INTERNATIONAL STANDARD

ISO 21079-1

First edition
2008-04-15

Chemical analysis of refractories containing alumina, zirconia and silica — Refractories containing 5 % to 45 % of ZrO_2 (alternative to the X-ray fluorescence method) —

Part 1: Apparatus, reagents and dissolution

Analyse chimique des matériaux réfractaires contenant de l'alumine, de la zircone et de la silice — Matériaux réfractaires contenant de 5 % à 45 % de ZrO_2 *(méthode alternative à la méthode par fluorescence de rayons X) —*

Partie 1: Appareillage, réactifs et dissolution

Reference number
ISO 21079-1:2008(E)

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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 21079-1 was prepared by Technical Committee ISO/TC 33, *Refractories*.

ISO 21079 consists of the following parts, under the general title *Chemical analysis of refractories containing alumina, zirconia and silica — Refractories containing 5 % to 45 % of* ZrO_2 *(alternative to the X-ray fluorescence method)*:

— *Part 1: Apparatus, reagents and dissolution*

— *Part 2: Wet chemical analysis*

— *Part 3: Flame atomic absorption spectrophotometry (FAAS) and inductively coupled plasma emission spectrometry (ICP-AES)*

INTERNATIONAL STANDARD **ISO 21079-1:2008(E)**

Chemical analysis of refractories containing alumina, zirconia and silica — Refractories containing 5 % to 45 % of ZrO_2 (alternative to the X-ray fluorescence method) —

Part 1:
Apparatus, reagents and dissolution

1 Scope

This part of ISO 21079 specifies methods for the chemical analysis of AZS (alumina, zirconia, and silica) refractory products (containing 5 % to 45 % of ZrO_2) and raw materials, using traditional ("wet") methods, inductively coupled plasma atomic emission (ICP-AE) spectrometry and flame atomic absorption (FAA) spectrometry. It covers apparatus, reagents and dissolution methods.

The range of determination is given in Table 1.

Table 1 — Range of determination (% by mass)

Component	Range	Component	Range
LOI	–1 to 40	MgO	0,01 to 2
SiO_2	0,1 to 45	Na_2O	0,01 to 3
Al_2O_3	1 to 80	K_2O	0,01 to 1
Fe_2O_3	0,01 to 2	Cr_2O_3	0,01 to 3
TiO_2	0,01 to 5	ZrO_2	5 to 45
CaO	0,01 to 2	HfO_2	0,01 to 2

NOTE The values other than LOI are on an ignited basis.

This part of ISO 21079 is not applicable to MgO-based refractories.

NOTE This part of ISO 21079 gives alternatives to the X-ray fluorescence (XRF) method given in ISO 12677.

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

3 Terms and definitions

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

4 Apparatus

Ordinary laboratory apparatus and the apparatus given in ISO 26845.

5 Reagents

5.1 General

Prepare the following reagents, and those given in ISO 26845, as necessary.

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

Reagents should conform to the requirements of ISO 6353-1, ISO 6353-2 and ISO 6353-3, as appropriate.

5.2 Standard solutions specific to ISO 21079

Traceable standard solutions may be used in place of the following.

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

Wash the surface of a sufficient amount of aluminium metal (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 into a 250 ml beaker. Cover with a watch glass, add 20 ml of hydrochloric acid (1+1), and heat to dissolve. After cooling, dilute to 1 000 ml in a volumetric flask with water.

5.2.2 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 dilute 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.

5.2.3 Chromium(III) oxide standard solution, Cr_2O_3 1 mg/ml.

Dry about 2 g to 3 g of potassium dichromate, $K_2Cr_2O_7$, 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.

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

Dilute 25 ml of the chromium(III) oxide standard solution (1 mg/ml), to 1 000 ml in a volumetric flask. Prepare this solution freshly when required.

5.2.5 CyDTA (trans-1,2-cyclohexanediamine-*N,N,N',N'*-tetraacetic acid monohydrate) standard solution, 0,02 mol/l.

Add 16 ml of sodium hydroxide solution (100 g/l) and 150 ml of water to 7,30 g of CyDTA, and dissolve by heating. After cooling, dilute to 1 000 ml with water.

5.2.6 EDTA standard solution, 5 g/l.

Dissolve 5 g of EDTANa$_2$ (ethylenediamine-tetraacetic acid disodium salt, dihydrate) in water and dilute to 1 000 ml in a volumetric flask. Store in a plastics bottle.

Standardize against calcium as follows.

Pipette 25 ml of standard calcium solution (1 mg/ml) into a 500 ml conical flask, add 10 ml of potassium hydroxide solution, and dilute to about 200 ml. Add about 0,015 g of screened calcein indicator and titrate with the EDTA solution (5 g/l), from a fluorescent green colour to pink.

Standardize against magnesium as follows.

Pipette 25 ml of standard magnesium solution (1 mg/ml), into a 500 ml conical flask. Add 20 drops of hydrochloric acid (concentrated) and 20 ml of ammonia solution (concentrated), and dilute to about 200 ml. Add about 0,04 g of methylthymol blue complexone indicator and titrate with the EDTA solution (5 g/l).

5.2.7 Hafnium oxide standard solution, HfO_2 1 mg/ml.

Transfer 1,5 g of hafnium oxide (of a purity more than 99,9 % by mass) into a 20 ml platinum crucible, ignite at 1 050 °C ± 25 °C for 1 h and cool in a desiccator. Weigh 0,500 0 g of the ignited residue into a 75 ml platinum crucible. After adding 3,0 g of anhydrous sodium carbonate, add 2,0 g of boric acid and mix.Then carry out melting at 1 050 ± 25 °C in an electric muffle furnace. After cooling, add 55 ml of sulfuric acid (1+9) and heat until dissolved on a steam bath. After cooling, dilute to 500 ml in a flask with water.

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

Wash the surface of a sufficient amount of iron metal (purity greater than 99,9 %) with hydrochloric acid (1+4). Then dissolve the oxidized layer, and wash with water, ethanol and diethyl ether in succession. Then dry in a desiccator. Weigh 0,699 4 g of this, transfer to a beaker (200 ml), 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.

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

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

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

Wash the surface of a sufficient amount of magnesium metal (purity, more 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.

5.2.11 Potassium oxide standard 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.

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

Weigh 0,7 g to 1,0 g of silicon(IV) oxide (purity, greater that 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 plastics 200 ml 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.

5.2.13 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.

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

Transfer 1 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.

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

Weigh 0,599 4 g of titanium (of a purity more 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.

5.2.16 Diluted titanium(IV) oxide standard solution, TiO_2 0,01 mg/ml.

Dilute 10 ml of titanium(IV) oxide standard solution (1,0 mg/ml) to 1 000 ml with water.

5.2.17 Zinc standard solution, Zn 0,02 mol/l.

Wash the surface of the zinc (of a purity more 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 (1,307 4 ± 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 1 000 ml in a volumetric flask with water.

Calculate the factor, F, of the 0,02 mol/l zinc solution using Equation (1).

$$F = \frac{m}{0{,}653\,7} \times \frac{A}{100} \tag{1}$$

where

m is the mass of the weighed zinc, in grams;

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

6 Sampling

Prepare the sample as described in ISO 26845.

7 General procedures

Carry out the chemical analysis and a blank test as specified in ISO 26845.

8 Determination of loss on ignition

Determine the loss on ignition as specified in ISO 26845.

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

9.1 General decomposition techniques

The techniques used in this standard method are traditional "wet" methods, flame photometry, atomic absorption spectrometry and ICP-AE spectrometry. To meet the needs of these procedures five separate decompositions are required.

Carry out one or more of the decompositions given in the following list, depending on the requirements of the particular technique(s) chosen:

a) decomposition by fusion followed by removal of the bulk of the silica present by dehydration, as described in 9.2.2;

b) decomposition by fusion followed by removal of the bulk of the silica present by coagulation, as described in 9.2.3;

c) decomposition by fusion and dissolution in sulfuric acid, as described in 9.2.4;

d) decomposition and removal of the silica by hydrofluoric acid for flame photometry, as described in 9.3.2;

e) decomposition and removal of the silica by hydrofluoric acid for atomic absorption spectroscopy, as described in 9.3.3;

f) decomposition and removal of the silica by hydrofluoric acid for ICP-AE spectrometry, as described in 9.3.4.

9.2 Dissolution methods by fusion

9.2.1 General

Prepare a solution for the determination of silicon(IV) oxide in solution, aluminium oxide, ferric oxide, titanium oxide, calcium oxide, magnesium oxide, chromium(III) oxide, zirconium oxide, and hafnium oxide by one of the following 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 dehydration;

b) 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;

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

9.2.2 Preparing a solution by fusion and dehydration

9.2.2.1 Principle

The sample is fused with anhydrous sodium carbonate and boric acid. The melt is dissolved in sulfuric acid and evaporated until dry for the dehydration of silicic acid. Soluble salts are dissolved in hot water and filtered. The precipitate is ignited and fused with anhydrous sodium carbonate to reduce boron contamination. The melt is dissolved in sulfuric acids, and dehydration and filtration are carried out. The precipitate is ignited and weighed both before and after treatment with hydrofluoric and sulfuric acids. The difference in mass gives the main or gravimetric silicon(IV) oxide content and this result needs to be added to that for residual silica in solution to give the figure for the total silicon(IV) oxide content. The solution is diluted to the standard volume to obtain a stock solution of the sample.

9.2.2.2 Mass of the test portion

Weigh 0,50 g of the test sample.

9.2.2.3 Procedure

9.2.2.3.1 Weigh 0,50 g of the sample, prepared as described in Clause 6, in a platinum dish, add 3,0 g of anhydrous sodium carbonate and 2,0 g of boric acid, and mix thoroughly. Heat over a burner or an electric muffle furnace cautiously at first, then gradually raise the temperature to the full heat of the burner or electric muffle furnace. Heat the dish and contents in the electric muffle furnace at 1 150 °C ± 50 °C until completely decomposed. Remove the dish from the electric muffle furnace, cover with a watch glass and allow to cool. Add 55 ml of sulfuric acid (1+15) and heat on a steam bath until the melt is completely decomposed.

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

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

9.2.2.3.2 Evaporate the solution obtained in 9.2.2.3.1 until dry, while occasionally breaking up the crust of salt using the flattened end of a glass rod. Drench the dry residue, add 30 ml of hot water and digest on a steam bath for 5 min to dissolve the salts. Filter the precipitate through medium filter paper into a beaker and wash several times with hot sulfuric acid (1+150) and then with hot water until free from chlorides. Reserve the filtrate and washings.

9.2.2.3.3 Transfer the paper and precipitate to a platinum dish and moisten with 1 drop of sulfuric acid (1+1). Ignite at a low temperature until the precipitate is free from carbonaceous matter and then heat in a electric muffle furnace at 1 100 °C ± 50 °C for 10 min. Fuse the residue in 3,0 g of anhydrous sodium carbonate and dissolve the melt in 55 ml of sulfuric acid (1+15). Repeat the dehydration and filtration procedure given in 9.2.2.3.2. Add the filtrate and washings to the reserved solution, obtained as described in 9.2.2.3.2.

9.2.2.3.4 Transfer the paper and precipitate to a platinum crucible and moisten with 1 drop of sulfuric acid (1+1). Ignite at a low temperature until the precipitate is free from carbonaceous matter and then heat in an electric muffle furnace at 1 100 °C ± 50 °C for 60 min. Allow to cool and weigh as the impure silica.

9.2.2.3.5 Moisten the residue with 3 drops of sulfuric acid (1+1) and 10 ml of hydrofluoric acid. Evaporate slowly until dry on a sand bath in a fume cupboard. Ignite the dry residue at 1 100 °C ± 25 °C for 5 min, allow to cool and weigh.

NOTE The difference between the two masses represents the gravimetric silica.

9.2.2.3.6 Fuse the residue in 1 g of anhydrous sodium carbonate and 0,3 g of boric acid, and dissolve the melt in the reserved solution obtained in 9.2.2.3.3. Allow to cool and dilute the solution to 500 ml in a volumetric flask and mix.

Designate this solution 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.

Designate the resulting solution as blank solution (B1).

9.2.3 Preparing a solution by fusion and coagulation

9.2.3.1 Principle

The sample is fused in anhydrous sodium carbonate and boric acid and the melt is dissolved in sulfuric 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 residue is fused in anhydrous sodium carbonate and boric acid and the melt is dissolved in sulfuric acid. Coagulation and filtration is repeated and 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 (S’1).

NOTE Because this procedure results in smaller residues after hydrofluoric acid treatment, it reduces the risk of overall errors created by errors in the determination of components in the residue.

9.2.3.2 Mass of test portion

Weigh 0,50 g of the test sample.

9.2.3.3 Procedure

9.2.3.3.1 Weigh 0,5 g of the sample, prepared as described in Clause 6, into a platinum dish, add 3,0 g of anhydrous sodium carbonate and 2,0 g of boric acid and mix thoroughly. Heat over a burner cautiously at first, then gradually raise the temperature to the full heat of the burner. Heat in the electric muffle furnace at 1 100 °C ± 25 °C until completely decomposed. Remove from the electric muffle furnace, cover with a watch glass and allow to cool. Add 55 ml of sulfuric acid (1+15) and transfer to a steam bath to facilitate the dissolution of the melt. When the solution is complete, remove the watch glass and rinse it with a minimum amount of water.

9.2.3.3.2 Allow the solution to remain on the steam bath until a stiff gel is formed. Add macerated filter paper to the gel and mix thoroughly. Slowly add 10 ml of the polyethylene oxide solution while stirring, mix thoroughly and allow to stand for 5 min. Filter the precipitate through an ashless filter paper (close) with hot sulfuric acid (1+150) and scrub with a rubber-tipped glass rod. Wash the precipitate with hot water until free from chloride. Reserve the filtrate and washings.

9.2.3.3.3 Transfer the paper and precipitate to a platinum dish and add 1 drop of sulfuric acid (1+1). Ignite at a low temperature until the precipitate is free from carbonaceous matter, and then heat in an electric muffle furnace at 1 100 °C ± 25 °C for 10 min. Allow to cool, add 3,0 g of anhydrous sodium carbonate and 0,1 g of boric acid and mix thoroughly. Heat over a burner cautiously at first, then raise the temperature to the full heat of the burner. Heat in the electric muffle furnace at 1 100 °C ± 25 °C for 5 min. Remove from the electric muffle furnace, cover with a watch glass and allow to cool. Add 55 ml of sulfuric acid (1+15) and transfer the dish to a steam bath to facilitate the dissolution of the melt. Repeat the coagulation and filtration as described in 9.2.3.3.2. Reserve the filtrate and washings.

9.2.3.3.4 Transfer the paper and precipitate to a platinum crucible and add 1 drop of sulfuric acid (1+1). Ignite at a low temperature until the precipitate is free from carbonaceous matter and then heat in an electric muffle furnace at 1 100 °C ± 25 °C for 60 min. Allow to cool in a desiccator and weigh. Moisten the contents of the crucible with water, and add 3 drops of sulfuric acid (1+1) and 10 ml of hydrofluoric acid. Evaporate until dry on a sand bath in a fume cupboard. Heat the crucible and residue, cautiously at first over a gas flame and finally at 1 100 °C ± 25 °C. Cool in a desiccator and weigh. The difference between the two masses is the gravimetric silica. Fuse the residue from the hydrofluoric acid treatment with 1,0 g of anhydrous sodium carbonate and 0,3 g of boric acid. Dissolve the melt in the main filtrate reserved in 9.2.3.3.2 and add the filtrate reserved in 9.2.3.3.3. If necessary, concentrate the solution to about 400 ml. Cool and dilute the solution to 500 ml in a volumetric flask.

Designate this solution as stock solution (S'1).

9.2.3.4 Blank test

Carry out the procedure described in 9.2.3.3 without the sample and omit the fusion of anhydrous sodium carbonate. Designate the solution equivalent to stock solution (S'1) as blank solution (B'1).

9.2.4 Preparing a solution by fusion and dissolution in sulfuric acid

9.2.4.1 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.

9.2.4.2 Mass of test portion

Weigh 0,50 g of the test sample.

9.2.4.3 Procedure

Weigh 0,5 g of the sample, prepared as described in Clause 6, in a platinum dish, add 3,0 g of anhydrous sodium carbonate and 2,0 g of boric acid and mix thoroughly. Heat over a burner cautiously at first, then raise the temperature to the full heat of the burner. Heat in an electric muffle furnace at 1 100 °C ± 25 °C for 15 min. Remove from the electric muffle furnace and allow to cool with a lid. Add 55 ml of sulfuric acid (1+9), and transfer to a boiling water bath to facilitate the dissolution of the melt. When the solution is complete, remove the lid and wash any spray into it with a minimum amount of water. Allow to cool, and dilute to 500 ml in a volumetric flask.

Designate this solution as stock solution (S''1).

9.2.4.4 Blank test

Carry out the procedure given in 9.2.4.3 without the sample and omit the fusion of anhydrous sodium carbonate. Designate the solution equivalent to stock solution (S''1) as blank solution (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, as described in 9.3.2;

b) decomposition and removal of the silica by hydrofluoric acid for atomic absorption spectrometry, as described in 9.3.3;

c) decomposition and removal of the silica by hydrofluoric acid for ICP-AE spectrometry, as described in 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 is made up to volume. This is designated as stock solution (S2).

9.3.2.2 Mass of test portion

Weigh 0,25 g of the test sample.

9.3.2.3 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) and 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, transfer to a 250 ml graduated flask and dilute to the mark with water.

Designate this solution as stock solution (S2).

9.3.2.4 Blank test

Carry out the procedure given in 9.3.2.3 without the sample. Designate the resulting solution as blank solution (B2).

9.3.3 Decomposition by hydrofluoric acid for atomic absorption spectrometry

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 is added and the solution is made up to volume. This is designated as 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 1ml of sulfuric acid (1+1) and 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 volumetric flask and dilute to the mark with water. Transfer immediately to a dry plastics bottle.

Designate this solution as stock solution (S3).

9.3.3.4 Blank test

Carry out the procedure given in with 9.3.3.3 without the sample.

Designate the resulting solution as blank solution (B3).

9.3.4 Decomposition by hydrofluoric acid for ICP-AE spectrometry

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 is made up to volume. This is designated as stock solution (S4).

9.3.4.2 Mass of test portion 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.

Designate this solution as stock solution (S4).

9.3.4.4 Blank test

Carry out the procedure given in 9.3.4.3 without the sample.

Designate the resulting solution as blank solution (B4).

10 Expression of test results

Express the test results in accordance with ISO 26845.

11 Examination and adoption of test results

If the absolute difference between the two test results is less than the error margin given in Table 2, report the mean test result as the final value.

If the absolute difference between the two test results is greater than the error margin given in Table 2, carry out the tests on two further samples. If the absolute difference between the two test results is equal to or more than the value given in Table 2, report the arithmetical mean of the two test results as the final value. If the range of four test results exceeds the value given in Table 2, report the median of the test results as the final value.

Table 2 — Permissible difference in test values

Mass fraction %	**Component**											
	LOI	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	Na_2O	K_2O	Cr_2O_3	ZrO_2	HfO_2
Under 0,1	0,02	0,02	—	0,02	0,01	0,02	0,02	0,02	0,02	0,01	—	0,02
~ 0,2	0,05	0,05	—	0,05	0,02	0,05	0,05	0,05	0,05	0,02	—	0,05
~ 0,5	0,05	0,05	—	0,05	0,03	0,05	0,05	0,05	0,05	0,03	—	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
~ 5	0,10	0,10	0,10	—	0,05	—	—	0,10	—	0,10	—	—
~ 10	0,20	0,20	0,20	—	—	—	—	—	—	—	0,10	—
~ 20	0,2	0,2	0,2	—	—	—	—	—	—	—	0,2	—
~ 50	0,2	—	0,2	—	—	—	—	—	—	—	0,2	—
Over 50	—	—	0,2	—	—	—	—	—	—	—	0,2	—

12 Test report

Prepare a test report as described in ISO 26845.

Annex A
(informative)

References for stock solutions and blank solutions in this part of ISO 21079

Throughout Parts 2 and 3 of ISO 21079:2008, reference is made to the various stock solutions and blank solutions prepared in this part of ISO 21079. Table A.1 gives a reference for each stock solution and blank solution to the subclause in this part of ISO 21079 where it is prepared.

Table A.1 — Reference to stock solution and blank solution in this part of ISO 21079

Stock or blank solution	Subclause
B1	9.2.2.4
B'1	9.2.3.4
B"1	9.2.4.4
B2	9.3.2.4
B3	9.3.3.4
B4	9.3.4.4
S1	9.2.2.3
S'1	9.2.3.3
S"1	9.2.4.3
S2	9.3.2.3
S3	9.3.3.3
S4	9.3.4.3

Bibliography

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

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

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

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

ICS 71.040.40; 81.080

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