

BS ISO 15238:2016



BSI Standards Publication

Solid mineral fuels — Determination of total cadmium content of coal

National foreword

This British Standard is the UK implementation of ISO 15238:2016. It supersedes BS ISO 15238:2003 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PTI/16, Solid mineral fuels.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

© The British Standards Institution 2016.
Published by BSI Standards Limited 2016

ISBN 978 0 580 92523 8

ICS 73.040

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 November 2016.

Amendments/Corrigenda issued since publication

Date	Text affected
------	---------------

INTERNATIONAL
STANDARD

BS ISO 15238:2016

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)

© ISO 2016



COPYRIGHT PROTECTED DOCUMENT

© ISO 2016, Published in Switzerland

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

	Page
Foreword	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Principle	1
5 Reagents	1
6 Apparatus	2
7 Preparation of sample	2
8 Procedure	3
8.1 Ashing of coal sample.....	3
8.2 Preparation of test solution.....	3
8.3 Preparation of blank solution.....	3
8.4 Graphite-furnace atomic absorption analysis.....	3
8.4.1 Calibration.....	3
8.4.2 Measurement of standards.....	3
8.4.3 Measurement of test solution.....	4
8.4.4 Measurement of blank solution.....	4
9 Expression of results	4
9.1 Ash content.....	4
9.2 Cadmium content.....	4
10 Precision	5
10.1 Repeatability limit.....	5
10.2 Reproducibility critical difference.....	5
11 Test report	5

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.

8 Procedure

8.1 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 (6.2). The coal layer in the crucible shall not exceed a thickness of 0,15 g/cm². Place the crucibles in a cold muffle furnace (6.3), 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.

8.2 Preparation of test solution

Place approximately 0,2 g (± 0,1 mg) of ash in a 125 ml plastics bottle (6.4). Add 3 ml of aqua regia (5.2), 5 ml of concentrated hydrofluoric acid solution (5.3), 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 (5.4). 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.

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

NOTE 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.

8.3 Preparation of blank solution

Prepare a blank solution by following exactly the dissolution procedure described above (see 8.2) but omitting the coal (ash) sample.

8.4 Graphite-furnace atomic absorption analysis

8.4.1 Calibration

Prepare standards by diluting aliquot portions of the cadmium standard solution (5.6), 3 ml of aqua regia solution (5.2), 5 ml of concentrated hydrofluoric acid solution (5.3) and 50 ml of saturated boric acid solution (5.4) to 100 ml with water.

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

8.4.2 Measurement of standards

Measure the absorbance of each standard solution using the graphite-furnace atomic absorption spectrometer (6.7). 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.

8.4.3 Measurement of test solution

Repeat the procedure described in 8.4.2 for the test solution (see 8.2). Determine the cadmium concentration in the test solution using the calibration graph (see 8.4.2).

8.4.4 Measurement of blank solution

Repeat the procedure described in 8.4.2 for the blank (see 8.3). Determine the cadmium concentration in the blank solution using the calibration graph (see 8.4.2).

9 Expression of results

9.1 Ash content

Calculate the ash content, ω_A , as a percentage by mass of the coal sample, using Formula (1):

$$\omega_A = \frac{(m_c - m_a)}{(m_b - m_a)} \times 100 \quad (1)$$

where

m_a is the mass of the ashing combustion crucible;

m_b is the mass of the ashing combustion crucible plus coal before ashing;

m_c is the mass of the ashing combustion crucible plus coal after ashing.

9.2 Cadmium content

Calculate the cadmium content, ω_{Cd} , in ng/g, in the coal test sample using Formula (2):

$$\omega_{Cd} = \omega_A \frac{(\rho_t - \rho_b)}{1\,000\,m} \times 1\,000 \quad (2)$$

where

ω_A is the ash content of the sample as analysed, in percentage by mass;

ρ_t is the cadmium concentration of the test solution, in $\mu\text{g/l}$;

ρ_b is the cadmium concentration of the blank solution, in $\mu\text{g/l}$;

m is the mass of the sample as analysed, in grams.

Report the result as the mean of duplicate determinations to the nearest 20 ng/g on the “as analysed” basis.

For calculation of the results to bases other than “as analysed”, see ISO 1170.

10 Precision

10.1 Repeatability limit

The means of the results of duplicate determinations, carried out at different times, in the same laboratory, by the same operator, with the same apparatus, on representative portions taken from the same general analysis sample, should not differ by more than the value given in [Table 1](#).

10.2 Reproducibility critical difference

The results of duplicate determinations, carried out in each of two laboratories, on representative portions taken from the same analysis sample after the last stage of sample preparation, should not differ by more than the value given in [Table 1](#).

Table 1

	Maximum acceptable differences between results obtained (calculated to the same moisture content)	
	Repeatability limit	Reproducibility critical difference
Cadmium content (ng/g)	$0,16\omega_{Cd} - 1$	$0,22\omega_{Cd} + 3$

11 Test report

The test report shall include the following information:

- a) identification of the sample tested;
- b) the method used by reference to this document, i.e. ISO 15238;
- c) the date of the determination;
- d) the results and the method of expression used.

British Standards Institution (BSI)

BSI is the national body responsible for preparing British Standards and other standards-related publications, information and services.

BSI is incorporated by Royal Charter. British Standards and other standardization products are published by BSI Standards Limited.

About us

We bring together business, industry, government, consumers, innovators and others to shape their combined experience and expertise into standards-based solutions.

The knowledge embodied in our standards has been carefully assembled in a dependable format and refined through our open consultation process. Organizations of all sizes and across all sectors choose standards to help them achieve their goals.

Information on standards

We can provide you with the knowledge that your organization needs to succeed. Find out more about British Standards by visiting our website at bsigroup.com/standards or contacting our Customer Services team or Knowledge Centre.

Buying standards

You can buy and download PDF versions of BSI publications, including British and adopted European and international standards, through our website at bsigroup.com/shop, where hard copies can also be purchased.

If you need international and foreign standards from other Standards Development Organizations, hard copies can be ordered from our Customer Services team.

Copyright in BSI publications

All the content in BSI publications, including British Standards, is the property of and copyrighted by BSI or some person or entity that owns copyright in the information used (such as the international standardization bodies) and has formally licensed such information to BSI for commercial publication and use.

Save for the provisions below, you may not transfer, share or disseminate any portion of the standard to any other person. You may not adapt, distribute, commercially exploit, or publicly display the standard or any portion thereof in any manner whatsoever without BSI's prior written consent.

Storing and using standards

Standards purchased in soft copy format:

- A British Standard purchased in soft copy format is licensed to a sole named user for personal or internal company use only.
- The standard may be stored on more than 1 device provided that it is accessible by the sole named user only and that only 1 copy is accessed at any one time.
- A single paper copy may be printed for personal or internal company use only.

Standards purchased in hard copy format:

- A British Standard purchased in hard copy format is for personal or internal company use only.
- It may not be further reproduced – in any format – to create an additional copy. This includes scanning of the document.

If you need more than 1 copy of the document, or if you wish to share the document on an internal network, you can save money by choosing a subscription product (see 'Subscriptions').

Reproducing extracts

For permission to reproduce content from BSI publications contact the BSI Copyright & Licensing team.

Subscriptions

Our range of subscription services are designed to make using standards easier for you. For further information on our subscription products go to bsigroup.com/subscriptions.

With **British Standards Online (BSOL)** you'll have instant access to over 55,000 British and adopted European and international standards from your desktop. It's available 24/7 and is refreshed daily so you'll always be up to date.

You can keep in touch with standards developments and receive substantial discounts on the purchase price of standards, both in single copy and subscription format, by becoming a **BSI Subscribing Member**.

PLUS is an updating service exclusive to BSI Subscribing Members. You will automatically receive the latest hard copy of your standards when they're revised or replaced.

To find out more about becoming a BSI Subscribing Member and the benefits of membership, please visit bsigroup.com/shop.

With a **Multi-User Network Licence (MUNL)** you are able to host standards publications on your intranet. Licences can cover as few or as many users as you wish. With updates supplied as soon as they're available, you can be sure your documentation is current. For further information, email subscriptions@bsigroup.com.

Revisions

Our British Standards and other publications are updated by amendment or revision.

We continually improve the quality of our products and services to benefit your business. If you find an inaccuracy or ambiguity within a British Standard or other BSI publication please inform the Knowledge Centre.

Useful Contacts

Customer Services

Tel: +44 345 086 9001

Email (orders): orders@bsigroup.com

Email (enquiries): cservices@bsigroup.com

Subscriptions

Tel: +44 345 086 9001

Email: subscriptions@bsigroup.com

Knowledge Centre

Tel: +44 20 8996 7004

Email: knowledgecentre@bsigroup.com

Copyright & Licensing

Tel: +44 20 8996 7070

Email: copyright@bsigroup.com

BSI Group Headquarters

389 Chiswick High Road London W4 4AL UK