coal and coke — Manual sampling*

3 Principle

Coal and carbonized products are ashed under controlled laboratory conditions. Mineral acids dissolve the ash. The individual elements are determined by atomic absorption spectrometry.

4 Reagents and materials

4.1 Purity of reagents

Use analytical reagent grade chemicals. Other grades can be used, provided it is documented that the reagent is of sufficiently high purity to permit its use without adversely affecting the accuracy of the determination.

4.2 Water, high purity, deionized (Type II water) as defined in ISO 3696, or equivalent purity.

4.3 Aqua regia.

Mix 1 part concentrated nitric acid (HNO_3 , sp. gr. 1,42), 3 parts concentrated hydrochloric acid (HCl , sp. gr. 1,19) and 1 part water.

SAFETY PRECAUTIONS — Polypropylene bottles are unsuitable for storage of this aqua regia solution because they have been found to become embrittled by it after a few months. Polyethylene bottles, on the other hand, have been found to take four years to become embrittled by this solution.

4.4 Boric acid solution, saturated.

Dissolve by heating 60 g of boric acid (H_3BO_3) in 1 l of de-ionized (Type II) water. Allow to cool.

4.5 Stock solutions, standard.

Prepare standard stock solutions from metals, oxides, or salts. Do not use sulfates or sulphuric acid (H_2SO_4). They have adverse effects on the flame, and some sulfates have low solubility. Prepare stock solutions of at least 100 µg/ml.

NOTE Commercially available standard stock solutions (traceable to a national certifying authority) can be used.

4.6 Hydrofluoric acid, concentrated (HF , sp. gr. 1,15).

WARNING — All acids should be handled with care but extra precautions are required with hydrofluoric acid. Even when diluted, this is an extremely dangerous acid, harmful to the eyes and skin: rubber gloves and goggles should be worn when using this acid. Any acid that touches the skin should be washed off immediately with copious quantities of water. Calcium gluconate gel should also be stored on the workbench for immediate application if hydrofluoric acid contacts skin. A physician should be consulted immediately if any acid is sprayed into the eyes or if prolonged contact with the skin occurs.

4.7 Caesium chloride buffer solution, 20 000 mg/l Cs.

Prepare solution by dissolving 25 g caesium chloride in water and adjust volume to 1 l.

5 Apparatus

5.1 Atomic absorption spectrometer, single or dual-beam instrument using a deuterium (D_2) arc background corrector, or other comparable background correction system.

5.2 Muffle furnace, as specified in ISO 1171.

5.3 Bottles, polypropylene or polycarbonate, at least 125 ml capacity, capable of withstanding 130 °C.

5.4 Volumetric flasks, polypropylene or polycarbonate, 100 ml capacity.

5.5 Steam bath or air oven.

5.6 Analytical balance, capable of weighing to 0,1 mg.

5.7 Crucibles, 50 ml capacity, made of quartz, high silica or platinum.

6 Preparation of the test sample

The sample used for the preparation of the ash shall be the general analysis test sample prepared in accordance with ISO 13909-4, ISO 13909-6 or ISO 18283.

NOTE Contact with metals such as stainless steel can result in contamination of the sample.

7 Procedure

7.1 General

A certified reference material (CRM), coal or coal derived ash, shall be analysed in duplicate with the same test batch as the samples. Variations to the procedure in Clause 7 are allowed provided the results of the analysis of the CRM are within the tolerances of the certified values quoted for the analyte trace elements and duplicate measurements of the CRM agree within the repeatability critical limit specified in Table 2.

Variations to the procedure include changes in sample mass, volumes of reagents and also include the use of different digestion equipment e.g. pressure vessels specifically designed for use in air ovens at elevated temperatures or in microwave ovens.

7.2 Preparation of ash

Weigh a clean, dry crucible (5.7). Record the mass as m_c , expressed in grams to the nearest 0,1 mg. Add sufficient test sample to produce 0,5 g to 0,75 g of ash and reweigh. Record the mass as m_{cs} , expressed in grams to the nearest 0,1 mg. Place the crucible in a cold muffle furnace, set to attain 300 °C in about 1 h, and then 500 °C over an additional 2 h. Control the heating rate to avoid mechanical loss. Continue to ash the sample until no carbonaceous material remains or for a maximum of 18 h. After cooling, weigh and record the mass as m_{ca} , expressed in grams to the nearest 0,1 mg. Grind the ash in a clean agate or mullite mortar, to pass a sieve with a 75 µm aperture, being careful to minimize loss of the ash. Transfer as much as possible of the ground ash to the quartz crucible and reheat at 500 °C for 1 h and cool.

NOTE 1 Heating rate is selected to avoid loss of sample during ashing.

NOTE 2 Stirring the sample each hour after attaining the final ashing temperature can reduce the time required to eliminate all carbonaceous residue.

Carry out a concurrent moisture determination on a representative portion of the general analysis sample (see Clause 6) in accordance with ISO 687, ISO 5068-2 or ISO 11722 as appropriate.

Record the value as M_a .

7.3 Preparation of the test solution

Clean laboratory ware in a 10 % solution of hot HCl and rinse thoroughly with deionized water prior to use.

The sample shall be ignited at 500 °C for 1 h the same day it is digested.

Weigh 0,5 g to 0,75 g to the nearest 0,1 mg of the ash sample and transfer to a plastic bottle. Record the mass in grams of ash sample taken as m_{ash} . Add 3 ml of aqua regia and 5 ml of hydrofluoric acid to the sample. Tighten the screw cap and place the bottle on a steam bath. Alternatively, bottles can be placed in a larger container in an air oven at 105 °C. Heat the sample for at least 2 h. If a residue remains, the mixture shall be reheated for a minimum of 1 h.

NOTE If the residue persists after additional heating incomplete extraction of some or all of the trace elements can occur.