INTERNATIONAL STANDARD

ISO 15238

Second edition
2016-11-15

Solid mineral fuels — Determination of total cadmium content of coal

Combustibles minéraux solides — Dosage du cadmium total dans le charbon

Reference number
ISO 15238:2016(E)

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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.

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 documents 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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

The committee responsible for this document is ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 5, *Methods of analysis*.

This second edition cancels and replaces the first edition (ISO 15238:2003), of which it constitutes a minor revision. This document incorporates changes related to dated references and other minor items following its systematic review.

Solid mineral fuels — Determination of total cadmium content of coal

1 Scope

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

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

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

3 Terms and definitions

No terms and definitions are listed in this document.

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

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

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

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.

5 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.

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

5.2 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.

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 % (*V/V*) 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 % (*V/V*) nitric acid solution.

6 Apparatus

6.1 Balance, capable of 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 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.

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

6.6 Boiling-water bath.

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

7 Preparation of sample

The test sample is the general analysis test sample prepared in accordance with ISO 5069-2 or ISO 13909-4 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 9), then, after weighing the test portion, determine the moisture content of a further portion of the test sample by the method described in ISO 5068-2 as appropriate.

