SLOVENSKI STANDARD
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Trda mineralna goriva ï Doloļ evanje celotnega kadmija v premogu (opomba: popravljeno in ponovno natisnjeno junija 2004)

Solid mineral fuels -- Determination of total cadmium content of coal

Combustibles minéraux solides -- Dosage du cadmium total dans le charbon

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Solid mineral fuels — Determination of total cadmium content of coal

Combustibles minéraux solides — Dosage du cadmium total dans le charbon

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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.

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 15238 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 5, *Methods of analysis*.

In this corrected version of ISO 15238:2003, Table 1 has been amended by the replacement of the published value of 0, $6\omega_{Cd} - 1$ for the Repeatability limit with the correct value of 0,$16\omega_{Cd} - 1$.

Introduction

Cadmium occurs naturally in coal. It is an element that can potentially be released during the combustion process.

Quantitative recovery can be achieved by strict adherence to the procedure set out in this International Standard.

The method may also be suitable for other elements, including lead and arsenic, although the choice of matrix modifier may vary.

Solid mineral fuels — Determination of total cadmium content of coal

1 Scope

This International Standard specifies a procedure for the determination of the total cadmium content of coal.

This procedure has not been validated with coals that spontaneously ignite. Prior to use with such sample types, users should validate the method.

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 331, *Coal — Determination of moisture in the analysis sample — Direct gravimetric method*

ISO 1170, *Coal and coke — Calculation of analyses to different bases*

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

ISO 5068, *Brown coals and lignites — Determination of moisture content — Indirect gravimetric method*

ISO 5069-2, *Brown coals and lignites — Principles of sampling — Part 2: Sample preparation for determination of moisture content and for general analysis*

ISO 13909-2, *Hard coal and coke — Mechanical sampling — Part 2: Coal — Sampling from moving streams*

3 Principle

The coal sample is ashed, followed by dissolution of the ash in a mixture of hydrochloric, nitric and hydrofluoric acids. The cadmium species present are quantified by graphite furnace atomic absorption spectroscopy.

4 Reagents

WARNING — Care should be exercised when handling the reagents, many of which are toxic and corrosive.

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade.

4.1 Water, conforming to the requirements of Grade 1 of ISO 3696.

4.2 Aqua regia solution. Mix 1 part by volume of concentrated nitric acid solution (relative density 1,42), 3 parts of concentrated hydrochloric acid solution (relative density 1,19), and 1 part water.

4.3 Concentrated hydrofluoric acid solution (relative density 1,15).

WARNING — Hydrofluoric acid is an extremely aggressive chemical which shall be handled with care.

4.4 Boric acid solution, saturated. Dissolve 60 g of boric acid in 1 litre of hot water, cool and allow to stand for three days before decanting the clear solution.

4.5 Cadmium standard stock solution, 10 µg/ml. Prepare the stock solution from a high purity metal oxide or salt, having a purity greater than 99,9 %. Dilute to volume with 1 % (*V/V*) nitric acid solution.

NOTE The cadmium standard stock solution may also be prepared from commercially available certified cadmium solution.

4.6 Cadmium standard solution, 0,1 µg/ml. Dilute 10,0 ml of cadmium standard stock solution (4.5) to 1,0 litre with 1 % (*V/V*) nitric acid solution.

5 Apparatus

5.1 Balance, capable of weighing to the nearest 0,1 mg.

5.2 Silica or platinum combustion crucible (45 × 35 × 14 mm).

5.3 Muffle furnace, with a temperature control.

5.4 Plastics bottles, made of high density polyethylene (HDPE) or fluorinated ethylene propylene (FEP), of 125 ml capacity, with screw-cap lids, capable of withstanding 130 °C when sealed and containing liquid.

5.5 Volumetric flasks, made of high density polyethylene (HDPE) or fluorinated ethylene propylene (FEP), of 100 ml capacity.

5.6 Boiling-water bath.

5.7 Graphite-furnace atomic absorption spectrometer, with background correction.

6 Preparation of sample

The test sample is the general analysis test sample prepared in accordance with ISO 5069-2 or ISO 13909-2 as appropriate. Ensure that the moisture content of the sample is in equilibrium with the laboratory atmosphere, exposing it if necessary, in a thin layer for the minimum time required to achieve equilibrium.

Before commencing the determination, mix the equilibrated sample for at least 1 min, preferably by mechanical means.

If the results are to be calculated other than on the “air-dried” basis (see Clause 8), then, after weighing the test portion, determine the moisture content of a further portion of the test sample by the method described in ISO 331 or ISO 5068 as appropriate.

7 Procedure

7.1 Ashing of coal sample

Weigh, to the nearest 0,1 mg, the amount of sample necessary to obtain a minimum of 0,2 g ash in one or more open 50 ml silica combustion crucibles (5.2). The coal layer in the crucible shall not exceed a thickness of 0,15 g/cm^2. Place the crucibles in a cold muffle furnace (5.3), set to attain (450 ± 10) °C in about 1 h, and

maintain at that temperature for a further 2 h. After cooling and weighing, calculate the percentage of ash in the coal. Carefully mix the ash in the crucibles before transferring it to a sample bottle.

NOTE The ashing time of 3 h is a minimum. Ashing overnight (18 h) is allowed.

7.2 Preparation of test solution

Place approximately 0,2 g (± 0,1 mg) of ash in a 125 ml plastics bottle (5.4). Add 3 ml of aqua regia (4.2), 5 ml of concentrated hydrofluoric acid solution (4.3), and tighten the screw cap. Place the bottle on a boiling water bath for 2 h. Remove the bottle and allow it to cool to ambient temperature, cautiously open, then add 50 ml of saturated boric acid solution (4.4). Warm again for approximately 1 h; if a residue remains at this stage it may be ignored. Remove the bottle from the water bath and cool again to ambient temperature before transferring the solution quantitatively to a 100 ml plastics volumetric flask, and diluting to 100 ml with water.

Care should be exercised when adding the acid mixture to the ash since the 450 °C ashing temperature may not have decomposed all carbonates present in the coal.

NOTE 1 If desired, it is permitted to prepare the solutions gravimetrically, adjusting the calculations in the appropriate manner.

NOTE 2 As an alternative method for the dissolution of the prepared ash, microwave dissolution or acid dissolution in a pressurized vessel can be used, if quantitative recovery of cadmium can be demonstrated.

7.3 Preparation of blank solution

Prepare a blank solution by following exactly the dissolution procedure described above (7.2) but omitting the coal (ash) sample.

7.4 Graphite-furnace atomic absorption analysis

7.4.1 Calibration

Prepare standards by diluting aliquot portions of the cadmium standard stock solution (4.5), 3 ml of aqua regia solution (4.2), 5 ml of concentrated hydrofluoric acid solution (4.3) and 50 ml of saturated boric acid solution (4.4) to 100 ml with water.

Standards containing 0,0, 1,0, 2,5 and 5,0 µg/l have been found suitable. The standards used should be verified for each instrument.

7.4.2 Measurement of standards

Measure the absorbance of each standard solution using the graphite-furnace atomic absorption spectrometer (5.7). Plot the corresponding absorbance response for each matrix-matched standard against concentration, producing a calibration curve for the instrument. Sample responses are then compared directly with the calibration.

The performance of the instrument shall be verified by the analysis of certified reference materials.

NOTE 1 No additional matrix modifier is used for cadmium determination and the instrument parameters allow for this by using a low char temperature.

NOTE 2 An alternative quantification procedure is the use of standard analyte additions to the final solution.

7.4.3 Measurement of test solution

Repeat the procedure described in 7.4.2 for the test solution (7.2). Determine the cadmium concentration in the test solution using the calibration graph (7.4.2).