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Coal — Determination of total cadmium

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 27, *Coal and coke*, Subcommittee SC 5, *Methods of analysis*.

This third edition cancels and replaces the second edition (ISO 15238:2016), which has been technically revised.

The main changes are as follows:

— the normative references have been updated;

— Formulae (1) and (2) have been modified.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Coal — Determination of total cadmium

1 Scope

This document specifies a procedure for the determination of the total cadmium mass fraction of coal.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 3696, *Water for analytical laboratory use — Specification and test methods*

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

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

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

ISO 18283, *Coal and coke — Manual sampling*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 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.

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

5 Reagents

WARNING — Care shall 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.

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

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

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

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

5.4 Boric acid solution, saturated. Dissolve 60 g of boric acid in 1 l of hot water, cool and allow to stand for 3 d before decanting the clear solution.

5.5 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 % volume fraction nitric acid solution.

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

5.6 Cadmium standard solution, 0,1 µg/ml. Dilute 10,0 ml of cadmium standard stock solution (5.5) to 1,0 l with 1 % volume fraction nitric acid solution.

6 Apparatus

6.1 Balance, capable of determining mass by weighing to the nearest 0,1 mg.

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

6.3 Muffle furnace, with a temperature control.

6.4 Plastic 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.

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

6.6 Water bath.

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

7 Preparation of sample

The test sample shall be prepared in accordance with ISO 13909-4 or ISO 18283, as appropriate. Ensure that the moisture 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 in other ways than on the "air-dried" basis (see Clause 9), then, after weighing the test portion, determine the moisture of a further portion of the test sample in accordance with the method described in ISO 5068-2 or ISO 11722, as appropriate.