



# SLOVENSKI STANDARD

SIST EN 955-2:1998

01-april-1998

---

## Kemijska analiza ognjevzdržnih izdelkov - 2. del: Izdelki, ki vsebujejo kremen in/ali glinico (metoda razklopa)

Chemical analysis of refractory products - Part 2: Products containing silica and/or alumina (wet method)

Chemische Analyse von feuerfesten Erzeugnissen - Teil 2: Silicium(IV)-Oxid und/oder Aluminiumoxid enthaltende Erzeugnisse (Naßchemisches Verfahren)

Analyse chimique des produits réfractaires - Partie 2: Produits contenant de la silice et/ou de l'alumine (méthode par voie humide)

[SIST EN 955-2:1998](https://standards.iteh.ai/catalog/standards/sist/20fc311f-b9dd-4b6c-973a-62446c5096c/sist-en-955-2-1998)

Ta slovenski standard je istoveten z: [EN 955-2:1995](https://standards.iteh.ai/catalog/standards/sist/20fc311f-b9dd-4b6c-973a-62446c5096c/sist-en-955-2-1998)

---

### ICS:

81.080

SIST EN 955-2:1998

en

**iTeh STANDARD PREVIEW**  
**(standards.iteh.ai)**

SIST EN 955-2:1998

<https://standards.iteh.ai/catalog/standards/sist/20fc311f-b9dd-4b6c-973a-b24f40c5096c/sist-en-955-2-1998>

EUROPEAN STANDARD

EN 955-2

NORME EUROPÉENNE

EUROPÄISCHE NORM

May 1995

ICS 81.080

Descriptors: Refractory materials, silicon dioxide, aluminium oxide, chemical analysis, determination of content, wet process, procedure

English version

**Chemical analysis of refractory products -  
Part 2: Products containing silica and/or alumina  
(wet method)**

Analyse chimique des produits réfractaires -  
Partie 2: Produits contenant de la silice et/ou  
de l'alumine (méthode par voie humide)

Chemische Analyse von feuerfesten Erzeugnissen  
- Teil 2: Silicium(IV)-Oxid und/oder  
Aluminiumoxid enthaltende Erzeugnisse  
(Naßchemisches Verfahren)

This European Standard was approved by CEN on 1995-01-17. CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

The European Standards exist in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

<https://standards.iteh.ai/catalog/standards/sist/20fc311f-b9dd-4b6c-973a-b24f40c5096c/sist-en-955-2>

**CEN**

European Committee for Standardization  
Comité Européen de Normalisation  
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

© 1995

All rights of reproduction and communication in any form and by any means reserved in all countries to CEN and its members.

Ref. No. EN 955-2:1995 E

## Contents

	Page
Foreword	3
1 Scope	4
2 Definitions	4
3 Sample preparation	4
4 Reagents	5
5 Apparatus	10
6 Determination of loss on ignition	11
7 Decomposition of the sample	11
8 Determination of silica and alumina, and oxides of iron, titanium, manganese and phosphorus	12
9 Determination of calcium oxide, magnesia and alkalis	17
10 Direct determination of alumina in silica refractories	21
11 Direct determination of alumina in aluminosilicates and high alumina refractories	23
12 Reporting of results	24
13 Test report	24
Annex A (normative) Calibrations	25

## iTeh STANDARD PREVIEW (standards.iteh.ai)

[SIST EN 955-2:1998](https://standards.iteh.ai/catalog/standards/sist/20fc311f-b9dd-4b6c-973a-b24f40c5096c/sist-en-955-2-1998)

<https://standards.iteh.ai/catalog/standards/sist/20fc311f-b9dd-4b6c-973a-b24f40c5096c/sist-en-955-2-1998>



## Foreword

This European standard has been prepared by the 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 November 1995, and conflicting national standards shall be withdrawn at the latest by November 1995.

According to the CEN/CENELEC Internal Regulations, the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

EN 955 "Chemical analysis of refractory products" consists of the following parts which are:

- Part 1: Magnesites and dolomites <sup>1)</sup>
- Part 2: Products containing silica and/or alumina (wet method)
- Part 3: Chrome-bearing products (wet method)
- Part 5: XRF fused cast bead method

## iTeh STANDARD PREVIEW (standards.iteh.ai)

SIST EN 955-2:1998

<https://standards.iteh.ai/catalog/standards/sist/20fc311f-b9dd-4b6c-973a-b24f40c5096c/sist-en-955-2-1998>

---

<sup>1)</sup> This document is currently prEN 30058 but is intended to become EN 955-1.

## 1 Scope

This part of EN 955 describes methods for chemical analysis of refractories containing predominantly silica ( $\text{SiO}_2$ ) and/or alumina ( $\text{Al}_2\text{O}_3$ ), using wet methods.

Annex A gives the instrument calibration requirements associated with the test method.

## 2 Definitions

For the purposes of this part of EN 955, the following definitions apply:

**2.1 silica refractory:** A refractory containing 93 % or more silica.

**2.2 alumino-silicate refractory:** A refractory containing between 7 % and 45 % alumina, the remainder being predominantly silica.

NOTE : Also classified as 'fireclay'.

**2.3 high alumina refractory:** A refractory containing more than 45 % alumina, the remainder being predominantly silica.

## 3 Sample preparation

Silica and alumino-silicate materials shall be ground in either agate, boron carbide or iron mortars. In the last case iron contamination shall be removed by treating with a magnet.

High alumina materials shall be ground in either alumina, boron carbide, tungsten carbide or iron mortars. In the last case, iron contamination shall be removed by treating with a magnet.

It may be necessary to prepare a sample by more than one method.

NOTE 1 : For example, the main analysis may be carried out on the iron-ground sample, with an iron (III) oxide determination on the agate-ground or alumina-ground sample, and then the results calculated to the latter iron content. Any metallic iron introduced will tend to oxidize to the iron (III) state by the heat treatment during the determination of the loss on ignition, and should be assumed to do so completely. Correction should be made for the difference in iron (III) oxide content of the two samples, expressed as metallic iron, which is the state present when weighing out the sample.

The sample for analysis shall be ground to pass completely through a 125  $\mu\text{m}$  sieve.

NOTE 2 : A non-metallic (e.g. nylon bolting cloth) sieve is preferable.

## 4 Reagents

### 4.1 General.

All reagents shall be of analytical grade and distilled or deionized water shall be used throughout the analysis.

### 4.2 Solid reagents

#### 4.2.1 Boric acid

4.2.2 *Calcein indicator (screened)*: Mix by grinding together 0,1 g of calcein, 0,06 g of thymolphthalein and 10 g of potassium chloride.

4.2.3 *Fusion mixture*: Mix anhydrous potassium and sodium carbonates in equimolar proportions.

4.2.4 *Methyl thymol blue complexone indicator*: Mix by grinding together 0,2 g of methyl thymol blue complexone and 20 g of potassium nitrate.

#### 4.2.5 Potassium periodate

#### 4.2.6 Potassium pyrosulfate

4.2.7 *Silica*: A quartz or sand of greater than 99 % m/m purity.

#### 4.2.8 Sodium carbonate (anhydrous)

#### 4.2.9 Polyethylene oxide

4.2.10 *Accelerator tablet* or finely macerated filter paper.

### 4.3 Solutions

#### 4.3.1 Acetic acid (glacial)

4.3.2 *Acetic acid solution (1 + 1)*: Add 250 ml of the acetic acid (4.3.1) to 250 ml of water and mix.

4.3.3 *Ammonia solution*, density = 0,88 g/ml

4.3.4 *Diluted ammonia solution (1 + 1)*: Add 500 ml of the ammonia solution (4.3.3) to 500 ml of water and mix.

4.3.5 *Ammonium acetate (400 g/l solution)*: Dilute 570 ml of the acetic acid (4.3.1) to 1700 ml with water, add carefully 570 ml of the ammonia solution (4.3.3) and mix. Adjust to pH 6,4 to 6,8 with either acetic acid or ammonia solution.

4.3.6 *Ammonium acetate, (100 g/l solution)*: Dilute 250 ml of the ammonium acetate (4.3.5) to 1 l.

**4.3.7 Ammonium acetate buffer solution:** Add 120 ml of the acetic acid (4.3.1) to 500 ml of water, followed by 74 ml of the ammonia solution (4.3.3). Dilute to 1 l and mix.

**4.3.8 Ammonium molybdate (80 g/l solution):** Dissolve 80 g of ammonium molybdate in water, filter if necessary, dilute to 1 l and store in a polyethylene bottle. Discard after four weeks, or earlier if any appreciable deposit is observed.

**4.3.9 Ammonium vanadate (2,5 g/l solution):** Dissolve 1,25 g of ammonium vanadate in water and dilute to 500 ml.

**4.3.10 Bromophenol blue (1 g/l solution):** 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.

**4.3.11 Chloroform:** B.P. grade.

**4.3.12 Cupferron (60 g/l solution):** Dissolve 3 g of cupferron in 50 ml of water; filter if necessary. Prepare this solution freshly when required. Store the solid reagent in a tightly stoppered bottle in the presence of a piece of ammonium carbonate to prevent decomposition.

NOTE : Unless this reagent is in good condition, brown colours of varying intensity may arise during use, for example in 8.4. Such colours may interfere with the detection of the end-point of the titration.

**4.3.13 2,4 Dinitrophenol (saturated solution):** Dissolve 1 g of 2,4 Dinitrophenol in 100 ml of hot water, cool and filter.

**4.3.14 Dithizone (0,25 g/l solution):** Dissolve 0,0125 g of dithizone in 50 ml of ethanol (4.3.15).

NOTE : This solution will keep for about one week.

**4.3.15 Ethanol (95 % V/V solution):** Industrial spirit.

**4.3.16 Hydrochloric acid solution:** density = 1,18 g/ml.

**4.3.17 Hydrochloric acid solution (1 + 1):** Add 500 ml of the hydrochloric acid solution (4.3.16) to 500 ml of water and mix.

**4.3.18 Hydrochloric acid solution (1 + 4):** Add 200 ml of the hydrochloric acid solution (4.3.16) to 800 ml of water and mix.

**4.3.19 Hydrochloric acid solution (1 + 19):** Add 50 ml of the hydrochloric acid solution (4.3.16) to 950 ml of water and mix.

**4.3.20 Hydrofluoric acid solution,** approximately 40 % V/V.

**4.3.21 Hydrogen peroxide (20 vol solution)**



**4.3.22 Hydroxylammonium chloride solution (100 g/l):** Dissolve 10 g of hydroxylammonium chloride in warm water, filter if necessary, cool and dilute to 100 ml.

**4.3.23 Nitric acid,** density = 1,42 g/ml.

**4.3.24 Nitric acid solution (1 + 2):** Add 100 ml of the nitric acid (4.3.23) to 200 ml of water and mix.

**4.3.25 Nitric acid solution (1 + 19):** Add 50 ml of the nitric acid (4.3.23) to 950 ml of water and mix.

**4.3.26 1,10 - Phenanthroline hydrate solution (10 g/l)**  
Prepare enough solution in the acetic acid (4.3.2) for immediate use.

**4.3.27 Orthophosphoric acid,** density = 1,75 g/ml.

**4.3.28 Orthophosphoric acid solution (2 + 3):** Add 200 ml of the orthophosphoric acid (4.3.27) to 300 ml of water and mix.

**4.3.29 Orthophosphoric acid solution (1 + 9):** Add 100 ml of the orthophosphoric acid (4.3.27) to 900 ml of water and mix.

**4.3.30 Polyethylene oxide solution (2,5 g/l):** Slowly add 0,5 g of the polyethylene oxide (4.2.9) to 200 ml of water, stirring, preferably with a mechanical stirrer, until dissolved. Discard the solution after two weeks.

**4.3.31 Potassium hydroxide solution (250 g/l):** Dissolve 250 g of potassium hydroxide in water, cool and dilute to 1 litre. Store in a polyethylene bottle.

**4.3.32 Sulfuric acid,** density = 1,84 g/ml.

**4.3.33 Sulfuric acid solution (1 + 1):** Carefully add 200 ml of the sulfuric acid (4.3.32) to 200 ml of water, cooling the solution and keeping it well mixed during the addition of the acid.

**4.3.34 Sulfuric-nitric acid mixture:** To 650 ml of water add 100 ml of the sulfuric acid solution (4.3.33) and 250 ml of the nitric acid (4.3.23).

**4.3.35 Tin (II) chloride solution (10 g/l):** Dissolve by warming 1 g of tin (II) chloride in 1,5 ml of the hydrochloric acid solution (4.3.16), cool and dilute to 100 ml. Make this solution immediately before use.

**4.3.36 Triethanolamine solution (1 + 1):** Add 500 ml of triethanolamine to 500 ml of water and shake thoroughly.

#### 4.4 Standard solutions

**4.4.1 Calcium solution.** ( $c(\text{CaO}) = 1,0 \text{ mg/ml}$ ). Dry about 2 g of calcium carbonate at 150 °C for 2 h. Weigh 1,785 g and dissolve in a slight excess of the hydrochloric acid (4.3.18). Boil the solution to expel carbon dioxide, cool and dilute to make in a 1000 ml volumetric flask.

**4.4.2 Cyclohex-1, 2-ylenediaminetetra-acetic acid (DCTA)** (0,05 mol/l approximately). Dissolve 18 g of DCTA in 500 ml of water by the progressive addition of the minimum amount of potassium hydroxide solution (4.3.31).

NOTE 1 : Approximately 25 ml is required.

Dilute to the mark in a 1000 ml volumetric flask. Store in a polyethylene bottle. Standardize against the zinc solution (4.4.19) as follows.

Transfer 50,0 ml of the DCTA solution to a 500 ml conical flask and add 5 to 6 drops of the hydrochloric acid (4.3.16). Add a few drops of bromophenol blue indicator solution (4.3.10) and then add the ammonium acetate buffer solution (4.3.7) until the indicator turns blue, followed by 10 ml in excess.

Add a volume of the ethanol (4.3.15) equal to the total volume of the solution, followed by 1 to 2 ml of the dithizone indicator solution (4.3.14) and titrate with the standard zinc solution (4.4.19) from blue-green to the first appearance of a permanent pink colour.

NOTE 2 : 1 ml of 0,05 mol/l DCTA is equivalent to 2,55 mg  $\text{Al}_2\text{O}_3$ .

**4.4.3 Ethylenediaminetetra-acetic acid (di-sodium salt, dihydrate) (EDTA) (5 g/l).** Dissolve 5 g EDTA in warm water, filter if necessary, cool and dilute to 1 l. Store in a polyethylene bottle. Standardize against the standard calcium (4.4.1) and magnesium (4.4.8) solutions as follows.

**Calcium.** Pipette 25,0 ml of the standard calcium solution (4.4.1) into a 500 ml conical flask, add 10 ml of the potassium hydroxide solution (4.3.31) and dilute to about 200 ml. Add 0,015 g of the screened Calcein indicator (4.2.2) and titrate with the EDTA solution from fluorescent green to pink.

**Magnesium.** Pipette 25,0 ml of the standard magnesium solution (4.4.8) into a 500 ml conical flask, add 20 drops of the hydrochloric acid (4.3.16), 20 ml of the ammonia solution (4.3.3) and dilute to about 200 ml. Add 0,04 g of the methylthymol blue complexone indicator (4.2.4) and titrate with the EDTA solution from blue to colourless.

**4.4.4 Iron solution (A) ( $c(\text{Fe}_2\text{O}_3) = 0,1 \text{ mg/ml}$ ):** Dissolve 0,491 g of iron (II) ammonium sulfate in water, add 10 ml of the sulfuric acid (4.3.33) and 5 ml of the hydrogen peroxide (4.3.21). Boil for 15 min to decompose the excess of hydrogen peroxide. Cool and dilute to the mark in a 1000 ml volumetric flask.

**4.4.5 Iron solution (B) ( $c(\text{Fe}_2\text{O}_3) = 0,01 \text{ mg/ml}$ ):** Dilute 50 ml of iron solution (A) (4.4.4) to the mark in a 500 ml volumetric flask. Prepare this solution freshly when required.

**4.4.6 Lithium solution (A) ( $c(\text{Li}_2\text{O}) = 400 \text{ mg/l}$ ):** Dehydrate lithium sulfate monohydrate by heating at 150 °C for 24 h. Dissolve 1,472 g of the anhydrous lithium sulfate in water and dilute to 1000 ml in a volumetric flask. Store in a polyethylene bottle.

**4.4.7 Lithium solution (B) ( $c(\text{Li}_2\text{O}) = 20 \text{ mg/l}$ ):** Dilute 25 ml of the lithium solution A (4.4.6) to the mark in a 500 ml in a volumetric flask. Store in a polyethylene bottle.

**4.4.8 Magnesium solution ( $c(\text{MgO}) = 1,0 \text{ mg/ml}$ ):** Dissolve 0,603 g of magnesium metal in a slight excess of the hydrochloric acid (4.3.18), cool and dilute to the mark in a 1000 ml volumetric flask. Before weighing, etch the metal in the hydrochloric acid (4.3.18) and then dry it with ethanol followed by diethyl ether.

**4.4.9 Manganese solution A. ( $c(\text{MnO}) = 0,1 \text{ mg/ml}$ ):** Dilute a suitable volume of previously standardized, approximately 0,02 mol/l potassium permanganate to the mark in a 1000 ml volumetric flask. Store in a glass bottle and discard if any brown stain or deposit appears.

NOTE : With exactly 0,02 mol/l potassium permanganate, 70,5 ml is required.

**4.4.10 Manganese solution B. ( $c(\text{MnO}) = 0,01 \text{ mg/ml}$ ):** Dilute 50 ml of the manganese solution A (4.4.9) to the mark in a 500 ml volumetric flask. Prepare this solution freshly when required.

**4.4.11 Phosphate solution. ( $c(\text{P}_2\text{O}_5) = 0,1 \text{ mg/ml}$ ):** Dissolve 0,192 g of potassium dihydrogen orthophosphate in water and dilute to the mark in a 1000 ml volumetric flask. Store in a polyethylene bottle.

**4.4.12 Potassium/sodium solution A ( $c(\text{K}_2\text{O}) = 200 \text{ mg/l}$ ;  $c(\text{Na}_2\text{O}) = 100 \text{ mg/l}$ ):** Dissolve 0,370 g of anhydrous potassium sulfate and 0,229 g of anhydrous sodium sulfate in water and dilute to the mark in a 1000 ml volumetric flask. Store in a polyethylene bottle.

NOTE : The anhydrous sulfates should be dried at  $(100 \pm 5) \text{ }^\circ\text{C}$  for 1 h before use.