



BSI Standards Publication

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

National foreword

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TECHNICAL SPECIFICATION

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13605**

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Solid mineral fuels — Major and minor elements in hard 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*



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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.

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.

In other circumstances, particularly when there is an urgent market requirement for such documents, a technical committee may decide to publish other types of document:

- an ISO Publicly Available Specification (ISO/PAS) represents an agreement between technical experts in an ISO working group and is accepted for publication if it is approved by more than 50 % of the members of the parent committee casting a vote;
- an ISO Technical Specification (ISO/TS) represents an agreement between the members of a technical committee and is accepted for publication if it is approved by 2/3 of the members of the committee casting a vote.

An ISO/PAS or ISO/TS is reviewed after three years in order to decide whether it will be confirmed for a further three years, revised to become an International Standard, or withdrawn. If the ISO/PAS or ISO/TS is confirmed, it is reviewed again after a further three years, at which time it must either be transformed into an International Standard or be withdrawn.

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/TS 13605 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 5, *Methods of analysis*.

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

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

1 Scope

This Technical Specification 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	5–100
Al ₂ O ₃	5–80
Fe ₂ O ₃	0,1–25
CaO	0,05–25
MgO	0,05–25
Na ₂ O	0,05–5
K ₂ O	0,05–5
TiO ₂	0,05–5
Mn ₃ O ₄	0,005–5
P ₂ O ₅	0,01–5
SO ₃	0,05–10
NOTE 1 Additional analytes may 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 Technical Specification has been tested for the following additional analytes: BaO (0,01 % to 1 %); SrO (0,01 % to 1 %) and ZnO (0,005 % to 1 %).	

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 1171, *Solid mineral fuels — Determination of ash*

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

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*

3 Terms and definitions

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

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 Technical Specification 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

6.1 General

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