BRITISH STANDARD

BS EN ISO 10058-1:2008

Chemical analysis of magnesite and dolomite refractory products (alternative to the X-ray fluorescence method)

Part 1: Apparatus, reagents, dissolution and determination of gravimetric silica (ISO 10058-1:2008)

ICS 73.080; 81.080

National foreword

This British Standard is the UK implementation of EN ISO 10058-1:2008. It partially supersedes BS EN ISO 10058:1996.

Together with BS EN ISO 10058-2 and BS EN ISO 10058-3, it supersedes BS EN ISO 10058:1996 which is withdrawn.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.

Compliance with a British Standard cannot confer immunity from legal obligations.

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Chemical analysis of magnesite and dolomite refractory products (alternative to the X-ray fluorescence method) - Part 1: Apparatus, reagents, dissolution and determination of gravimetric silica (ISO 10058-1:2008)

Analyse chimique des produits de magnésie et de dolomie (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 10058-1:2008)

Chemische Analyse von feuerfesten Erzeugnissen aus Magnesit und Dolomit (Alternative zur Röntgenfluoreszenzanalyse) - Teil 1: Geräte, Reagenzien, Aufschluss und gravimetrische Bestimmung von Silicium(IV)-oxid (ISO 10058-1:2008)

This European Standard was approved by CEN on 1 November 2008.

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Foreword

This document (EN ISO 10058-1:2008) 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of an identical text or by endorsement, at the latest by June 2009, and conflicting national standards shall be withdrawn at the latest by June 2009.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.

This document supersedes EN ISO 10058:1996.

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Endorsement notice

The text of ISO 10058-1:2008 has been approved by CEN as a EN ISO 10058-1:2008 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.

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ISO 10058-1 was prepared by Technical Committee ISO/TC 33, *Refractories*.

This first edition of ISO 10058-1, together with ISO 10058-2 and ISO 10058-3, cancels and replaces ISO 10058:1992 which has been technically revised to include the increasing use of flame atomic absorption spectrophotometry (FAAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES) methods as well some improvements in the wet chemical analyses procedures developed in Japan.

ISO 10058 consists of the following parts, under the general title *Chemical analysis of magnesite and dolomite refractory products (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 spectrophotometry (FAAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES)*

Chemical analysis of magnesite and dolomite refractory products (alternative to the X-ray fluorescence method) —

Part 1: Apparatus, reagents, dissolution and determination of gravimetric silica

1 Scope

This part of ISO 10058 specifies apparatus, reagents, dissolution and gravimetric silica analysis for the chemical analysis of magnesite and dolomite refractory products and raw materials.

This part of ISO 10058 is applicable to the ranges of determination given in Table 1.

ISO 10058 specifies procedures for the chemical analysis of magnesite and dolomite refractory products and raw materials within the ranges of determination given in Table 1.

Table 1 — Range of determination (percentage by mass)

Component	Range	Component	Range
SiO_2	0,1 to 10	MgO	30 to 99,9
Al_2O_3	0,05 to 10	Na_2O	0,01 to 1
Fe_2O_3	0,01 to 10	K_2O	0,01 to 1
TiO_2	0,01 to 1	Cr_2O_3	0,01 to 3
MnO	0,01 to 1	ZrO_2	0,01 to 1
CaO	0,01 to 60	P_2O_5	0,01 to 5
LOI	0,01 to 60	—	—
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

Normal laboratory apparatus and other apparatus as defined in ISO 26845.

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.

Use reagents that are defined in ISO 26845 and the following.

5.1 Standard volumetric solutions.

5.1.1 Standard volumetric CyDTA solution, $c(\text{CyDTA}) = 0{,}01$ mol/l.

Add 8 ml of sodium hydroxide solution (100 g/l) and approximately 150 ml of water to 3,65 g of trans-1,2-cyclohexanediamine -*N*,*N*,*N*′,*N*′ -tetraacetic acid monohydrate (CyDTA), and dissolve by heating. After cooling, dilute to 1 000 ml with water; approximately 25 ml of water are required.

Determine the exact strength of this solution by titration against the standard volumetric zinc solution, $c(\text{Zn}) = 0{,}01$ mol/l.

5.1.2 Standard volumetric zinc solution, $c(\text{Zn}) = 0{,}01$ mol/l.

Wash the surface of the zinc (purity greater than 99,9 % by mass) with hydrochloric acid (1+3) and dissolve the oxidized layer; subsequently, wash with water, ethanol and diethyl ether in succession, then dry in a desiccator. Weigh 0,65 g (recorded to the nearest 0,000 1 g) of zinc and transfer it to a 300 ml beaker. Cover with a watch glass. Add 20 ml of water and 5 ml of nitric acid carefully, and heat to dissolve on a steam bath. After cooling, dilute to 1 000 ml in a volumetric flask with water.

Calculate the factor, F, of this zinc solution using Equation (1).

$$F = \frac{m}{0{,}653\,8} \times \frac{A}{100} \tag{1}$$

where

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

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

5.1.3 Standard volumetric EDTA solution, $c(\text{EDTA}) = 0{,}02$ mol/l.

Dissolve 7,5 g of EDTA_2Na (ethylenediamine-tetraacetic acid disodium salt dihydrate) in 1 l of water. Store in a plastic bottle.

Determine the factor, F, of the 0,02 mol/l standard volumetric EDTA solution as follows.

Transfer 50 ml of 0,01 mol/l zinc solution, obtained in 5.1.2, 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) and, while stirring, add 3 to 4 drops of Eriochrome Black T solution as an indicator and titrate with 0,02 mol/l standard volumetric EDTA solution. Observe and record the end point, which is when the reddish purple colour of the solution is altered to blue.

Calculate the factor, F, of the 0,02 mol/l standard volumetric EDTA solution using Equation (2).

$$F = \frac{f \times 50}{2 \times V} \qquad (2)$$

where

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

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

5.2 Standard solutions.

5.2.1 Standard aluminium oxide solution, Al_2O_3 1 mg/ml.

Wash the surface of a sufficient amount of aluminium metal (approximately 0,6 g to 0,7 g), purity of more 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 aluminium and transfer it to a 250 ml beaker. Cover the beaker with a watch glass, add 20 ml hydrochloric acid (1+1), and heat to dissolve. After cooling, dilute to 1 000 ml with water in a volumetric flask.

5.2.2 Standard calcium oxide solution, CaO 1 mg/ml.

Dissolve 1,785 g of pure calcium carbonate that has been previously dried at 150 °C for 2 h, 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 with water in a volumetric flask.

5.2.3 Standard chromium(III) oxide solution, Cr_2O_3 1 mg/ml.

Dry between 2 g to 3 g of potassium dichromate at 110 °C for at least 2 h. Weigh 1,935 5 g of this and dissolve in water. Dilute to 1 000 ml with water in a volumetric flask.

5.2.4 Dilute standard chromium(III) oxide solution, Cr_2O_3 0,025 mg/ml.

Pipette 25 ml of the standard chromic oxide solution (1 mg/ml) to a 1 000 ml volumetric flask and dilute to the mark with water. Prepare this solution freshly when required.

5.2.5 Standard iron(III) oxide solution, Fe_2O_3 1 mg/ml.

Wash the surface of a sufficient amount of iron metal (purity greater than 99,9 % by mass) with hydrochloric acid (1+4). Then dissolve the oxidized layer, wash with water, ethanol, and diethyl ether in succession. Dry in a desiccator. Weigh 0,699 4 g of this, transfer to a beaker (200 ml) and cover with a watch glass. Add 40 ml of hydrochloric acid (1+1) and heat on a steam bath until dissolved. After cooling, dilute to 1 000 ml with water in a volumetric flask.

5.2.6 Dilute standard iron(III) oxide solution, Fe_2O_3 0,2 mg/ml.

Pipette 200 ml of the standard iron(III) oxide solution (Fe_2O_3 1 mg/ml) into a 1 000 ml volumetric flask and dilute to the mark with water. Prepare this solution freshly when required.

5.2.7 **Dilute standard iron(III) oxide solution**, Fe_2O_3 0,04 mg/ml.

Pipette 40 ml of the standard iron(III) oxide solution (Fe_2O_3 1 mg/ml) into a 1 000 ml volumetric flask and dilute to the mark with water. Prepare this solution freshly when required.

5.2.8 **Standard magnesium oxide 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. 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 20 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.

5.2.9 **Standard manganese(II) oxide solution**, MnO 1 mg/ml.

Wash the surface of a sufficient mass of manganese metal (purity greater than 99,9 % by mass) with hydrochloric acid (1+3) to dissolve the oxidized layer. Then wash with water, ethanol, and diethyl ether in succession and dry in a desiccator. Weigh 0,774 5 g of this metal, transfer to a 200 ml beaker and cover with a watch glass. Add 40 ml of nitric acid (1+1) and heat to dissolve; after cooling, transfer to a 1 000 ml volumetric flask.

5.2.10 **Dilute standard manganese(II) oxide solution**, MnO 0,04 mg/ml.

Transfer 40 ml of the standard manganese(II) oxide solution (MnO 1 mg/ml) to a 1 000 ml volumetric flask and dilute to the mark with water. Prepare this solution freshly as required.

5.2.11 **Standard phosphorus(V) oxide solution**, P_2O_5 1 mg/ml.

Heat about 3 g of potassium dihydrogen phosphate at 110 °C ± 5 °C for 3 h, and allow to cool in a desiccator. Weigh 1,917 6 g, transfer to a beaker and dissolve with approximately 300 ml of water. Dilute to 1 000 ml with water in a volumetric flask.

5.2.12 **Dilute standard phosphorus(V) oxide solution**, P_2O_5 0,04 mg/ml.

Transfer precisely 40 ml of the standard phosphorus(V) oxide solution (5.2.11) into a 1 000 ml volumetric flask and dilute to the mark with water.

5.2.13 **Dilute standard phosphorus(V) oxide solution**, P_2O_5 0,01 mg/ml.

Pipette 10 ml of the standard phosphorus(V) oxide solution (5.2.11) into a 1 000 ml volumetric flask and dilute to the mark with water. Prepare this solution freshly when required.

5.2.14 **Standard potassium oxide solution**, K_2O 1 mg/ml.

Transfer 1 g to 1,5 g of potassium chloride into a platinum crucible (e.g. 30 ml) and ignite at 600 °C ± 25 °C for approximately 60 min. Allow the crucible and contents to cool in a desiccator. Weigh 0,791 4 g of this and transfer to a 200 ml beaker. Dissolve in 100 ml of water, transfer to a 500 ml volumetric flask, and dilute to the mark with water.

5.2.15 **Standard silicon(IV) oxide solution**, SiO_2 1 mg/ml.

Weigh 1,5 g to 2 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 1,000 g of this silicon(IV) oxide into a platinum crucible (e.g. 50 ml). Fuse the silicon(IV) oxide with 5,0 g of anhydrous sodium carbonate. Cool and wipe the outside of the crucible, and dissolve in warm water (150 ml) in a plastic 200 ml beaker while stirring using a plastic rod. Cool and dilute without heating to 1 000 ml in a volumetric flask. Transfer this solution to a plastics bottle immediately.

5.2.16 Dilute standard silicon(IV) oxide solution, SiO_2 0,08 mg/ml.

Pipette 40 ml of the standard silicon(IV) oxide solution (5.2.15) into a 500 ml volumetric flask and dilute to the mark with water. Prepare this solution freshly when required.

5.2.17 Dilute standard silicon(IV) oxide solution, SiO_2 0,04 mg/ml.

Pipette 20 ml of the standard silicon(IV) oxide solution (5.2.15) into a 500 ml volumetric flask and dilute to the mark with water. Prepare this solution freshly when required.

5.2.18 Standard sodium oxide solution, Na_2O 1 mg/ml.

Transfer 1 g to 1,5 g of sodium chloride into a platinum crucible (e.g. 30 ml) and ignite at 600 °C ± 25 °C for approximately 60 min. Allow the crucible and contents to cool in a desiccator. Weigh 0,942 9 g of this and transfer to a 200 ml beaker. Dissolve in 100 ml of water, transfer to a 500 ml volumetric flask, and dilute to the mark with water.

5.2.19 Standard titanium(IV) oxide solution, TiO_2 1 mg/ml.

Wash the surface of a sufficient amount of titanium metal (purity greater than 99,9 % by mass) with hydrochloric acid (1+3) and dissolve the oxidized layer. Subsequently, wash with water, ethanol and diethyl ether in succession, then dry in a desiccator. Weigh 0,599 4 g of this titanium metal, and transfer to a platinum dish. Cover with a watch glass made of ethylene 4-fluoride resin, then add 40 ml of hydrofluoric, 15 ml of sulfuric (1+1) and 2 ml nitric acid, and heat to dissolve on a steam bath. Remove the watch glass, rinse the watch glass with water and heat the solution on a sand bath until the appearance of strong sulfuric acid fumes. After cooling, rinse the inner wall of the platinum dish with a small amount of water and heat again until fumes are seen. After cooling, dilute to 1 000 ml with water in a volumetric flask.

5.2.20 Dilute standard titanium(IV) oxide solution, TiO_2 0,01 mg/ml.

Pipette 10 ml of the standard titanium(IV) oxide solution (5.2.19) into a 1 000 ml volumetric flask and dilute to the mark with water. Prepare this solution freshly when required.

5.2.21 Standard zirconium oxide solution, ZrO_2 1 mg/ml.

Transfer about 0,3 g of zirconium oxide (purity greater than 99,9 % by mass) into a platinum crucible (e.g. 30 ml), heat strongly at 1 150 °C ± 50 °C for approximately 30 min, and allow to cool in a desiccator. Weigh 0,200 0 g of this into a platinum crucible (e.g. 30 ml), add 4 g of potassium disulfate and fuse, covering with a platinum lid.

Fuse over a gas burner at as low a temperature as possible, otherwise sulfur trioxide will be lost before the attack is complete.

After cooling, put the platinum crucible into a 200 ml beaker, add 100 ml of sulfuric acid (1+9), and warm to dissolve. After cooling, remove and wash the platinum crucible with water, transfer to a 200 ml volumetric flask and dilute to the mark with water.

5.2.22 Dilute standard zirconium oxide solution, ZrO_2 0,005 mg/ml.

Pipette 5 ml of the standard zirconium oxide solution (5.2.21) into a 1 000 ml volumetric flask and dilute to the mark with water. Prepare this solution freshly when required.

5.2.23 Standard scandium solution, Sc 1,0 mg/ml.

Dry about 2,0 g of scandium oxide at 110 °C ± 5 °C for 60 min and cool in a desiccator. Weigh 1,533 8 g of this and transfer it to a 600 ml beaker. Gradually add 100 ml of hydrochloric acid (1+1) to dissolve and dilute precisely to 1 000 ml with water in a volumetric flask.

A suitable commercial standard solution may be used.

5.2.24 Standard yttrium solution, Y 1,0 mg/ml.

Dry about 2,0 g of yttrium oxide at 110 °C ± 5 °C for 60 min and cool in a desiccator. Weigh 1,27 g of this and transfer it to a 600 ml beaker. Dissolve by gradually adding 100 ml of hydrochloric acid (1+1) and dilute precisely to 1 000 ml with water in a volumetric flask.

A suitable commercial standard solution may be used.

6 Sample preparation

Prepare the sample as described in ISO 26845:2008, Clause 7.

7 Determination of loss on ignition (gravimetric)

Determine the loss on ignition in accordance with ISO 26845:2008, Clause 9.

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

8.1 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 8.2.2);
- decomposition by fusion and dissolution in sulfuric acid (see 8.2.3);
- decomposition and removal of the silica by hydrofluoric acid for flame photometry (see 8.3.2);
- decomposition and removal of the silica by hydrofluoric acid for AAS (see 8.3.3);
- decomposition and removal of the silica by hydrofluoric acid for ICP-AES (see 8.3.4).

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

8.2 Dissolution methods by fusion

8.2.1 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 (see 8.2.2);

b) fusion with alkali carbonate and boric acid, and dissolution of the melt in sulfuric acid (see 8.2.3).

8.2.2 Preparing a solution by fusion and coagulation

8.2.2.1 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).

8.2.2.2 Mass of the test portion

Weigh 0,50 g of the test sample.

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

8.2.2.3 Procedure

8.2.2.3.1 Weigh the dried sample into a platinum dish (e.g. 75 ml), add 3,0 g of anhydrous sodium carbonate and 1,0 g of boric acid and mix thoroughly and intimately. Cover it loosely with a platinum lid. Heat over a burner or an electric furnace slowly, then gradually raise the temperature to the full heat. Finally, heat the crucible in an electric furnace at 1 100 °C ± 25 °C for about 10 min. 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.

8.2.2.3.2 Remove the dish from the furnace and allow it to cool. Then add 5 ml of ethanol, 30 ml of hydrochloric acid (1+1) and 2 ml of sulfuric acid (1+1), 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 viscous 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.

8.2.2.3.3 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 and then heat the crucible and its contents in an electric furnace at 1 100 °C ± 50 °C for 60 min. Cool in a desiccator, weigh and record as 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, weigh and record as m_2. Calculate the difference between m_1 and m_2.

8.2.2.3.4 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 (8.2.2.3.2) 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).

8.2.2.4 Blank test

Carry out the procedure given in 8.2.2.3 without the sample, but omit the fusion of the flux.

Designate the resulting solution as blank solution (B1).

8.2.3 Decomposition by fusion and dissolution in sulfuric acid

8.2.3.1 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.

8.2.3.2 Mass of test portion

Weigh 0,50 g of the test sample.

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

8.2.3.3 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. 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 designated as stock solution (S′1).

8.2.3.4 Blank test

Carry out the procedure given in 8.2.3.3 without the sample, but omit the fusion of the flux.

Designate the resulting solution as blank solution (B′1).

8.3 Dissolution methods by hydrofluoric acid attack

8.3.1 General

One of the following techniques is used:

a) decomposition and removal of the silica by hydrofluoric acid for flame photometry (see 8.3.2);

b) decomposition and removal of the silica by hydrofluoric acid for AAS (see 8.3.3);

c) decomposition and removal of the silica by hydrofluoric acid for ICP-AES (see 8.3.4).

8.3.2 Decomposition by hydrofluoric acid for flame photometry

8.3.2.1 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 solution is designated as stock solution (S2).

8.3.2.2 Mass of test portion

Weigh 0,20 g of the test sample.

8.3.2.3 Procedure

8.3.2.3.1 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 carry out decomposition by heating 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 on a sand bath until no more sulfuric acid fumes appear.

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 a lid made of ethylene 4-fluoride resin 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.

8.3.2.3.2 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 200 ml plastic beaker. Wash thoroughly with hot water.

To minimize the volatilization of hydrochloric acid, carry out the dissolution as quickly as possible.

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

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

This solution is designated as stock solution (S2).

8.3.2.4 Blank test

Carry out the procedure given in with 8.3.2.3 without the sample.

Designate the resulting solution as blank solution (B2).

8.3.3 Decomposition by hydrofluoric acid for AAS

8.3.3.1 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 is added and the solution is made up to volume.

This solution is designated as stock solution (S3).

8.3.3.2 Mass of test portion

Weigh 0,20 g of the test sample.

8.3.3.3 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 close pore filter paper into a 200 ml beaker, washing thoroughly with hot water.

After cooling, add 10 ml of La_2O_3 (50 g/l ISO 26845:2008, 5.1.50) 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).

8.3.3.4 Blank test

Carry out the procedure given in 8.3.3.3 without the sample.

Designate the resulting solution as blank solution (B3).

8.3.4 Decomposition by hydrofluoric acid for ICP-AES

8.3.4.1 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 solution is designated as stock solution (S4).

8.3.4.2 Mass of test portion

Weigh 0,20 g of the test sample.

8.3.4.3 Procedure

Prepare a solution of the sample as in 8.3.3.3 but omitting the addition of 10 ml of lanthanum solution.

This solution is designated as stock solution (S4).

8.3.4.4 Blank test

Carry out the procedure given in 8.3.4.3 without the sample.

Designate the resulting solution as blank solution (B4).

9 Calculation and expression of test results

Calculate and express the test results in accordance with ISO 26845.

10 Examination and adoption of test results

The maximum permissible difference in test values shall be in accordance with ISO 26845 and Table 2.

Table 2 — Permissible difference in test values

Mass fraction	**Component**													
%	LOI	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	MnO	CaO	MgO	Na_2O	K_2O	Cr_2O_3	ZrO_2	P_2O_5	B_2O_3
Under 0,1	0,02	0,02	0,02	0,02	0,01	0,01	0,02	—	0,02	0,02	0,01	0,02	0,02	0,02
~0,2	0,05	0,05	0,05	0,05	0,02	0,02	0,05	—	0,05	0,05	0,02	0,02	0,03	0,03
~0,5	0,05	0,05	0,05	0,05	0,03	0,05	0,05	—	0,05	0,05	0,03	0,03	0,05	0,05
~2	0,05	0,05	0,05	0,05	0,04	0,05	0,05	—	0,05	0,05	0,05	0,05	0,05	0,05
~5	0,10	0,10	0,10	0,10	—	—	0,10	—	—	—	0,10	—	0,10	—
~10	0,20	0,20	0,20	0,20	—	—	0,20	—	—	—	—	—	—	—
~20	0,2	—	—	—	—	—	0,2	—	—	—	—	—	—	—
~50	0,2	—	—	—	—	—	0,2	0,2	—	—	—	—	—	—
Over 50	0,2	—	—	—	—	—	0,2	0,2	—	—	—	—	—	—

11 Test report

Prepare a test report as described in ISO 26845.

Annex A
(informative)

References for stock solutions and blank solutions in ISO 10058-1:2008

Throughout ISO 10058-2:2008 and ISO 10058-3:2008, reference is made to the various stock solutions and blank solutions prepared in this part of ISO 10058. Table A.1 gives a reference, for each stock solution and blank solution, to the subclause in this part of ISO 10058 where it is prepared.

Table A.1 — References to stock and blank solutions in ISO 10058-1:2008

Solution	Subclause
S1	8.2.2.3
B1	8.2.2.4
S′1	8.2.3.3
B′1	8.2.3.4
S2	8.3.2.3
B2	8.3.2.4
S3	8.3.3.3
B3	8.3.3.4
S4	8.3.4.3
B4	8.3.4.4

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