SLOVENSKI STANDARD
SIST EN ISO 12677:2011

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Nadomešča:
SIST EN ISO 12677:2003

Kemijska analiza ognjevzdržnih izdelkov z XRF - Metoda z vlito talino (ISO 12677:2011)

Chemical analysis of refractory products by X-ray fluorescence (XRF) - Fused cast-bead method (ISO 12677:2011)

Chemische Analyse von feuerfesten Erzeugnissen durch RFA - Schmelzaufschluss-Verfahren (ISO 12677:2011)

Analyse chimique des matériaux réfractaires par fluorescence de rayons X - Méthode de la perle fondue (ISO 12677:2011)

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Chemical analysis of refractory products by X-ray fluorescence (XRF) - Fused cast-bead method (ISO 12677:2011)

Analyse chimique des matériaux réfractaires par fluorescence de rayons X - Méthode de la perle fondue (ISO 12677:2011)

Chemische Analyse von feuerfesten Erzeugnissen durch Röntgenfluoreszenz-Analyse (RFA) - Schmelzaufschluss-Verfahren (ISO 12677:2011)

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Foreword

This document (EN ISO 12677:2011) has been prepared by Technical Committee ISO/TC 33 "Refractories" in collaboration with Technical Committee CEN/TC 187 “Refractory products and materials” the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by April 2012, and conflicting national standards shall be withdrawn at the latest by April 2012.

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This document supersedes EN ISO 12677:2003.

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Endorsement notice

The text of ISO 12677:2011 has been approved by CEN as a EN ISO 12677:2011 without any modification.

INTERNATIONAL STANDARD

ISO 12677

Second edition
2011-10-01

Chemical analysis of refractory products by X-ray fluorescence (XRF) — Fused cast-bead method

Analyse chimique des matériaux réfractaires par fluorescence de rayons X — Méthode de la perle fondue

Reference number
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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.

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 12677 was prepared by Technical Committee ISO/TC 33, *Refractories*.

This second edition cancels and replaces the first edition (ISO 12677:2003), which has been technically revised. Although the method in this International Standard has been considerably modified editorially and in layout, the technical changes are limited. Some minor corrections have been made to certain equations. The only significant changes are a reference to a further International Standard method (being prepared) for the preparation of reduced materials for analysis by this standard, and instructions on how to add other constituents to calibrations at the end of 10.2.1, *Purity and preparation of reagents*.

Chemical analysis of refractory products by X-ray fluorescence (XRF) — Fused cast-bead method

1 Scope

This International Standard specifies a method for the chemical analysis of refractory and technical ceramic raw materials, intermediates and products, by means of the X-ray fluorescence (XRF) fused cast-bead method. Typical materials that can be analysed by this standard are given in Clause 3. This International Standard is not applicable to non-oxide materials, such as silicon carbides or nitrides, etc. The method is applicable to a wide range of materials containing a wide range of elements.

NOTE 1 The presence of significant amounts of certain elements, such as tin, copper, zinc and chromium, can present difficulties in the fusion process. In this case, the Bibliography can be referred to.

NOTE 2 Constituents at concentrations greater than 99 % (on a dried basis) are reported by difference, provided that all likely minor constituents and any loss on ignition have been determined. These figures can also be checked by direct determination.

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 Guide 35:2006, *Reference materials — General and statistical principles for certification*

ISO 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

ISO 26845, *Chemical analysis of refractories — General requirements for wet chemical analysis, atomic absorption spectrometry (AAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES) methods*

3 Types of material

Listed below are various types of ceramic material that have been successfully analysed by this method and for which statistical data is available (see Annex I). The list is not exhaustive but serves as a guide to those using this International Standard for the first time.

a) High alumina > 45 % Al_2O_3

b) Alumino-silicate 7 % to 45 % Al_2O_3

c) Silica > 93 % SiO_2

d) Zircon

e) Zirconia and zirconates

f) Magnesia

g) Magnesia/alumina spinel (~70/30)

h) Dolomite

i) Limestone

j) Magnesia/chromic oxide

k) Chrome ore

l) Chrome-alumina

m) Alumina/magnesia spinel (~70/30)

n) Zirconia-alumina-silica cast material (AZS)

o) Calcium silicates

p) Calcium aluminates

q) Magnesium silicates

A list of elemental ranges and required detection limits are given in Annex A.

NOTE 1 Some of the above material types can be accommodated for common calibrations (see 10.3.4).

NOTE 2 Reduced materials, such as silicon carbide, cannot be determined directly by this International Standard and so are not listed above. Such materials require special methods both for loss on ignition and fusion into a bead prior to XRF analysis. Suitable procedures are described in ISO 21068-1, ISO 21068-2 and ISO 21068-3 and further methods are under development by the refractory standards system. Once reduced materials are suitably ignited and subsequently prepared as fused beads, this standard can be applied to the rest of the procedure.

WARNING — Failure to pretreat reduced materials, such as silicon carbide, properly not only leads to erroneous results but will also cause damage to valuable platinum alloy crucibles and dishes.

4 Principle

The powdered sample is fused with a suitable flux to destroy its mineralogical and particulate composition. The resultant melt is cast into the shape of a glass bead which is then introduced into an XRF spectrometer. The intensities of the fluorescent X-rays of the required elements in the bead are measured and the chemical composition of the sample is analysed by reference to previously determined calibration graphs or equations and applying corrections for inter-element effects. The calibration equations and inter-element corrections are established from beads produced using pure reagents and/or series reference materials (SeRMs), prepared in the same way as the samples. Certified reference materials (CRMs) may be used providing they meet all the requirements of 10.2.2 and 10.4.1.

Because of the universality of the fused cast-bead technique, various fluxes and modes of calibration are permitted, providing they have been demonstrated as being able to meet certain criteria of repeatability, sensitivity and accuracy. Provided that a laboratory's own methods conform to all the various criteria set down, they will be accepted as conforming to this International Standard.

5 Apparatus

5.1 Fusion vessels, of a non-wetted platinum alloy (Pt/Au 95 %/5 % is suitable). Lids, if used, shall be of a platinum alloy (not necessarily non-wetted).

NOTE A useful guide to the care of platinum is given in Reference [5] of the Bibliography.

5.2 Casting moulds, of a non-wetted platinum alloy (Pt/Au 95 %/5 % is suitable).

NOTE Vessels that serve both as fusion vessels and casting moulds can be used.

5.3 Heat reservoir for casting mould (optional), required in special circumstances when using moulds of small sizes, so that the mould does not cool too rapidly when removed from the furnace. A small piece of flat refractory material is suitable, e.g. a piece of sillimanite batt with dimensions 10 mm × 50 mm × 50 mm.

5.4 Air jet (optional), required to cool the mould rapidly. This may be any device whereon a narrow jet of air can be directed to the centre of the base of the casting dish. A convenient way of doing this is to use the base of a Bunsen burner without a barrel to serve as an air jet.

NOTE In most cases, it is very important to cool the melt rapidly. This is necessary to obtain a homogeneous bead and to free the melt from the dish.

A water-cooled metal plate may also be used.

5.5 Fusion apparatus, electric resistance furnaces or high-frequency induction furnaces that may be heated up to a fixed temperature of between 1 050 °C and 1 250 °C may be used.

5.6 Automatic fusion apparatus, for use in automatic bead preparation (see 9.2) where required.

5.7 Balance, capable of weighing to ±0,1 mg.

5.8 Mechanical mixer, that moves in a linear or rotary way.

NOTE Vibratory mixers cannot be used as they induce segregation.

6 Sample grinding

This International Standard shall start with a laboratory sample.

NOTE 1 Bulk sampling is not within the scope of this method but can be found in ISO 26845.

The sample shall be ground using tungsten carbide. The appropriate corrections for tungsten carbide (and its binder if necessary) shall be applied to loss on ignition and analysis figures in accordance with Annex B.

NOTE 2 It is permissible to apply the sample grinding methods cited in conventional chemical methods for the classes of materials covered. However, the tungsten carbide method is the preferred method.

The maximum particle size shall be 100 µm.

NOTE 3 The purpose of grinding is to obtain a sample sufficiently fine to be fused easily but below a set limit of introduced contamination. But for certain samples that are difficult to fuse (e.g. chrome ores), finer grinding to less than 60 µm might be necessary.

One of the following two methods shall be used to obtain the required particle size.

a) For mechanical grinding devices, establish what grinding times are sufficient to grind the various samples to be analysed to the correct fineness and thereafter apply these minimum times for grinding. In order to establish grinding times, use the mechanical grinder to prepare typical materials analysed for progressively increasing lengths of time of ~2 min. Sieve each ground sample through a 100 µm sieve until a grinding time is reached where the entire sample passes through the sieve. Then use this time for that material or the longest time of any material analysed, if applied to all materials. When grinding hard materials, such as chromite, sieving shall be used, but this might induce segregation. Therefore, after sieving, the sample shall be mixed thoroughly by stirring or tumbling prior to transferring to a sample tube. Because heavier minerals can segregate on standing, it is advisable to stir the sample once more, prior to weighing out.

b) After hand-grinding for 20 s, sieve the ground powder through a sieve of 100 µm aperture, in accordance with ISO 565. Regrind any material remaining on the sieve for a further 20 s, sieve and repeat this procedure until the whole of the sample passes through the sieve. Transfer the sample to a suitable container and mix for 1 min, using a mechanical mixer such as a vertical linear mixer.