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Determination of sulfur in refractory products and raw materials by gravimetric, photometric and titrimetric methods

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National foreword

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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.

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INTERNATIONAL STANDARD

ISO 22016

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Determination of sulfur in refractory products and raw materials by gravimetric, photometric and titrimetric methods

Dosage du soufre dans les produits réfractaires et les matériaux bruts par gravimétrie, et des méthodes photométriques et titrimétriques

Reference number
ISO 22016:2015(E)

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Foreword

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The committee responsible for this document is ISO/TC 33, *Refractories*.

INTERNATIONAL STANDARD ISO 22016:2015(E)

Determination of sulfur in refractory products and raw materials by gravimetric, photometric and titrimetric methods

1 Scope

This International Standard specifies methods for the wet chemical analysis of refractory products and their raw materials with below 5 % (mass percentage) sulfur.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 836, *Terminology for refractories*

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

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 836, ISO 26845, and the following apply.

3.1
sulfur
general term for elemental sulfur, sulfides, sulfates, sulfites, etc. in refractory products and their raw materials

4 Apparatus

4.1 General

The following apparatus is required in addition to ordinary laboratory apparatus and the apparatus given in ISO 26845.

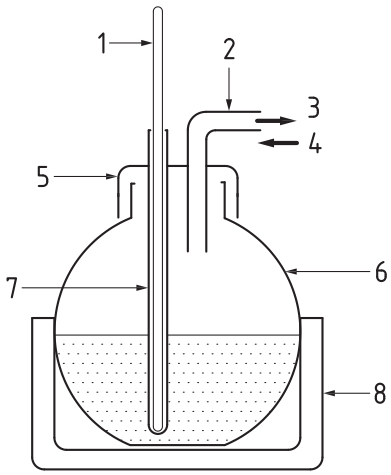
4.1.1 Platinum crucible, with a nominal volume of 25 ml, made, for example, in Pt/Au 95/5 %.

4.1.2 pH-meter.

4.1.3 Electrically heated furnace, adjustable to (1 150 ± 25) °C.

4.2 Apparatus specified in method B

4.2.1 Apparatus for tin(II): strong phosphoric acid solution preparation, an example of an apparatus consisting of a quartz flask is shown in Figure 1.

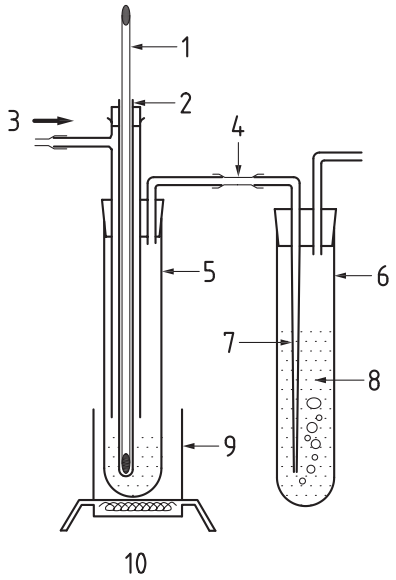


Key

1	thermometer (360 °C)	5	cap made of quartz
2	quartz tubes (⌀ 10 mm)	6	quartz flask (500 ml)
3	tap aspirator	7	quartz tube (⌀ 10 mm)
4	N_2 gas	8	heating mantle

Figure 1 — Example of apparatus for tin(II): strong phosphoric acid solution preparation

4.2.2 Sample decomposition and gas absorption equipment, example of apparatus is shown in Figure 2.



Key

1	thermometer 0 °C to 310 °C	6	test tube for absorption (⌀ 30 mm)
2	quartz tube	7	glass tube for absorption
3	N_2 gas	8	zinc acetate solution
4	polyethylene tetrafluoride resin connecting tube	9	aluminium foil wall
5	test tube for decomposition (⌀ 30 mm)	10	heater (300 W)

Figure 2 — Example of sample decomposition and gas absorption equipment

5 Reagents

5.1 General

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

Use only reagents of recognized analytical grade during the analysis unless otherwise stated 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.

5.1.1 Silver nitrate solution (10 g/l), dissolve 1 g of silver nitrate into 100 ml of water. Store the solution in an amber-coloured bottle.

5.1.2 Sodium carbonate solution (5 g/l), dissolve 5 g of anhydrous sodium carbonate into 1 l of water.

5.1.3 Sodium thiosulfate pentahydrate for Method B, $Na_2S_2O_3 \cdot 5H_2O$, (ISO 6353-2, R 36), minimum mass fraction 99,0 %.

5.1.4 Potassium nitrate.

5.1.5 Barium chloride di-hydrate for Method A2, $BaCl_2 \cdot 2H_2O$.

5.1.6 Barium chloride solution.

5.1.6.1 100 g/l anhydrous $BaCl_2$ for Method A1, 10 g of anhydrous barium chloride is dissolved in water and made up 100 ml.

5.1.6.2 100 g/l $BaCl_2 \cdot 2H_2O$ for Method A2, 10,0 g Barium chloride di-hydrate (5.1.5) is dissolved in water and made up to 100 ml.

5.1.6.3 5,42 g/l anhydrous $BaCl_2$ for Method C, 0,542 g of anhydrous barium chloride is dissolved in water and made up to 100 ml.

5.1.7 Tin(II): strong phosphoric acid solution for Method B, transfer 500 g of phosphoric acid into the quartz flask in Figure 1 in Clause 5, heat it to 250 °C for 1 h to dehydrate, draw with water aspirator to remove steam generated, and cool. Add 80 g of anhydrous tin(II) chloride, heat the mixture under nitrogen gas to 300 °C, cool to room temperature under nitrogen gas, and store in a desiccator.

5.1.8 Zinc acetate solution for Method B, dissolve 40 g of anhydrous zinc acetate into approximately 300 ml of water, add 30 ml of acetic acid to the solution, dilute to 1 000 ml with water, and store in a plastic bottle.

5.1.9 Starch solution for Method B, dissolve 0,1 g of starch (soluble) into approximately 10 ml of water, add this solution into 200 ml of hot water (80 °C ± 10 °C) while agitating, boil for about 1 min and cool to room temperature, store in a cold place, and discard after 10 days.

5.1.10 Sulfate solution, S 0,1mg/ml, dry about 1 g of sodium sulfate, Na_2SO_4, at 110 °C ± 5 °C for 2 h. Dissolve 0,443 g in water, diluting to 1 000 ml in a volumetric flask.

5.1.11 Potassium iodate, KIO_3, reference material for volumetric analysis or reagent of at least mass fraction 99,95 % purity.

5.1.12 Potassium iodide, KI, (ISO 6353-2, R 25), minimum mass fraction 99,5 %.

5.1.13 Iodine solution for Method B, 0,01 mol/l, weigh 1,27 g of iodine and 10 g of potassium iodide (5.1.12) and dissolve in approximately 50 ml of water, diluting to 1 000 ml in a volumetric flask. Store the solution in an amber-coloured bottle.

5.1.14 Mixture acid for Method C, hydrochloric acid (concentrated) and nitric acid (V) (concentrated) in a ratio of 3:1, prepared before use.

5.1.15 Ammonia solution, concentrated, ρ= 0,91 g/ml and 1+1 solution for method C.

5.1.16 EDTA acid ammonium salt solution for Method C, dissolve 10 g of EDTA in 200 ml of water, add 100 ml of ammonia (concentrated), and dilute to 500 ml with water.

5.1.17 Sodium carbonate/potassium carbonate mixture for Method A2, one part Na_2CO_3 and one part K_2CO_3 are well mixed.

5.1.18 Potassium nitrate, KNO3.

5.1.19 Hydrochloric acid, HCl ≈ 6 mol/l, supplied as 6M or dilute ~530 ml of conc. HCl (SG 1.18) to ~1 l in water

5.1.20 Hydrochloric acid, HCl ≈ 2 mol/l, supplied as 2M or dilute one part of hydrochloric acid (5.1.9) with two parts of water.

5.1.21 Hydrofluoric acid, HF, w(HF) = 40 %, ρ = 1,13 g/ml.

5.2 Standard solutions for method B

5.2.1 0,01 mol/l sodium thiosulfate standard solution, weigh 2,6 g of sodium thiosulfate pentahydrate (5.1.3) and 0,2 g of sodium carbonate, dissolve in 1 l of dissolved oxygen-free water. Store the solution in an airtight container. Let it stand for 2 days prior to use.

5.2.1.1 Standardization

Dry 0,5 g of potassium iodate (5.1.11) at 130 °C for 2 h and allow it to cool in a desiccator. Weigh 0,3 g to 0,4 g to the nearest 0,1 mg and dissolve in water, diluting to 1 000 ml in a volumetric flask. Pipette 25 ml into a 200 ml Erlenmeyer flask, add 2 g of potassium iodide (5.1.12), and 2 ml of sulfuric acid (1+1), immediately stopper the flask, shake gently for 1 min, and allow it to stand for 5 min in a dark place. Add starch solution and titrate with the 0,01 mol/l sodium thiosulfate standard solution until it becomes faint yellow which shows the end point is near. Complete disappearance of blue colour indicates the end point.

Separately, add 25 ml of water and 2 g of potassium iodide into a 200 ml Erlenmeyer flask, add 2 ml of sulfuric acid (1+1), immediately stopper the flask, shake gently, and allow it to stand for 5 min in a dark place. Carry out a blank test under the same conditions as above and correct the volume needed for titration.

5.2.1.2 Calculation

Calculate the factor according to Formula (1):

$$f = \frac{m \times 25/1\,000}{0{,}000\,356\,67 \times (v_1 - v_2)} \times \frac{a}{100} \quad (1)$$

where

f is the factor of 0,01 mol/l sodium thiosulfate standard solution;

m is the mass of potassium iodate weighed out, in g;

a is the purity of potassium iodate, in %;

0,000 356 67 is the mass of potassium iodate equivalent to 1 ml of 0,01 mol/l sodium thiosulfate standard solution, in g;

v_1 is the volume of 0,01 mol/l sodium thiosulfate standard solution needed for titration of potassium iodate, in ml;

v_2 is the volume of 0,01 mol/l sodium thiosulfate standard solution needed for titration of blank test, in ml.

6 Sample preparation

The sampling shall be conducted according to ISO 8656-1 or as agreed upon between the parties involved. The sample shall be dried at (110 ± 5) °C to constant weight. If necessary, any agglomerated material shall be ground and homogenised.

7 Determination of sulfur

7.1 General

The determination of sulfur is carried out using one of the following methods.

— **Method A**: Fusion with alkali carbonate - barium sulfate gravimetric method. This method is applied to samples with 0,01% to 5% sulfur percentage (mass percentage).

— **Method B**: Decomposition by tin(II) - strong phosphoric acid solution - Iodine and thiosulfate back-titration method. This method is applied to samples with below 0,1% sulfur percentage (mass percentage).

— **Method C**: Barium sulfate (VI) precipitated by oxidation is determined by flame photometry. This method is applied to samples up to 5% sulfur percentage (mass percentage).

7.2 Method A

NOTE The sulfur analytical procedures for the gravimetric method are described individually although they are based on the same principle and subdivided into methods A1 and A2.

7.2.1 Method A1

7.2.1.1 Principle

Sodium carbonate and potassium nitrate are added to a sample and the mixture is fused by heating in an electric furnace. The sulfur contained in the sample is stabilised as alkali sulfate. The fused salts are dissolved with water and the resulting solution is filtered to separate the insoluble matter. Ethanol and hydrochloric acid are added to the filtrate and heated in order to reduce chromium (VI) oxide and to remove carbon dioxide from the decomposition of sodium carbonate. Barium chloride solution is added into the resulting solution to precipitate sulfur in the form of barium sulfate. The precipitate is filtered and calcined and then weighed.

7.2.1.2 Mass of test portion to be weighed

The mass of the test portion depends on the sulfur content. Weigh the mass given in Table 1 and record it to the nearest 0,1 mg.

Table 1 — Mass of test portion

Sulfur content	Mass of test portion
% by mass	g
0,01 to 0,50	1,0
0,5 to 5,0	0,5

7.2.1.3 Procedure

Weigh a sample in a platinum dish (e.g. 75 ml), add 10 g of sodium carbonate and 0, 2 g of potassium nitrate, and mix thoroughly. Cover the platinum dish loosely with platinum lid and put into an electric furnace, then gradually raise the temperature to 1 000 °C ± 25 °C, hold the temperature for about 30 min and allow to cool.

Place the platinum dish and the platinum lid into a 300 ml beaker and then add 100 ml of water into the beaker, cover with a watch glass, and heat on a water bath until the fused matter is dissolved. Remove the platinum dish and the platinum lid, rub off the insoluble matter with a rubber policeman, and rinse with hot water through a fine filter paper (closed-pore grade 42) into a 500 ml beaker, and wash several times with hot sodium carbonate solution (5.1.2). Retain the filtrate and washings in a 500 ml beaker.

To this solution, add precisely 10,0 ml of the standard solution of sulfate salt, two drops of methyl orange indicator 0,01 mol/l, and 5 ml of ethanol (95 %). While stirring, add hydrochloric acid (1+1) drop wise until neutral point and then add an excess of 5 ml. Dilute with water to approximately 200 ml, cover with watch glass, and heat until boiling.

While stirring, slowly add 10 ml of hot barium chloride solution to the beaker, place it in boiling water bath, heat for 2 h, and then allow to stand overnight. Filter through a filter paper (closed-pore grade 42 for fine precipitation) and then wash the precipitate and filter paper with hot water. Rinsing of the precipitate and the filter paper is carried out until the washings do not produce a white precipitation on addition of silver nitrate to a portion of the washings.

Transfer the precipitate with the filter paper to a tared platinum or porcelain crucible (e.g. 30 ml). Heat over a burner or an electric furnace at low temperature until ashing of the filter paper is complete and then heat at 825 °C ± 25 °C for about 30 min. Cool crucible in a desiccator to room temperature and weigh.

7.2.1.4 Blank test

Carry out the procedure in accordance with 7.2.1.3 without sample. The procedures of no fusion are carried out because the platinum crucible is attacked when the reagents are fused without samples, and the reagents, which are not fused, can be solved in the procedure.

7.2.1.5 Calculation

Calculate the mass fraction of sulfur, w_s, in the sample as percentage, by using Formula (2):

$$w_s = \frac{(m_1 - m_2) \times 0{,}137\ 4}{m} \times 100 \qquad (2)$$

where

m_1 is the mass of the precipitation in 7.2.1.3, in g;

m_2 is the mass of precipitation in 7.2.1.4, in g;

0,137 4 is the mass ratio of sulfur (S) against the precipitation (BaSO4);

m is the mass of test portion to be weighed, in g.

7.2.2 Method A2

7.2.2.1 Principle

The sample is fused with a mixture of sodium carbonate, potassium carbonate and potassium nitrate. After cooling, the melt is dissolved and the sulfate is precipitated as barium sulfate with barium chloride. After fuming with hydrofluoric acid, the barium sulfate is determined gravimetrically.

7.2.2.2 Procedure

About 1 g of the sample prepared according to Clause 6 is weighed to 0,1 mg into the platinum crucible (4.1.1) and mixed with 6 g of the sodium-carbonate-potassium carbonate mixture (5.1.17), and 0,5 g of potassium nitrate (5.1.18). The platinum crucible (4.1.1) and contents are then heated in an electrically-heated oven (4.1.3) to 1,150 °C.

NOTE The decomposition time is about 20 min (see Reference [1]).

After completion of the fusion in the furnace, the crucible is removed and as the melt cools, the dish is rotated and rocked in order to produce a thin layer of melt all over the walls of the crucible. While still

hot, the crucible is immersed to half way up its sides in water so that the melt breaks away in small pieces to aid dissolution.

The melt is then dissolved in 150 ml of water and filtered through a medium-hard white-band type filter paper (grade 40). The filtrate is washed with hot, neutral pH water.

Acidify the cold filtrate to a pH of 4 to 5 using hydrochloric acid (5.1.19). After checking the pH, boil the solution to remove carbon dioxide.

After cooling the solution, adjust the pH to just exceed neutrality using ammonia solution (5.1.15). Then, with about 6 ml of hydrochloric acid (5.1.20), bring the pH to 3 to 4, checking with a pH meter. The weakly acidic solution is then brought to boiling point while being stirred and then 10 ml of barium chloride (5.1.6) is added. The solution with the precipitate is then boiled for a few more minutes and then heated for a further 15 min. This converts the precipitate into a more filterable form.

After about 12 h, the precipitate is filtered through a fine grade 42 filter paper and the filtrate washed with hot water until free of chloride.

The filter paper and the precipitate is transferred to a previously weighed crucible (4.1.1) and then ashed to a maximum of 800 °C. The residue is then treated with 1 ml of hydrofluoric acid (5.1.21), evaporated until dry and then heated at 800 °C to constant weight, to anneal the barium sulfate.

7.2.2.3 Calculation

The total sulfur content, expressed as a percentage by mass, is calculated according to Formula (3):

$$\mathrm{W(S)} = \left(M \times f \times 100\right) / m_1 \tag{3}$$

where

M is the the final mass of barium sulfate after annealing, in g;

f is 0,137 4, the mass ratio of sulfur (S) against the precipitation ($BaSO_4$);

m_1 is the sample mass in grams.

The total content of sulfur shall be rounded up to the nearest 0,01 %.

7.3 Method B

7.3.1 Principle

A sample is heated in the solution of tin (II): strong phosphoric acid solution (5.1.7) in order to decompose sulfur into hydrogen sulfide. The generated hydrogen sulfide is transferred by bubbling of nitrogen gas into zinc acetate solution and absorbed. Excess iodine solution is added to the zinc acetate solution to oxidize the sulfide and the un-reacted iodine is determined by back-titration of sodium thiosulfate solution with starch as an indicator.

7.3.2 Mass of test portion

Weigh, to the nearest 0,1 mg, 1,0 g of test sample.

7.3.3 Procedure

Weigh the sample and transfer it directly into the test tube for decomposition (key 5 in Figure 2) which has previously been dried. Add 15 ml of tin (II): strong phosphoric acid solution to the test tube and dissolve the sample into the liquid by gently shaking.

Add 50 ml of zinc acetate solution into the test tube for absorption (key 6 in Figure 2) and set up the sample decomposition and absorption equipment.

Heat the test tube for decomposition (key 5 in Figure 2) and keep at 280 °C to 290 °C for approximately 45 min under a flow of nitrogen gas.

After heating, continue to pass nitrogen gas over the sample for approximately 5 min and remove the test tube for absorption (key 7 in Figure 2). Wash the glass tube for absorption (key 7 in Figure 2) and mix the washings into the absorption solution.

Transfer the solution into a 200 ml Erlenmeyer flask by washing with water, add 5 ml of 0,01 mol/l iodine solution and titrate immediately with 0,01 mol/l sodium thiosulfate standard solution. Add four or five drops of the starch solution when the solution becomes slightly yellow and titrate until the end point which is when the purple colour has disappeared.

7.3.4 Blank test

Carry out the procedure of 7.3.3without a sample. The same amount of 0,01 mol/l iodine solution shall be added as was added to the sample.

7.3.5 Calculation

Calculate the mass fraction of sulfur, w_S, in the sample as percentage, by using Formula (4):

$$w_S = \frac{(v_1 - v_2) \times f \times 0{,}000\ 160\ 3}{m} \times 100 \tag{4}$$

where

v_1 is the used amount of 0,01 mol/l sodium thiosulfate standard solution in 7.3.4 in ml;

v_2 is the used amount of 0,01 mol/l sodium thiosulfate standard solution in 7.3.3, in ml;

f is the factor of 0,01 mol/l sodium thiosulfate standard solution;

0,000 160 3 is the mass of sulfur in 1 ml of 0,01 mol/l sodium thiosulfate standard solution;

m is the mass of test portion to be weighed, in g.

7.4 Method C

7.4.1 Principle

The sample is first oxidised in solution and then precipitated and filtered as $BaSO_4$ using $BaCl_2$. The precipitate is re-dissolved in ammoniacal EDTA and determined by flame photometry.

7.4.2 Mass of test portion

Weigh, to the nearest 0,1 mg, 1,0 g of test sample.

7.4.3 Procedure

Weigh 1 g of a sample and transfer into a 400 ml beaker, add 30 ml of the mixture of acids (5.1.14), cover the beaker with a watch glass, and leave for 1 h to 2 h. Then evaporate until dry. After cooling, add 100 ml of hot water into the residue and heat on a bath at ca. 90 °C until the salt is dissolved. Precipitate the hydroxides with ammonia solution (1+1) and heat for several minutes. Filter the hot solution through a medium-speed grade 40 filter paper and wash with hot water three to four times. Add 20 ml of barium chloride solution to the filtrate, stir, and heat for 1 h on a bath. Cool and filter the solution through a hard

grade 42 filter paper and wash several times until the filtrate ceases to form a precipitate with silver nitrate solution (5.1.1).

Quantitatively, transfer the deposition into a 200 ml beaker by means of 100 ml solution of EDTA acid ammonium salt (5.1.16) and stir while heating gently until the salt is dissolved. After cooling the solution, transfer to a 250 ml volumetric flask and make up to the mark with water.

Photometric calibration standards shall be prepared from dried barium chloride, the solution of EDTA ammonium salt (5.1.16) shall also be added to the standards.

The volume and concentration of $BaCl_2$ solution corresponds to 5 % content of total sulfur. In the event that this value is exceeded, the weighted sample should be reduced accordingly.

7.4.4 Calculation

Total sulfur content should be calculated on the basis of Formula (5):

$$\%SO_3 = \frac{C \cdot 100\%}{4 \cdot m} \cdot \frac{80{,}07}{137{,}33} = \frac{C}{m} \cdot 14{,}576 \qquad (5)$$

where

C is the concentration of Ba^{2+} ions indicated by the flame photometer, in g/l;

m is the weighted sample, in g;

$\frac{80{,}07}{137{,}33}$ is the Ba to SO_3 conversion factor.

8 Test report

The test report shall contain, as a minimum, the following information:

a) name of the testing establishment;

b) date of test;

c) reference to this International Standard, i.e. ISO 22016, and analytical method A1, A2, B, or C selected;

d) description of materials tested;

e) chemical components and test results.

Annex A
(informative)

Statistical results of sulfur analyses in refractories and raw materials

A.1 Analysis samples

(sample number) (materials)

a) SA 001: pyrophyllite raw materials 1

b) SA 002: Al2O3 – CaSO4 mix

c) SA 003: pyrophyllite raw materials 2

d) SA 004: Al2O3 – K2SO4 mix

e) SA 005: clay – CaSO4 mix

f) SA 006: clay – K2SO4 mix

g) SA 007: used magnesia chrome bricks

h) SA 008: used spinel bricks

A.2 Analytical results

Analytical results by Methods A1 and C are shown in Table A.1 and Table A.2, respectively.

Table A.1 — Round robin results for sulfur analyses by Method A1, % by mass

sample	n	Laboratories				
		L1	L2	L3	L4	$= x$
SA 001	x_1	0,101	0,104	0,109	0,109	
	x_2	0,100	0,099	0,105	0,106	
	$\bar{x}$	0,100	0,102	0,107	0,108	0,104
SA 002	x_1	0,272	0,281	0,308	0,323	
	x_2	0,262	0,269	0,307	0,293	
	$\bar{x}$	0,267	0,275	0,308	0,303	0,288
SA 003	x_1	1,016	1,007	1,010	1,052	
	x_2	1,003	1,040	1,038	1,024	
	$\bar{x}$	1,010	1,024	1,024	1,038	1,024
SA 004	x_1	1,556	1,589	1,478	1,488	
	x_2	1,452	1,388	1,468	1,542	
	$\bar{x}$	1,504	1,489	1,473	1,515	1,495

Table A.1 *(continued)*

sample	n	Laboratories				
		L1	L2	L3	L4	$\overline{\overline{x}}$
SA 005	x_1	2,748	2,843	2,870	2,912	
	x_2	2,698	2,770	2,865	2,852	
	$\overline{x}$	2,723	2,806	2,868	2,882	2,820
SA 006	x_1	4,840	4,825	4,918	4,931	
	x_2	4,924	4,864	4,934	4,918	
	$\overline{x}$	4,882	4,844	4,926	4,924	4,894
SA 007	x_1	0,485	0,536	0,541	0,519	
	x_2	0,512	0,522	0,543	0,492	
	$\overline{x}$	0,498	0,529	0,542	0,506	0,518
SA 008	x_1	0,600	0,609	0,624	0,659	
	x_2	0,604	0,629	0,626	0,629	
	$\overline{x}$	0,602	0,619	0,625	0,644	0,622

Table A.2 — CRM measurement results by Method C, % by mass

CRM	Certified value % SO3	Obtained value % SO3
Cement X0201	0,115	0,109
Cement X0202	0,046	0,049
Calcium 1d	0,257	0,246
Bauxite 69b	0,551	0,534

Bibliography

[1] Bock R (1972), Autschlussmethoden der anorganischen und organischen Chemie, Veelag Chemie, S,83,

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