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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

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

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.

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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 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_2O_3	2 to 30
Fe_2O_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_2O	0,01 to 1
K_2O	0,01 to 1
Cr_2O_3	2 to 60
ZrO_2	0,01 to 0,5
P_2O_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 mm^2) 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).

$$S = \frac{\left[\dfrac{1\,000\,000 - (m + m')}{f} - m_\mathrm{B}\right]}{\dfrac{m_\mathrm{A} - m_\mathrm{B}}{20}} \tag{1}$$

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)]:

$m_\mathrm{A} = m_\mathrm{A} +$ (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 ± 4,0 mg per ± 1 °C room temperature, and ± 1,3 mg per ± 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.

5.1.9 L (+)-ascorbic acid solution, 100 g/l.

Dissolve 10 g of L (+)-ascorbic acid in water and dilute to 100 ml. This solution shall be kept in the dark and at low temperature. Discard after 2 weeks.

5.1.10 Boric acid solution, 40 g/l.

Dissolve 40 g of boric acid with water and dilute to 1 l.

5.1.11 Chloroform.

5.1.12 Cupferron solution, 60 g/l.

Dissolve 6 g of cupferron in 100 ml of water; filter if necessary. Prepare this solution freshly, as required. Store the solid reagent in a tightly stoppered bottle in the presence of a piece of ammonium carbonate to prevent decomposition.

5.1.13 Diantipyrylmethane (DAM) solution, 10 g/l.

Dissolve 1 g of diantipyrylmethane monohydrate in 30 ml of hydrochloric acid (1+50), and dilute to 100 ml with water.

5.1.14 Fusion mixture, an equimolecular mixture of sodium and potassium carbonates.

5.1.15 Hydrazinium dichloride solution, 150 g/l.

Dissolve 15 g of hydrazinium dichloride in water and dilute to 100 ml.

5.1.16 Hydrochloric acid (concentrated), (ISO 6353-2, R 13), 35 % by mass.

5.1.17 Hydrochloric acid (1+1).

Add 1 volume of hydrochloric acid (concentrated) (5.1.16) to 1 volume of water.

5.1.18 Hydrochloric acid (1+50).

Add 1 volume of hydrochloric acid (concentrated) (5.1.16) to 50 volumes of water.

5.1.19 Hydrofluoric acid (concentrated), (ISO 6353-3, R 67), 40 % to 42 % by mass.

5.1.20 Hydrofluoric acid (1+9).

Add 1 volume of hydrofluoric acid (concentrated) (5.1.19) to 9 volumes of water. Store in a plastic container.

5.1.21 Hydroxyammonium chloride solution, 100 g/l.

Dissolve 10 g of hydroxyammonium chloride in warm water, filter if necessary, cool and dilute to 100 ml.

5.1.22 Liquid cation-exchange resin, for example, Amberlite LA-2 resin (1+4), made by adding 50 ml of the resin to 200 ml of chloroform and mixing.

Transfer the mixture to a 500 ml separating funnel and shake gently with 25 ml of H_2SO_4 (1+9) for a few seconds, releasing the pressure frequently. Run off the organic layer into a 250 ml measuring cylinder.

5.1.23 Nitric acid (concentrated), 70 % by mass.

5.1.24 Nitric acid (1+1).

Add 1 volume of nitric acid (concentrated) (5.1.23) to one volume of water.

5.1.25 2,2′, 2″-Nitrilotriethanol (1+1).

Add 1 volume of 2,2′, 2″-nitrilotriethanol to one volume of water.

5.1.26 1,10-Phenanthrolinium chloride solution, 1 g/l.

Dissolve 0,1 g of 1,10-phenanthrolinium chloride monohydrate in water, dilute to 100 ml, and keep in a dark and cool place. However, discard the solution if it colours during storage.

5.1.27 Phosphoric acid (concentrated), 85 % by mass.

5.1.28 Phosphoric acid, (2+3).

Add 2 volumes of phosphoric acid (concentrated) (5.1.27) to 3 volumes of water, then mix and allow to cool.

5.1.29 Phosphoric acid (1+9).

Add 1 volume of phosphoric acid (concentrated) (5.1.27) to 9 volumes of water, then mix and allow to cool.

5.1.30 Potassium hydroxide solution, 250 g/l.

Dissolve 250 g of potassium hydroxide in water, cool and dilute to 1 l. Store in a polyethylene bottle.

5.1.31 Potassium periodate.

5.1.32 Sodium carbonate, anhydrous.

In some brands of this agent, a minute quantity of calcium oxide is included. When determining calcium oxide by inductively coupled plasma atomic emission spectrometry (ICP-AES), a pure high-quality agent needs to be used.

5.1.33 Sodium hydroxide solution, 100 g/l.

Cautiously dissolve 20 g of sodium hydroxide in 150 ml of water, while stirring and cooling. After cooling, dilute to approximately 200 ml.

5.1.34 Sulfuric acid (concentrated), (ISO 6353-2, R 37), minimum 95 % by mass.

5.1.35 Sulfuric acid (1+1).

Cautiously add, while stirring, 200 ml of sulfuric acid (concentrated) (5.1.34) to 200 ml of water, cooling the solution.

5.1.36 Sulfuric acid (1+9).

Cautiously add, while stirring, 20 ml of sulfuric acid (concentrated) (5.1.34) to 180 ml of water, cooling the solution.

5.1.37 L (+)-tartaric acid solution, 100 g/l.

Dissolve 100 g of L (+)-tartaric acid with water and dilute to 1 l.

5.2 Indicators

5.2.1 Bromophenol blue solution, 1 g/l.

Grind 0,1 g of bromophenol blue with 1,5 ml of sodium hydroxide solution (4 g/l), dilute to 100 ml with water and mix.

5.2.2 Calcein indicator (screened), (3,3vv-bis[*N*,*N*′-bis(carboxylmethyl) aminomethyl] fluorescein).

Mix, by grinding together, 0,2 g calcein, 0,12 g of thymolphthalein and 20,0 g of potassium chloride.

5.2.3 Dithizone solution, 0,25 g/l.

Dissolve 0,012 5 g of dithizone in 50 ml of ethanol (95 %). The solution will keep for a week.

5.2.4 Xylenol orange solution, 1 g/l.

Dissolve 0,10 g of the xylenol orange with water and dilute to 100 ml. This solution shall be kept in the dark and at low temperature. Discard after 4 weeks.

5.3 Standard solutions

5.3.1 0,1 mol/l ammonium iron(II) sulfate solution.

Measure 300 ml of water, add gradually 30 ml of sulfuric acid (concentrated) (5.1.34) while agitating and allow it to cool. Then add 40 g of ammonium iron(II) sulfate hexahydrate and 700 ml of water to dissolve it, and store the solution in an airtight container. Prepare this when it is needed.

Dry the required amount (about 0,3 g) of potassium dichromate, reference material for volumetric analysis, at 150 °C for about 60 min, and allow it to cool in a desiccator. Weigh 0,12 g to the nearest 0,1 mg in a 200 ml conical flask and add 100 ml of water to dissolve it. Then add gradually 30 ml of sulfuric acid (concentrated) (5.1.34) while agitating and allow it to cool. As an indicator, add several drops of ferroin solution, and titrate it with the 0,1 mol/l ammonium iron(II) sulfate solution. Make it the end point when the solution turns from bluish green to reddish brown.

Calculate the factor, F, of the 0,1 mol/l ammonium iron(II) sulfate solution using Equation (2).

$$F = \frac{m}{0{,}004\,903 \times V} \times \frac{A}{100} \tag{2}$$

where

m	is the mass of the weighed potassium dichromate, in grams (g);
A	is the purity of the potassium dichromate, in percentage (%) by mass;
0,004 903	is the mass of potassium dichromate equivalent to 1 ml of 0,1 mol/l ammonium iron(II) sulfate solution, in grams (g);
V	is the volume of the 0,1 mol/l ammonium iron(II) sulfate solution needed for titration, in millilitres (ml).

5.3.2 Aluminium oxide standard solution, Al_2O_3 1 mg/ml.

Wash the surface of a sufficient amount (about 1 g) of aluminium metal (purity greater than 99,9 % by mass) with hydrochloric acid (1+4) to dissolve the oxidized layer. Then wash with water, ethanol, and diethyl ether in succession, and dry in a desiccator. Weigh 0,529 2 g of the aluminium and transfer into a 250 ml beaker. Cover with watch glass, add 20 ml hydrochloric acid (1+1), and heat to dissolve. After cooling, dilute to 1 000 ml in a volumetric flask with water.

5.3.3 Calcium oxide standard solution, CaO 1 mg/ml.

Dissolve 1,785 g of pure calcium carbonate, previously dried at 150 °C, in a slight excess of diluted hydrochloric acid (1+4) in a 250 ml beaker covered with a watch glass. Boil to expel carbon dioxide, cool and dilute to 1 000 ml in a volumetric flask.

5.3.4 **Diluted calcium oxide standard solution**, CaO 0,05 mg/ml.

Dilute precisely the calcium oxide standard solution (5.3.3) with water to a concentration of one twentieth.

5.3.5 **Chromium (III) oxide standard solution**.

Dry about 2 g to 3 g of potassium dichromate at 110 °C for at least 2 h. Weigh 1,935 g of this and dissolve in water, diluting to 1 000 ml in a volumetric flask.

5.3.6 0,01 mol/l CyDTA **solution**.

Add 8 ml of sodium hydroxide solution (100 g/l) and about 150 ml of water to 3,65 g of 1,2-cyclohexanediamine-*N*,*N*,*N*′,*N*′-tetraacetic acid monohydrate (CyDTA) and dissolve by heating. After cooling, dilute to 1 000 ml with water.

5.3.7 0,05 mol/l CyDTA **solution**.

Add 40 ml of sodium hydroxide solution (100 g/l) and about 500 ml of water to 18,25 g of 1,2-cyclohexanediamine-*N*,*N*,*N*′,*N*′-tetraacetic acid monohydrate (CyDTA) and dissolve by heating. After cooling, dilute to 1 000 ml with water.

Transfer 250 ml of magnesium oxide standard solution (MgO 1 mg/ml) (5.3.14) to a 500 ml conical flask. Add 100,0 ml of the CyDTA solution. Then add 2 g of ammonium chloride and 25 ml of ammonia solution, concentrated, 25 % by mass (5.1.4). Titrate with the CyDTA solution, using Solochrome Black 6B as the indicator, from red through purple to the last change to a clear ice blue.

Calculate the factor, F, of the 0,05 mol/l CyDTA standard solution using Equation (3).

$$F = \frac{f \times \dfrac{1 \times 250}{40{,}30}}{0{,}05 \times V} \tag{3}$$

where

f is the factor of the magnesium oxide standard solution (MgO 1 mg/ml) (see ISO 26845);

V is the volume of the 0,05 mol/l CyDTA solution, in millilitres (ml).

5.3.8 **0,02 mol/l EDTA standard solution**.

Dissolve 7,5 g of EDTA2Na (ethylenediamine-tetraacetic acid disodium salt dihydrate) with 1 l of water. Store in a plastic bottle. Transfer 50 ml of 0,01 mol/l zinc solution, obtained in 5.3.31, to a 300 ml beaker and add 50 ml of water. Drop in sodium hydroxide solution (100 g/l) of up to pH 6 to pH 8 and add 2 ml of buffer solution (pH 10) (see 4.1.18 of ISO 26845:2008) and, while stirring, add 3 to 4 drops of Eriochrome Black T solution as an indicator and titrate with 0,02 mol/l EDTA solution. Observe the end point when the reddish purple colour of the solution is altered to blue.

Calculate the factor, F, of the 0,02 mol/l EDTA standard solution using Equation 4.

$$F = \frac{f \times 50}{2 \times V} \tag{4}$$

where

f is the factor of the 0,01 mol/l zinc solution;

V is the volume of the 0,02 mol/l EDTA solution, in millilitres (ml).

5.3.9 0,05 mol/l EGTA standard solution.

Dissolve 19,1 g of EGTA [ethylene glycol di(aminoethyl)-tetra acetic acid] in 500 ml of water by progressively adding a minimum amount of potassium hydroxide solution, 250 g/l (5.1.30).

NOTE Approximately 25 ml should be required.

Dilute to 1 l. Store in a polyethylene bottle.

Transfer 10 ml of magnesium oxide solution (MgO 10 mg/ml) (see ISO 26845) to a 250 ml volumetric flask and 20 ml of the EGTA standard solution. Dilute to 150 ml and add potassium hydroxide, 250 g/l (5.1.30) until no further precipitation occurs. Add 10 ml in excess, followed by 10 ml of Magflok solution (see ISO 26845). Dilute to 250 ml, shake and allow to stand for about 10 min to settle. Filter through a dry, coarse filter paper (125 mm) into a dry beaker. Pipette 200 ml of the filtrate into a 500 ml conical flask and add 15 ml of potassium hydroxide solution, 250 g/l (5.1.30). Titrate with calcium oxide standard solution (CaO 1 mg/ml) using calcein as indicator to the first appearance of green fluorescence.

Calculate the factor, F, of the 0,05 mol/l EGTA standard solution using Equation (5).

$$F = \frac{0{,}013\,50 \times f \times V}{0{,}05 \times 20 \times \frac{4}{5}} \tag{5}$$

where

f is the factor of the calcium oxide standard solution (CaO 1 mg/ml);

V is the volume of the calcium oxide standard solution (CaO 1 mg/ml), in millilitres (ml).

5.3.10 Internal standard solution.

Transfer 10 ml of standard scandium oxide solution (5.3.23) and standard yttrium oxide solution (5.3.30) into a volumetric 100 ml flask and dilute to the mark with water. Prepare when necessary.

5.3.11 Iron(III) oxide standard solution, Fe_2O_3 1 mg/ml.

Wash the surface of a sufficient amount (about 1 g) of iron metal (purity greater than 99,9 % by mass) with hydrochloric acid (1+4). Dissolve the oxidized layer, wash with water, ethanol, and diethyl ether in succession. Then dry in a desiccator. Weigh 0,699 4 g of this, transfer to a 200 ml beaker, and cover with a watch glass. Add 30 ml of hydrochloric acid (1+1), and heat on a steam bath until dissolved. After cooling, dilute to 1 000 ml in a volumetric flask with water.

5.3.12 Iron(III) oxide standard solution, Fe_2O_3 0,2 mg/ml.

Wash the surface of the iron (purity greater than 99,9 % by mass) with hydrochloric acid (1+3) and dissolve the oxidized layer. Wash with water, ethanol and diethyl ether in succession and dry in a desiccator. Weigh 0,139 9 g of iron, transfer to a 200 ml beaker and cover with a watch glass. Add 20 ml of hydrochloric acid (1+1) and heat to dissolve on a steam bath. After cooling, dilute with water to 1 000 ml in a volumetric flask.

5.3.13 Diluted iron(III) oxide standard solution, Fe_2O_3 0,04 mg/ml.

Transfer 40 ml of the iron(III) oxide standard solution, 1 mg/ml (5.3.11) to a 1 000 ml volumetric flask and dilute to the mark with water. Prepare this solution freshly on the day of use.

5.3.14 Magnesium oxide standard solution, MgO 1 mg/ml.

Wash the surface of a sufficient amount of magnesium metal (purity greater than 99,9 % by mass) with hydrochloric acid (1+1) to dissolve the oxidized layer. Then wash with water, ethanol, and diethyl ether in succession and dry in a desiccator. Weigh 0,301 5 g of the washed magnesium, transfer to a 200 ml beaker and cover with a watch glass. Add 10 ml of hydrochloric acid (1+1), and heat on a steam bath until dissolved. After cooling, transfer to a 500 ml volumetric flask, and dilute to the mark with water.

5.3.15 Diluted magnesium oxide standard solution, MgO 0,1 mg/ml.

Dilute the magnesium oxide standard solution (5.3.14) to one tenth with water.

5.3.16 Manganese(II) oxide standard solution, MnO 1 mg/ml.

Wash the surface of the manganese (purity greater than 99,9 % by mass) with hydrochloric acid (1+4) to dissolve the oxidized layer. Wash with water, ethanol, and diethyl ether in succession and dry in a desiccator. Weigh 0,774 5 g of manganese and transfer it to a 200 ml beaker. Add 20 ml of hydrochloric acid (1+1) and heat until dissolved using a water bath. After cooling, transfer to a 1 000 ml volumetric flask.

5.3.17 Diluted manganese(II) oxide standard solution, MnO 0,04 mg/ml.

Add 10 ml of nitric acid (1+1) to 40 ml of the manganese(II) oxide standard solution and dilute to 1 000 ml with water. Prepare this solution freshly for each test.

5.3.18 Phosphorus(V) oxide standard solution, P_2O_5 0,1 mg/ml.

Heat about 0,5 g of potassium dihydrogen phosphate at 110 °C ± 5 °C for 3 h and allow to cool in a desiccator. Weigh 0,191 7 g, transfer to a 200 ml beaker and dissolve with 100 ml of water. Transfer into a 1 000 ml volumetric flask and dilute to the mark with water.

5.3.19 Diluted phosphorus(V) oxide standard solution, P_2O_5 0,04 mg/ml.

Dilute the standard phosphorus(V) oxide solution precisely to the concentration of four tenths by water.

5.3.20 Diluted phosphorus(V) oxide standard solution, P_2O_5 0,01 mg/ml.

Dilute the standard phosphorus(V) oxide solution precisely to the concentration of one tenth by water.

5.3.21 1/60 mol/l potassium dichromate solution.

Take the required amount (about 6 g) of potassium dichromate, dry at 150 °C for about 60 min, and allow it to cool in a desiccator. Weigh 4,9 g to 5,0 g of this to the nearest 0,1 mg, transfer it to a 1 000 volumetric flask, and add water to dissolve it. Further add water up to the mark. Store in an airtight container.

Calculate the factor, F, of the 1/60 mol/l potassium dichromate solution using Equation (6).

$$F = \frac{m}{4{,}903} \times \frac{A}{100} \tag{6}$$

where

m is the mass of the weighed potassium dichromate, in grams (g);

A is the purity of the potassium dichromate, in percentage (%) by mass;

4,903 is the mass of potassium dichromate in 1 l of 1/60 mol/l potassium dichromate solution, in grams (g).

5.3.22 Potassium oxide standard solution, K_2O 1 mg/ml.

Transfer 2 g to 3 g of potassium chloride into a platinum crucible (30 ml) and ignite at 600 °C ± 25 °C for 60 min. Allow to cool in a desiccator. Weigh 1,582 9 g of this substance and transfer it to a 200 ml beaker.

NOTE For example, weigh precisely on a platinum weighing dish, and transfer into a beaker taking care not to scatter any of this substance. Wash off the residue adhering to the platinum dish with a small amount of water.

Dissolve in 100 ml of water, transfer to a 1 000 ml volumetric flask and dilute to the mark with water.

5.3.23 Scandium oxide standard solution, Sc 1 mg/ml.

Dry 0,2 g of scandium oxide at 110 °C ± 5 °C for 60 min, cool in a desiccator and weigh out 0,153 4 g. Transfer to a 100 ml beaker and gradually add 10 ml of hydrochloric acid (1+1) to dissolve. Dilute precisely to 100 ml in a volumetric flask with water.

A commercial scandium oxide standard solution may be used.

5.3.24 Silicon(IV) oxide standard solution, SiO_2 1 mg/ml.

Weigh 0,7 g to 1,0 g of silicon(IV) oxide (purity greater than 99,9 % by mass) in a platinum crucible (e.g. 30 ml) and heat for 30 min at 1 150 °C ± 50 °C. Cool in a desiccator and then weigh 0,500 0 g of this silicon(IV) oxide into a platinum crucible (e.g. 30 ml). Fuse the silicon(IV) oxide with 3,0 g of anhydrous sodium carbonate. Cool and wipe the outside of the crucible, and dissolve in warm water (150 ml) in a 200 ml plastic beaker while stirring using a plastic rod. Cool and dilute without heating to 500 ml in a volumetric flask. Transfer this solution to a plastic bottle immediately.

5.3.25 Diluted silicon(IV) oxide standard solution, SiO_2 0,04 mg/ml.

Dilute 10 ml of silicon(IV) oxide standard solution (1,0 mg/ml) to 250 ml with water.

5.3.26 Sodium oxide standard solution, Na_2O 1 mg/ml.

Transfer 2 g to 3 g of sodium chloride into a platinum crucible (30 ml) and ignite at 600 °C ± 25 °C for 60 min. Allow to cool in a desiccator. Weigh 1,885 9 g of this substance and transfer it to a 200 ml beaker.

Dissolve in 100 ml of water, transfer to a 1 000 ml volumetric flask and dilute to the mark with water.

5.3.27 Titanium(IV) oxide standard solution, TiO_2 1,0 mg/ml.

Weigh 0,599 4 g of titanium (purity greater than 99,9 % by mass) on a 100 ml platinum dish and cover with a watch glass made of polyethylene tetrafluoride resin. Add 20 ml of hydrofluoric acid, 15 ml of sulfuric acid (1+1) and 0,5 ml of nitric acid. Heat until dissolved on a steam bath. Remove the watch glass and rinse the watch glass with water. Heat the solution on a sand bath until sulfuric acid fumes can be detected. After cooling, rinse the inner wall of the platinum with a small amount of water and heat again until fumes are detectable. After cooling, add water and dilute to 1 000 ml in a volumetric flask with water.

5.3.28 Diluted titanium(IV) oxide standard solution, TiO_2 0,2 mg/ml, prepared by transfering 100 ml of titanium(IV) oxide standard solution (5.3.27) into a 500 ml volumetric flask and diluting to the mark with water.

Prepare when necessary.

5.3.29 Diluted titanium(IV) oxide standard solution, TiO_2 0,01 mg/ml, prepared by diluting precisely standard titanium(IV) oxide solution (TiO_2 0,2 mg/ml) (5.3.28) to the concentration of one twentieth by water.

5.3.30 Yttrium oxide solution, Y 1 mg/ml.

Dry 0,2 g of yttrium oxide at 110 °C ± 5 °C for 60 min, cool in a desiccator and weigh out 0,127 0 g. Transfer to a 100 ml beaker and gradually add 10 ml of hydrochloric acid (1+1) to dissolve. Dilute precisely to 100 ml in a volumetric flask with water.

A commercial yttrium oxide standard solution may be used.

5.3.31 0,01 mol/l zinc standard solution.

Wash the surface of the zinc (purity greater than 99,9 % by mass) with hydrochloric acid (1+4) and dissolve the oxidized layer. Wash with water, ethanol and diethyl ether in succession and dry in a desiccator. Weigh 330 mg ± 0,1 mg of zinc, cover with a watch glass and transfer it to a 300 ml beaker. Add 20 ml of water, carefully add 10 ml of nitric acid and heat until dissolved on a steam bath. After cooling, dilute to 500 ml in a volumetric flask with water.

Calculate the factor, F, of the 0,01 mol/l zinc solution using Equation (7).

$$F = \frac{m}{0,326\,95} \times \frac{A}{100} \qquad (7)$$

where

m is the mass of the weighed zinc, in grams (g);

A is the purity of the zinc, in percentage (%) by mass.

5.3.32 0,05 mol/l zinc standard solution.

Wash the surface of the zinc (purity greater than 99,9 % by mass) with hydrochloric acid (1+4) and dissolve the oxidized layer. Wash with water, ethanol and diethyl ether in succession and dry in a desiccator. Weigh 3 269 mg ± 0,1 mg of zinc, cover with a watch glass and transfer it to a 500 ml beaker. Add 50 ml of water, carefully add 15 ml of nitric acid and heat until dissolved on a steam bath. After cooling, dilute to 1 000 ml in volumetric flask with water.

Calculate the factor, F, of the 0,05 mol/l zinc solution using Equation (8).

$$F = \frac{m}{1,663\,5} \times \frac{A}{100} \qquad (8)$$

where

m is the mass of the weighed zinc, in grams (g);

A is the purity of the zinc, in percentage (%) by mass.

5.3.33 Zirconium oxide standard solution, ZrO_2 1 mg/ml.

Transfer 0,3 g of zirconium oxide (purity greater than 99,9 % by mass) into a platinum crucible (30 ml) and heat strongly at 1 100 °C ± 25 °C for 60 min. Allow to cool in a desiccator. Weigh out 0,200 0 g into a platinum crucible (30 ml) and add 4 g of potassium disulfate and fuse. After cooling, put the whole platinum crucible into a 200 ml beaker, add 100 ml of sulfuric acid (1+9) and heat until dissolved. After cooling, remove the platinum crucible and wash it. Dilute precisely to 200 ml with water.

5.3.34 Diluted zirconium oxide standard solution, ZrO_2 0,005 mg/ml.

Transfer precisely 5 ml of the zirconium oxide standard solution to a 1 000 ml volumetric flask and dilute to the mark with water.

6 Sample preparation

Prepare the sample as described in ISO 26845.

7 Basic procedure

Carry out each chemical analysis and a blank test as given in Clause 9.

8 Determination of loss on ignition (gravimetric)

Determine the loss on ignition as given in ISO 26845:2008, Clause 12.

9 Decomposition of the sample and preparation of solutions used in the analysis

9.1 General decomposition techniques

To meet all the requirements of "wet" methods, flame photometry, atomic absorption spectrometry (AAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES), the following decomposition procedures are required:

- decomposition by fusion followed by removal of the bulk of the silica present by coagulation (see 9.2.2);
- decomposition by fusion and dissolution in sulfuric acid (see 9.2.3);
- decomposition and removal of the silica by hydrofluoric acid for flame photometry (see 9.3.2);
- decomposition and removal of the silica by hydrofluoric acid for AAS (see 9.3.3);
- decomposition and removal of the silica by hydrofluoric acid for ICP-AES (see 9.3.4);
- decomposition and separation of components by cation-exchange resin column for traditional methods (see 9.4.2);
- decomposition and separation of components by resin solution for traditional methods (see 9.4.3).

NOTE Only those decomposition procedures used in the particular techniques chosen need to be prepared.

9.2 Dissolution methods by fusion

9.2.1 General

A solution is prepared for the determination of silicon(IV) oxide in solution, iron(III) oxide, titanium(IV) oxide, manganese(II) oxide, chromium(III) oxide, zirconium oxide, and phosphorus(V) oxide by one of the following two methods:

a) fusion with alkali carbonate and boric acid, dissolution of the melt in hydrochloric acid and removal of the bulk of the silicon(IV) oxide by coagulation;

b) fusion with alkali carbonate and boric acid, and dissolution of the melt in sulfuric acid.

9.2.2 Preparing a solution by fusion and coagulation

9.2.2.1 Principle

The sample is fused in anhydrous sodium carbonate and boric acid and the melt is dissolved in hydrochloric acid. A gel of silica is formed by brief evaporation and this is coagulated with polyethylene oxide. The precipitate is filtered, washed and ignited in a platinum crucible. The precipitate is ignited and weighed both before and after hydrofluoric acid treatment. The difference in mass gives the gravimetric silica. After hydrofluoric acid treatment, the residue is fused with anhydrous sodium carbonate and boric acid and the melt is dissolved in the reserved filtrate.

The solution is diluted to a standard volume to obtain stock solution (S1).

9.2.2.2 Mass of test portion to be weighed

Weigh 0,50 g of the test sample.

The sample used for the determination of loss on ignition may be used if it has not sintered.

9.2.2.3 Procedure

9.2.2.3.1 Weigh the dried sample into a platinum dish (e.g. 75 ml), add 4,0 g of anhydrous sodium carbonate and 2,7 g of boric acid and mix thoroughly. Cover it loosely with a platinum lid. Heat over a burner or in an electric furnace, slowly at first, then gradually raise the temperature to full heat. Finally, heat the crucible and contents in an electric furnace at 1 100 °C ± 25 °C for about 10 min until completely decomposed. Remove the crucible from the furnace and cover it with a platinum lid or a watch glass.

NOTE 1 If rapidly heated, the sample tends to scatter owing to the dehydration of boric acid.

NOTE 2 A long fusion time makes the dissolution of molten substance difficult in hydrochloric acid.

9.2.2.3.2 Remove the dish from the furnace and allow it to cool. Then add 5 ml of ethanol, 30 ml of hydrochloric acid (1+1) (5.1.17) and 2 ml of sulfuric acid (1+1) (5.1.35), cover with a lid and heat on a steam bath until the melt is completely dissolved. Remove the watch glass and wash it with water. Destroy the surface layer of salts and crush the precipitate into fine powder using a glass rod with a flat end. Heat until the mixture becomes syrup and then add 5 ml of hydrochloric acid (1+1) and 10 ml of polyethylene oxide solution. Mix thoroughly and allow to stand for 5 min. Filter with ashless filter paper into a 300 ml beaker and wash several times with hot hydrochloric acid (1+50). Wash with hot water until no chloride is detected. Keep the filtrate and washings in the beaker covered with the platinum lid or the watch glass.

9.2.2.3.3 Transfer the precipitate with the filter paper to a platinum crucible (30 ml) and add a drop of sulfuric acid (1+1). Heat over a burner or an electric furnace at low temperature until ashing of the filter paper is complete, then heat the crucible and its contents in an electric furnace at 1 100 °C ± 50 °C for 60 min. Cool in a desiccator and weigh (m_1). Moisten the content in the crucible with water, add 3 drops of sulfuric acid (1+1) and 10 ml of hydrofluoric acid and heat on a sand bath until dry. Heat at 1 100 °C ± 50 °C for 10 min, cool in a desiccator and weigh. Calculate the difference between the first mass (m_1) and second mass (m_2).

9.2.2.3.4 Add 1,0 g of anhydrous sodium carbonate and 0,3 g of boric acid to the residue in the crucible and fuse it by heating. After cooling, add 5 ml of hydrochloric acid (1+1), dissolve by heating and mix with the conserved filtrate. If necessary, condense the conserved filtrate in a 300 ml beaker, transfer to a 250 ml volumetric flask and dilute to the mark with water.

This solution is designated as stock solution (S1).

9.2.2.4 Blank test

Carry out the procedure given in 9.2.2.3 without the sample, but omit the fusion of the flux.

The resulting blank solution is designated (B1).

9.2.3 Decomposition by fusion and dissolution in sulfuric acid

9.2.3.1 Principle

The sample is fused with anhydrous sodium carbonate and boric acid. The melt is dissolved in sulfuric acid. The solution is diluted to the standard volume to obtain stock solution (S′1) of the sample.

9.2.3.2 Mass of test portion to be weighed

Weigh 0,50 g of the test sample.

The sample used for the loss on ignition may be used if it has not sintered.

9.2.3.3 Procedure

Weigh 0,50 g of the dried sample in a platinum crucible (75 ml), add 4,0 g of anhydrous sodium carbonate and 2,7 g of boric acid and mix thoroughly. Cover loosely with a platinum lid, if necessary. Heat over a burner or an electric furnace slowly at first and gradually raise the temperature to the full heat. Finally, heat the crucible and contents in an electric furnace at 1 100 °C ± 25 °C for about 10 min until completely decomposed. Remove the crucible from the furnace and cover it with a platinum lid or watch glass. Add 55 ml of sulfuric acid (1+9) and, while stirring, heat on a steam bath until the melt is completely dissolved. Remove the watch glass and wash it with a small amount of water. Transfer the resulting solution to a 250 ml volumetric flask and dilute to the mark with water.

This solution is referred to as stock solution (S′1).

9.2.3.4 Blank test

Carry out the procedure given in 9.2.3.3 without the sample, but omit the fusion of the flux.

The resulting blank solution is designated (B′1).

9.3 Dissolution methods by hydrofluoric acid attack

9.3.1 General

The following techniques are used:

a) decomposition and removal of the silica by hydrofluoric acid for flame photometry (see 9.3.2);

b) decomposition and removal of the silica by hydrofluoric acid for AAS (see 9.3.3);

c) decomposition and removal of the silica by hydrofluoric acid for ICP-AES (see 9.3.4).

9.3.2 Decomposition by hydrofluoric acid for flame photometry

9.3.2.1 Principle

A portion of the sample is decomposed using hydrofluoric acid, the remaining fluorides being removed by evaporation to dryness with sulfuric acid. The residue is dissolved in hydrochloric acid and the solution made up to volume. This becomes stock solution (S2).

9.3.2.2 Mass of test portion to be weighed

Weigh 0,20 g of the test sample.

9.3.2.3 Procedure

Weigh 0,20 g of the dried sample into a platinum dish (150 ml), moisten with water, add 3 ml of sulfuric acid (1+1), 2 ml of nitric acid, 10 ml of hydrofluoric acid and mix thoroughly. Carefully decompose the sample by heating it on a sand bath. Heat until dry and until white fumes appear from the sulfuric acid. After cooling, rinse the inner wall of the platinum dish with a small quantity of water. Add 2 ml of nitric acid, 5 ml of hydrofluoric acid and evaporate the solution until no more sulfuric acid fumes appear on a sand bath.

NOTE 1 For mixing the content in a platinum dish, a thick and head-bent platinum alloy wire (e.g. platinum-rhodium), a platinum spoon or an ethylene 4-fluoride resin rod/spoon may be used. During the decomposition process, violent reactions can cause scattering of the solution and care needs to be taken to avoid this. If bubbles are generated over the solution surface before white smoke appears from sulfuric acid, cover with a platinum lid or one made of ethylene 4-fluoride and remove from the sand bath after the smoke appears. After cooling, wash the platinum lid and the inner wall of the platinum dish with a small quantity of water and heat again.

After cooling, add 5,0 ml of hydrochloric acid (1+1) and 20 ml of water. Dissolve by heating on a steam bath and filter using a plastic funnel and ashless filter paper into a 100 ml plastic beaker. Wash thoroughly with hot water.

NOTE 2 The volatilization of hydrochloric acid occurs, so dissolution needs to be carried out as quickly as possible.

NOTE 3 Contamination of the solution by fine particles does not influence measurement.

After cooling, transfer the solution to a 100 ml plastic volumetric flask and dilute to the mark with water.

This solution is referred to as stock solution (S2).

9.3.2.4 Blank test

Carry out the procedure given in with 9.3.2.3 without the sample. The resulting blank solution is designated (B2).

9.3.3 Decomposition by hydrofluoric acid for AAS

9.3.3.1 Principle

A portion of the sample is decomposed using hydrofluoric acid, the remaining fluorides being removed by evaporation to dryness with sulfuric acid. The residue is dissolved in hydrochloric acid, lanthanum solution added and the solution made up to volume. This becomes stock solution (S3).

9.3.3.2 Mass of test portion

Weigh 0,20 g of the test sample.

9.3.3.3 Procedure

Weigh the sample into a platinum dish (e.g. 75 ml) and ignite gently over a burner to remove carbonaceous matter. Allow the dish to cool and moisten the residue with water, add 2 ml of sulfuric acid (1+1), 2 ml of nitric acid, and 10 ml of hydrofluoric acid. Transfer the dish and contents to a sand bath, cover with a lid and allow the reaction to proceed for about 15 min. Remove the lid, rinsing it with water into the dish and then evaporate to dryness, being careful to avoid spurting. After cooling, rinse the inner wall of the platinum dish with a small quantity of water. Then add 1 ml of sulfuric acid (1+1), 2 ml of nitric acid, and repeat the evaporation to dryness. After cooling, add 5,0 ml of hydrochloric acid (1+1) and approximately 20 ml of water and warm on a steam bath to dissolve. Filter, if necessary, using a filter paper (closed-pore), into a 200 ml beaker, washing thoroughly with hot water.

After cooling, add 10 ml of lanthanum solution, transfer to a 100 ml plastic volumetric flask and dilute to the mark with water.

This solution is designated as stock solution (S3).

9.3.3.4 Blank test

Carry out the procedure given in 9.3.3.3 without the sample.

The resulting blank solution is designated (B3).

9.3.4 Decomposition by hydrofluoric acid for ICP-AES

9.3.4.1 Principle

A portion of the sample is decomposed using hydrofluoric acid, the remaining fluorides being removed by evaporation to dryness with sulfuric acid. The residue is dissolved in hydrochloric acid and the solution made up to volume. This becomes stock solution (S4).

9.3.4.2 Mass of test portion to be weighed

Weigh 0,20 g of the test sample.

9.3.4.3 Procedure

Prepare a solution of the sample as in 9.3.3.3 but omitting the addition of 10 ml of lanthanum solution.

This solution is designated as stock solution (S4).

9.3.4.4 Blank test

Carry out the procedure given in 9.3.4.3 without the sample.

The resulting blank solution is designated (B4).

9.4 Separation methods by ion-exchange

9.4.1 General

The following techniques are used:

a) decomposition and separation of components by cation-exchange resin column for traditional methods (see 9.4.2);

b) decomposition and separation of components by resin solution for traditional methods (see 9.4.3).

9.4.2 Separation methods by cation-exchange resin column

9.4.2.1 Principle

A sample is fused with sodium carbonate and boric acid and it is dissolved in nitric acid. The acid concentration is adjusted and the solution is eluted on a column filling up cation-exchange resin and the resulting dichromic acid is eluted. Aluminium and titanium are eluted with hydrofluoric acid (1+150) and iron is eluted with hydrofluoric acid (1+20). After the column is washed with water, calcium and magnesium are eluted with hydrochloric acid (1+2). Perchloric acid and sulfuric acid are added to the eluate containing hydrofluoric acid and the solution is heated to remove hydrofluoric acid. The treated eluates and an eluate containing hydrochloric acid are used to determine each component.

The solutions are diluted to standard volumes as stock solution (SE-a), (SE-b) and (SE-c).

9.4.2.2 Procedure

Weigh 0,500 0 g of the sample, prepared in accordance with Clause 6, in a 75 ml platinum dish. Add 4,0 g of anhydrous sodium carbonate, 2,7 g of boric acid and fuse using the method given in 9.2.2.3.1. Cover with a watch glass and allow to cool. Add 60 ml of nitric acid (1+3) and heat on a steam bath while stirring with a glass rod until the melt has completely dissolved. Wash the watch glass and glass rod with water and remove. After cooling, transfer to a 250 ml volumetric flask and dilute with water to the mark.

This solution is designated as stock solution (S5).

Transfer precisely an aliquot portion of stock solution (see Note 1), pour it onto a column of cation-exchange resin and elute it from the column. Wash the funnel inside twice with 10 ml of nitric acid (1+20) and pour 60 ml of nitric acid (1+20) as eluent. Discard all the eluate at this point.

NOTE 1 The volume of the aliquot portion of stock solution depends on the mass fraction of magnesium oxide as shown in Table 2.

When the previous eluent has completely finished dropping from the end of the column, it is to be followed by the next eluent.

Table 2 — Aliquot portions taken from stock solution

Mass fraction of magnesium oxide %	**Aliquot portion of stock solution** ml
< 50	100
> 50	50
NOTE Lower volume results in reduced accuracy.	

Set a polyethylene tetrafluoride beaker under the column of the cation-exchange resin. Wash the funnel inside wall twice with 10 ml of hydrofluoric acid (1+150) and pour 70 ml of hydrofluoric acid (1+150) to elute aluminium and titanium species.

Set a polyethylene tetrafluoride beaker under the column of the cation-exchange resin. Wash the funnel inside wall twice with 10 ml hydrofluoric acid (1+20) and pour 70 ml of hydrofluoric acid (1+20) to elute iron species.

Set a 200 ml plastic beaker under the column of the cation-exchange resin. Wash the funnel inside wall twice with 10 ml of water and pour 80 ml of water to remove the hydrofluoric acid.

Set a 100 ml volumetric flask under the column of the cation-exchange resin. Wash the funnel inside the wall twice with 10 ml of hydrochloric acid (1+2) and pour 70 ml of hydrochloric acid (1+2) to elute manganese, calcium and magnesium species. Dilute the eluate to the mark with water. This solution is referred to as stock solution (SE-c).

Pour 70 ml of water onto the cation-exchange resin to regenerate a column.

NOTE 2 The regenerated column can immediately be used for the next separation. In the case where a column will not be used for some time, discard the top part of the resin (10 mm), take the remaining resin from the column with water and keep this resin in water.

Add 5 ml of sulfuric acid (1+1) and 1 ml of perchloric acid in a polyethylene tetrafluoride beaker as prepared in 4.1. Heat on a hot plate with sulfuric acid fume until the content has evaporated. After cooling, wash the inside of the beaker with a small amount of water and heat again with sulfuric acid fume until it has evaporated. After cooling, wash the inside of the beaker with a small amount of water and heat until it has evaporated. After cooling, add 5 ml of hydrochloric acid (1+1) and heat until dissolved. After cooling, transfer into a 100 ml volumetric flask and dilute to the mark with water. This solution is referred to as stock solution (SE-a).

Add 2 ml of sulfuric acid (1+1) and 1 ml of perchloric acid to a polyethylene tetrafluoride beaker as prepared in 4.1. Heat on a hotplate with sulfuric acid fume until the content has evaporated. After cooling, wash the inside of the beaker with a small amount of water and heat until it has evaporated. After cooling, add 5 ml of hydrochloric acid (1+1) and heat until dissolved. After cooling, transfer into a 100 ml volumetric flask and dilute to the mark with water. This solution is referred to as stock solution (SE-b).

9.4.2.3 Blank test

Carry out the procedure given in 9.4.2.2 without the sample, but omit the fusion with sodium carbonate. These solutions are referred to as blank solutions (BE-a), (BE-b) and (BE-c).

9.4.3 Separation methods by ion-exchange resin solution

9.4.3.1 Principle

A stock solution is prepared for aluminium oxide, iron(III) oxide, titanium oxide, manganese(II) oxide, calcium oxide and magnesium oxide by fusing 1 g of sample, dissolving in acid and extracting the chromium to an organic phase in a separating funnel.

The solutions are diluted to standard volumes as stock solution (S6).

9.4.3.2 Mass of test portion to be weighed

Weigh 0,500 0 g of the test sample.

Prepare a 1,000 g sample by grinding finely and drying at 110 °C.

9.4.3.3 Procedure

Put the sample into a platinum crucible. Add 8 g of fusion mixture and 4 g of boric acid and mix thoroughly. Cover the crucible with a lid, leaving a slight gap to allow ingress of air. Heat over a Bunsen or Meker flame and slowly raise the temperature until the mixture begins to melt and keep at this temperature until melting is complete.

IMPORTANT — It is important that this initial stage of heating should be slow, as a vigorous reaction takes place on melting, and spattering occurs easily.

Raise the temperature slowly and steadily to the full heat of the flame (approximately 950 °C). After 5 min at this temperature, swirl the contents of the crucible every 2 min making sure that the particles of the sample on the sides of the crucible come into contact with the hot molten flux.

NOTE If swirling is begun at too early a stage, it is difficult to detach unfused particles from the sides of the crucible.

Continue heating at this temperature for 45 min to 60 min, depending on the anticipated Al_2O_3 content, until the sample is fused. Check that decomposition is complete by visually checking that black unfused material at the bottom of the melt is absent. Add a further 1 g to 2 g of boric acid if any of the sample remains unfused after 60 min. Allow to cool and place the crucible in a porcelain basin to avoid loss of solution if the reaction on the addition of the acid is too vigorous. Raise the lid slightly and add 35 ml of dilute sulfuric acid (1+3). Replace the lid and allow the reaction to proceed until effervescence ceases and, if the melt does not decompose, complete the dissolution by warming. Transfer the solution from the crucible to a 400 ml beaker and dilute with water to 100 ml and cool.

Carry out the separation in diffuse light, and not bright sunlight, to minimize reduction of chromate. Transfer the cold solution to a 500 ml separating funnel with the minimum amount of water ensuring that the volume does not exceed 150 ml. Add 50 ml of resin solution (1+4). Stopper the funnel and shake vigorously for 1 min. Release the pressure in the funnel by carefully removing the stopper; rinse the stopper and neck of the funnel with water. Allow the layers to separate, then withdraw and discard the organic phase. Repeat the extraction with 20 ml of resin solution (1+4) and again withdraw the organic phase. Add 10 ml of dilute sulfuric acid (1+9) to the aqueous phase and repeat the extraction with a further 20 ml of resin solution (1+4), again discarding the organic phase. Remove the traces of resin by three successive extractions with 20 ml portions of chloroform. Transfer the aqueous phase and washings from the funnel to a beaker and boil the solution to remove traces of chloroform. Cool, dilute with water to 500 ml in a volumetric flask and mix.

This solution is referred to as stock solution (S6).

9.4.3.4 Blank test

Carry out the procedure given in 9.4.3.3 without the sample, but omit the fusion with sodium carbonate. These solutions are referred to as blank solution (B6).

10 Calculation and expression of test results

Calculate and express the results as given in ISO 26845.

11 Examination and adoption of test results

Examine and adopt the test results as given in ISO 26845.

12 Test report

Prepare a test report as described in ISO 26845.

Annex A
(informative)

References for stock solutions and blank solutions in ISO 20565-1:2008

Throughout ISO 20565:2008 (parts 2 and 3), reference is made to the various stock solutions and blank solutions prepared in this part of ISO 20565. Table A.1 gives a reference for each stock solution and blank solution to the subclause in this part of ISO 20565 where it is prepared.

Table A.1 — References to stock and blank solutions in ISO 20565-1:2008

Stock or blank solution	Subclause
S1	9.2.2.3
B1	9.2.2.4
S′1	9.2.3.3
B′1	9.2.3.4
S2	9.3.2.3
B2	9.3.2.4
S3	9.3.3.3
B3	9.3.3.4
S4	9.3.4.3
B4	9.3.4.4
SE-a, SE-b, SE-c	9.4.2.2
BE-a, BE-b, BE-c	9.4.2.3
S5	9.4.2.2
S6	9.4.3.3
B6	9.4.3.4

Bibliography

[1] ISO 12677:2003, *Chemical analysis of refractory products by XRF — Fused cast bead method*

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