INTERNATIONAL STANDARD

ISO 13605

First edition
2018-10

Solid mineral fuels — Major and minor elements in coal ash and coke ash — Wavelength dispersive x-ray fluorescence spectrometric method

Combustibles minéraux solides — Éléments en minorité et en majorité dans les cendres de houille et de coke — Méthode spectrométrique par fluorescence aux rayons X à une longueur d'onde dispersive

Reference number
ISO 13605:2018(E)

© ISO 2018



COPYRIGHT PROTECTED DOCUMENT

© ISO 2018

All rights reserved. Unless otherwise specified, or required in the context of its implementation, 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
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

© ISO 2018 – All rights reserved

Contents

Page

Foreword ... **iv**

1 Scope ... **1**

2 Normative references ... **1**

3 Terms and definitions ... **2**

4 Principle ... **2**

5 Reagents ... **2**
5.1 General ... 2
5.2 Flux and heavy absorber ... 2

6 Apparatus ... **2**

7 Sample ... **3**
7.1 Coal and coke ash prepared in the laboratory ... 3
7.2 Coal and coke ash ... 4

8 Procedure ... **4**
8.1 Number of determinations ... 4
8.2 Test portion ... 4
8.3 Check test ... 4
8.4 Calibration ... 4
8.5 Preparation of the fused discs ... 4
8.5.1 General ... 4
8.5.2 Disc storage ... 5
8.5.3 Fused disc quality ... 5
8.6 XRF measurement ... 5
8.6.1 Instrumental conditions ... 5
8.6.2 Monitor measurements ... 6

9 Calculation ... **7**
9.1 General ... 7
9.2 Correction of instrumental drift ... 7
9.3 Correction for dead time losses ... 7
9.4 Matrix corrections ... 7

10 Reporting results ... **8**

11 Precision ... **8**

12 Test report ... **9**

Annex A (normative) **Acceptance of analytical results for laboratory method** ... **10**

Annex B (informative) **Reagents suitable for the preparation of synthetic calibration standards** ... **12**

Annex C (normative) **Standard deviation of glass disc preparation** ... **13**

Annex D (informative) **Calculation of phosphorus using P_2O_5** ... **15**

Bibliography ... **16**

© ISO 2018 – All rights reserved

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 of the voluntary nature of standards, 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 www.iso.org/iso/foreword.html.

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

This first edition of ISO 13605 cancels and replaces ISO/TS 13605:2012, which has been technically revised.

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.

© ISO 2018 – All rights reserved

Solid mineral fuels — Major and minor elements in coal ash and coke ash — Wavelength dispersive x-ray fluorescence spectrometric method

WARNING — Use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This document sets out a wavelength dispersive X-ray fluorescence (XRF) procedure for the determination of silicon, aluminium, iron, calcium, magnesium, sodium, potassium, titanium, manganese, phosphorus and sulfur.

The method is applicable to coal ashes, coke ashes and boiler ashes having components within the concentration ranges specified in Table 1.

Table 1 — Ranges of application of the method

Oxide	Concentration range %
SiO_2	5 to 100
Al_2O_3	5 to 80
Fe_2O_3	0,1 to 25
CaO	0,05 to 25
MgO	0,05 to 25
Na_2O	0,05 to 5
K_2O	0,05 to 5
TiO_2	0,05 to 5
Mn_3O_4	0,005 to 5
P_2O_5[a]	0,01 to 5
SO_3	0,05 to 10

NOTE 1 Additional analytes can be included in the method, provided that appropriate validation using reference materials is carried out.

NOTE 2 The precision statistics can be determined using suitable reference materials.

NOTE 3 The method described in this document has been tested for the following additional analytes: BaO (0,01 % to 1 %); SrO (0,01 % to 1 %) and ZnO (0,005 % to 1 %).

[a] For information relating to phosphorus calculations, refer to Annex D.

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 1171, *Solid mineral fuels — Determination of ash*

ISO 1213-2, *Solid mineral fuels — Vocabulary — Part 2: Terms relating to sampling, testing and analysis*

© ISO 2018 – All rights reserved

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 18283, *Hard coal and coke — Manual sampling*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1213-2 apply.

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

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

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

4 Principle

The specimen on which X-ray fluorescence measurements are made is prepared by incorporating the test portion of the sample, via fusion, into a borate glass disc using a casting or press-quenching procedure. By using such a specimen, particle size effects are eliminated.

Calibration is carried out using pure chemicals and/or reference standards, and by making matrix corrections for inter-element effects.

It is expected that laboratories using this document have experience in analysing coal ashes of certain compositions and that they employ calibrations that cover these compositions.

5 Reagents

5.1 General

Unless otherwise specified, all reagents shall be of analytical grade and only distilled water or water of equivalent purity shall be used.

5.2 Flux and heavy absorber

Borate fluxes of differing compositions have been found suitable for preparing glass discs from coal/coke ash samples. These fluxes are based on mixtures of one or more of the following: lithium tetraborate, lithium metaborate and lanthanum oxide. Flux may be purchased commercially or prepared by fusion of a mixture of individual reagents.

NOTE The levels of contamination in the flux should be checked. Because levels of contamination may vary from batch to batch, the same batch of flux should be used for all specimens (synthetic standards, external standards and unknowns). When using a fresh batch of flux, reference material specimens should be prepared to determine whether adjustments to the calibration are required.

6 Apparatus

All apparatus shall be constructed from materials that are thermally stable and chemically inert under the conditions of the procedure.

© ISO 2018 – All rights reserved