

Chemical analysis of chrome-bearing refractory products and chrome-bearing raw materials (alternative to the X-ray fluorescence method)

**Part 1: Apparatus, reagents,
dissolution and determination of
gravimetric silica (ISO 20565-1:2008)**

ICS 81.080

National foreword

This British Standard is the UK implementation of EN ISO 20565-1:2008. Together with BS EN ISO 20565-2:2008 and BS EN ISO 20565-3:2008, it supersedes BS 1902-2.2:1974 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee RPI/1, Refractory products and materials.

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.

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 March 2009

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ISBN 978 0 580 72652 1

Amendments/corrigenda issued since publication

Date	Comments
30 September 2010	Addition of supersession details

English Version

**Chemical analysis of chrome-bearing refractory products and
chrome-bearing raw materials (alternative to the X-ray
fluorescence method) - Part 1: Apparatus, reagents, dissolution
and determination of gravimetric silica (ISO 20565-1:2008)**

Analyse chimique des produits réfractaires contenant du chrome et des matières premières contenant du chrome (méthode alternative à la méthode par fluorescence de rayons X) - Partie 1: Appareillage, réactifs, mise en solution et détermination de la teneur en silice par gravimétrie (ISO 20565-1:2008)

Chemische Analyse von chromhaltigen feuerfesten Erzeugnissen und chromhaltigen Rohstoffen (Alternative zum Röntgenfluoreszenzverfahren) - Teil 1: Geräte, Reagenzien, Aufschluss und gravimetrische Bestimmung von Silicium(IV)-oxid (ISO 20565-1:2008)

This European Standard was approved by CEN on 8 November 2008.

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Foreword

This document (EN ISO 20565-1:2008) 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 June 2009, and conflicting national standards shall be withdrawn at the latest by June 2009.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

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Endorsement notice

The text of ISO 20565-1:2008 has been approved by CEN as a EN ISO 20565-1:2008 without any modification.

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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.

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ISO 20565-1 was prepared by Technical Committee ISO/TC 33, *Refractories*, in collaboration with Technical Committee CEN/TC 187, *Refractory products and materials*.

ISO 20565 consists of the following parts, under the general title *Chemical analysis of chrome-bearing refractory products and chrome-bearing raw materials (alternative to the X-ray fluorescence method)*:

- *Part 1: Apparatus, reagents, dissolution and determination of gravimetric silica*
- *Part 2: Wet chemical analysis*
- *Part 3: Flame atomic absorption spectrometry (FAAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES)*

Chemical analysis of chrome-bearing refractory products and chrome-bearing raw materials (alternative to the X-ray fluorescence method) —

Part 1:

Apparatus, reagents, dissolution and determination of gravimetric silica

1 Scope

This part of ISO 20565 specifies methods for the chemical analysis of chrome-bearing refractory products and chrome-bearing raw materials, using traditional (“wet”) methods, ICP-AES spectrometry and FAAS spectrometry. It covers apparatus, reagents, dissolution and determination of gravimetric silica.

It is applicable in the ranges of determination given in Table 1.

ISO 20565 provides alternatives to the X-ray fluorescence (XRF) method given in ISO 12677.

Table 1 — Range of determination (% by mass)

Component	Range
SiO ₂	0,5 to 10
Al ₂ O ₃	2 to 30
Fe ₂ O ₃	0,5 to 25
TiO ₂	0,01 to 1
MnO	0,01 to 1
CaO	0,01 to 3
MgO	15 to 85
Na ₂ O	0,01 to 1
K ₂ O	0,01 to 1
Cr ₂ O ₃	2 to 60
ZrO ₂	0,01 to 0,5
P ₂ O ₅	0,01 to 5
LOI	–0,5 to 5
NOTE These values are after the loss on ignition (LOI) has been taken into account.	

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 6353-1, *Reagents for chemical analysis — Part 1: General test methods*

ISO 6353-2, *Reagents for chemical analysis — Part 2: Specifications — First series*

ISO 6353-3, *Reagents for chemical analysis — Part 3: Specifications — Second series*

ISO 26845:2008, *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 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 26845 apply.

4 Apparatus

Use normal laboratory apparatus and the following.

NOTE Other apparatus is defined in ISO 26845.

4.1 Polyethylene tetrafluoride beaker, 200 ml.

Heat in nitric acid for at least 2 h and wash in water.

Instead of a polyethylene tetrafluoride beaker, a 150 ml platinum dish may be used.

4.2 Volumetric flasks, 100 ml each, made of plastics material as appropriate for each solution, calibrated as follows.

Wash the plastic flask and stand it to dry naturally, or wash it with water, ethanol and diethylether and dry it by sending air into it. Cut the 20 graduations off a sheet of section paper (1 mm²) into a strip and attach it on the marked line of the plastic flask with the central line of the paper. Weigh the flask to the nearest milligram.

Pour water (at a temperature approximately equal to the room temperature) up to the lower end (B) of the strip and weigh the flask. Then add water up to the upper end (A) of the strip and weigh the flask. Separately, measure the water temperature (°C), the room temperature (°C) and the atmospheric pressure (kPa). Obtain the correct marked line [i.e. the number of graduations counted from bottom edge (B) of graduation paper], S , by using Equation (1).

$$S = \frac{\left[\frac{1000\,000 - (m + m')}{f} - m_B \right]}{\frac{m_A - m_B}{20}} \quad (1)$$

where

m_A is the mass of water up to the top edge (A) of the graduation paper, in milligrams (mg), i.e. [(mass obtained by second weighing) — (mass of Erlenmeyer flask)]:

$$m_A = m_A + (\text{mass of water from A to B});$$

- m_B is the mass of water up to bottom edge (B) of the graduation paper, in milligrams (mg), i.e. [(mass obtained by first weighing) — (mass of Erlenmeyer flask)];
- m is the correction value, in milligrams (mg), at 20 °C room temperature, and 101,325 kPa atmospheric pressure;
- m' is the correction value, in milligrams (mg), due to the deviation from room temperature of 20 °C, and an atmospheric pressure 101,325 kPa; it is $\pm 4,0$ mg per ± 1 °C room temperature, and $\pm 1,3$ mg per $\pm 0,133$ kPa atmospheric pressure;
- f is 100 ml (nominal capacity of plastic flask to be calibrated).

Change the paper strip for a fresh paper strip marked with the correct marked line made on the plastic flask. When using this flask, employ the marked line newly obtained above.

5 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

Reagents shall conform to the requirements of ISO 6353-1, ISO 6353-2 and ISO 6353-3 as appropriate. Specific requirements for reagents are given in the appropriate clause.

Reagents that are listed in ISO 26845 and the following.

NOTE Where solutions are referred to as (1+1), etc., this implies v/v unless otherwise stated.

5.1 Stock reagents

5.1.1 Acetic acid (ISO 6353-2, R 1), concentrated, minimum 99,7 % by mass.

5.1.2 Aluminum chloride solution, Al (25 mg/ml).

Dissolve 123,5 g of aluminum chloride into water and dilute to 1 l with water.

5.1.3 Ammonium chloride.

5.1.4 Ammonia solution (concentrated) (ISO 6353-2, R 3), minimum 25 % by mass.

5.1.5 Ammonia solution (1+1).

Add 1 volume of ammonia solution (concentrated) (5.1.4) to 1 volume of water.

5.1.6 Ammonia solution (1+9).

Add 1 volume of ammonia solution (concentrated) (5.1.4) to 9 volumes of water.

5.1.7 Ammonium acetate solution, 200 g/l.

Dissolve 200 g of ammonium acetate with water and dilute to 1 l.

5.1.8 Ammonium molybdate solution, 20 g/l.

Dissolve 2 g of ammonium molybdate tetrahydrate in 20 ml of hot water, filter if necessary, and dilute to 100 ml with the addition of 60 ml of sulfuric acid (1+1) and water.