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

Analyse chimique des matériaux réfractaires — Exigences générales pour les méthodes d'analyse chimique par voie humide, par spectrométrie d'absorption atomique (AAS) et par spectrométrie d'émission atomique avec plasma induit par haute fréquence (ICP-AES)

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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 26845 was prepared by Technical Committee ISO/TC 33, *Refractories*.

It is to be used in conjunction with ISO 10058-1, ISO 10058-2 and ISO 10058-3, ISO 20565-1, ISO 20565-2 and ISO 20565-3, ISO 21079-1, ISO 21079-2 and ISO 21079-3, and ISO 21587-1, ISO 21587-2 and ISO 21587-3.

Introduction

This International Standard gives the general requirements common to the standards used for the chemical analysis of refractories and refractory products, i.e.:

ISO 10058, *Chemical analysis of magnesite and dolomite refractory products (alternative to the X-ray fluorescence method):*
- *Part 1: Apparatus, reagents, dissolution and gravimetric silica*
- *Part 2: Wet chemical analysis*
- *Part 3: Flame atomic absorption spectrometry (FAAS) and inductively coupled plasma emission spectrometry (ICP-AES)*

ISO 20565 *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 gravimetric silica*
- *Part 2: Wet chemical analysis*
- *Part 3: Flame atomic absorption spectrometry (FAAS) and inductively coupled plasma emission spectrometry (ICP-AES)*

ISO 21079 *Chemical analysis of refractories containing alumina, zirconia and silica — Refractories containing 5 % to 45 % of ZrO2 (alternative to the X-ray fluorescence method) —*
- *Part 1: Apparatus, reagents and dissolution*
- *Part 2: Wet chemical analysis*
- *Part 3: Flame atomic absorption spectrometry (FAAS) and inductively coupled plasma emission spectrometry (ICP-AES)*

ISO 21587 *Chemical analysis of aluminosilicate refractory products (alternative to the X-ray fluorescence method) —*
- *Part 1: Apparatus, reagents, dissolution and gravimetric silica*
- *Part 2: Wet chemical analysis*
- *Part 3: Inductively coupled plasma and atomic absorption spectrometry methods*

These International Standards give wet chemical, AAS and ICP methods for the analysis of refractory materials and products. They are to be used as an alternative to ISO 12677, when the laboratory does not have an XRF instrument or its instrument does not meet the requirements of ISO 12677: *Chemical analysis of refractory products by XRF — Fused cast bead method*.

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

1 Scope

This International Standard specifies apparatus, reagents, sampling, sample preparation, terms and definitions, basic procedures, loss on ignition and reporting of results applicable to the following standards, which are used for the chemical analysis of refractory products and raw materials by wet chemical, AAS and ICP-AES:

ISO 10058-1, ISO 10058-2 and ISO 10058-3;

ISO 20565-1, ISO 20565-2 and ISO 20565-3;

ISO 21079-1, ISO 21079-2 and ISO 21079-3;

ISO 21587-1, ISO 21587-2 and ISO 21587-3.

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 31-0, *Quantities and units — Part 0: General principles*

ISO 836, *Terminology for refractories*

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

ISO 5022, *Shaped refractory products — Sampling and acceptance testing*

ISO 6286, *Molecular absorption spectrometry — Vocabulary — General — Apparatus*

ISO 6353-1:1982, *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 6955, *Analytical spectroscopic methods — Flame emission, atomic absorption, and atomic fluorescence — Vocabulary*

ISO 8656-1, *Refractory products — Sampling of raw materials and unshaped products — Part 1: Sampling scheme*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 836 and the following apply.

3.1
dry unshaped refractories
particles and/or powder of unshaped refractories without liquid (mortar and mixture of refractory aggregate and pitch or resin)

3.2
wet unshaped refractories
particles and/or powder of unshaped refractories with liquid (mortar and mixture of refractory aggregate and pitch or resin)

3.3
alumina-zirconia-silica refractories
refractories in which aluminium oxide (1 % to 80 % by mass), zirconium oxide (including hafnium oxide) (5 % to 50 % by mass) and silicon(IV) oxide (0,1 % to 45 % by mass) are used as chemical components

4 Apparatus

Standard laboratory apparatus and the following.

4.1 Spectrometers

4.1.1 Atomic absorption (AA) spectrometer, conforming to the requirements of ISO 6955. An instrument which can be operated using a dinitrogen oxide/acetylene flame for the determinations of aluminium oxide and calcium oxide is appropriate.

4.1.2 Flame emission spectrophotometer, comprising a stand-alone flame emission instrument or an AA spectrometer used in emission mode.

4.1.3 Inductively coupled plasma atomic emission (ICP-AE) spectrometer.

4.1.4 Molecular absorption spectrometer, conforming to the requirements of ISO 6286.

4.1.5 Photometer, with a 1 cm cell.

4.2 Heaters

4.2.1 Burner, such as a Mecker burner or a Bunsen burner.

4.2.2 Electric muffle furnace, suitable for use above 1 150 °C.

4.2.3 Sand bath, comprising an electrically heated or burner-heated iron plate with sand, which can be heated to the temperature at which white smoke of sulfuric acid occurs.

4.2.4 Steam bath, electric- or burner-type, which can be controlled at the appropriate temperature.

4.3 Devices

4.3.1 Analytical electronic balance, readable to the nearest 0,1 mg.

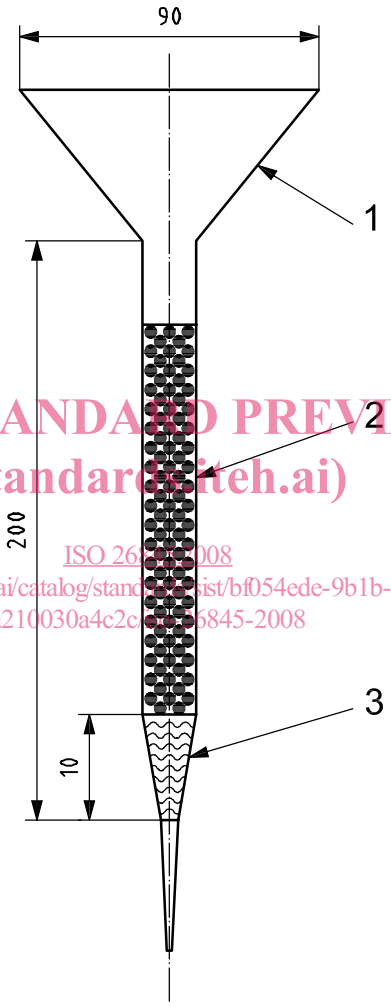
4.3.2 Magnetic stirrer, comprising a stirring system using a magnetic rotating bar coated with ethylene-4-fluoride resin.

4.3.3 Filter paper, ashless, medium pore.

4.3.4 Cation-exchange resin column, consisting of plastic wool (10 mm) packed into a plastic tube (diameter 12 mm × 200 mm) with a funnel attached to the top and an eluent outlet sharp-pointed to the bottom.

Pour a slurry (18 ml) of the water-expanded strongly acidic cation-exchange resin [divinylbenzene 8 % (DVB), 75 µm to 150 µm] into the column. Adjust the flow rate to 1,0 ml to 1,5 ml per minute by the packing condition of the plastic wool. Wash the column with hydrochloric acid (1+2, 120 ml) and water (70 ml). An example of a normal column is shown in Figure 1.

Dimensions in millimetres



Key

1 polypropylene funnel

2 cation-exchange resin

3 polypropylene wool

Figure 1 — Example of a cation-exchange resin column

4.4 Platinum ware

The platinum apparatus may be of platinum or platinum alloy.

4.4.1 Platinum crucible, with a volume of 20 ml or 30 ml.

4.4.2 Platinum dish, with the diameters of the base and wall almost the same, and a height of approximately half the diameter. 75 ml or 150 ml volume are commonly used sizes.

4.5 Glassware

4.5.1 Burette, with a 0,1 ml scale and a maximum volume of 50 ml.

4.5.2 Conical flask, capable of holding a volume of 500 ml and 1 l.

4.5.3 Desiccator, containing dried silica gel as the drying agent.

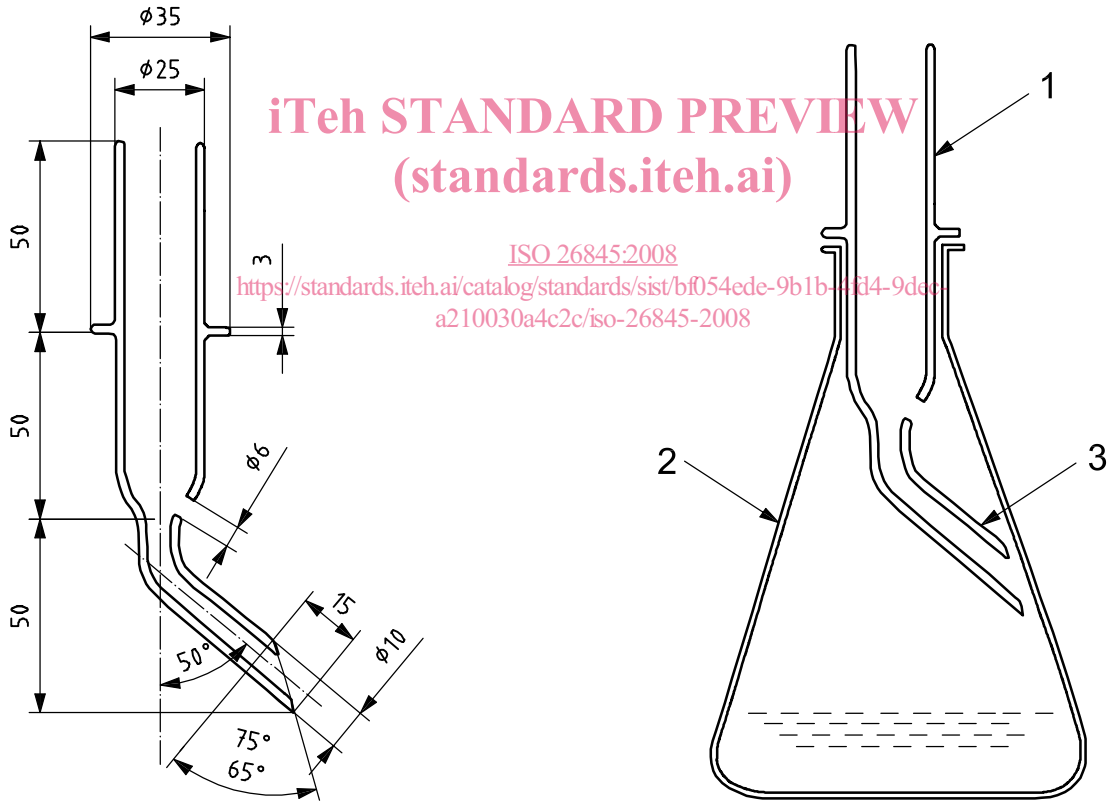
4.5.4 Erlenmeyer flask, capable of holding a volume of 500 ml.

4.5.5 Glass beakers, with a range of appropriate volumes (100 ml, 200 ml, 300 ml, 400 ml, 500 ml, etc.).

4.5.6 One-mark pipette, suitable for the transfer of each sample solution or standard solution.

4.5.7 Reflux steel head, comprising a funnel, with a long leg capable of being bent so that the end touches the inside wall of a flask (see Figure 2).

Dimensions in millimetres



a) Steel head **b) Steel head setting**

Key

1 steel head
2 erlenmeyer flask
3 head that contacts internal wall

Figure 2 — Steel head for reflux

4.5.8 Separating funnels, 250 ml and 500 ml, with stoppers.

4.5.9 Volumetric flasks, made of glassware (100 ml, 250 ml, 500 ml, 1 000 ml, etc.) as appropriate for each solution.

4.5.10 Volumetric pipette, of capacity 5 ml.

4.5.11 Watch glass, of diameter 75 mm.

4.6 Porcelain ware

4.6.1 Porcelain basin, with a diameter of 125 mm.

4.6.2 Porcelain crucible, capable of holding a volume of 15 ml.

4.7 Plastic ware

4.7.1 Plastic beaker, of capacity 100 ml.

4.7.2 Polyethylene tetrafluoride beaker, of capacity 200 ml. Heat in nitric acid for at least 2 h and wash in water.

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

4.7.3 Volumetric flasks, made of plastic, as appropriate for each solution, and calibrated as follows. Either

a) wash a plastic flask thoroughly, invert it and allow it to stand to dry naturally, or

b) wash a plastic flask with water, ethanol and diethylether and dry it by blowing air into it.

Cut 20 graduations from a sheet of section paper (1 mm squares) into a strip and attach it to the marked line of the plastic flask, so that the line coincides with the central line of the paper. Weigh the flask to the nearest milligram.

Add water at approximately room temperature up to the lower end (B) of the strip, and weigh the flask. Then add more water up to the upper end (A) of the strip, and weigh the flask. Separately, measure the water temperature, in degrees Celsius, the room temperature, in degrees Celsius and the atmospheric pressure, in kilopascals, and obtain the correct marked line, S, the number of graduations counted from the bottom edge (B) of the graduation paper, using the following equation:

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

where

m_A is the mass of water up to the top edge (A) of the graduated paper, in milligrams, [i.e. (mass obtained by second weighing) – (mass of Erlenmeyer flask)]; $m_A = m_B +$ [mass of water from (A) to (B)];

m_B is the mass of water up to to bottom edge (B) of the graduated paper, in milligrams, [i.e. (mass obtained by first weighing) – (mass of Erlenmeyer flask)] ;

m is the correction value, in milligrams, at 20 °C room temperature, and 101,325 kPa {760 mmHg} atmospheric pressure;