SLOVENSKI STANDARD
oSIST prEN ISO 14720-2:2025

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Preskušanje keramičnih materialov - Ugotavljanje žvepla v neoksidativnih keramičnih surovinah in osnovnih materialih - 2. del: Atomska emisijska spektrometrija z induktivno sklopljeno plazmo (ICP AES) ali ionska kromatografija (IC) po izpostavitvi ognju v pretoku kisika (ISO/DIS 14720-2:2025)

Testing of ceramic materials - Determination of sulfur in non-oxidic ceramic raw materials and ceramic materials - Part 2: Inductively coupled plasma optical emission spectrometry (ICP-OES) or ion chromatography (IC) after burning in the oxygen flow (ISO/DIS 14720-2:2025)

Prüfung keramischer Werkstoffe - Bestimmung des Schwefelgehaltes in nichtoxidischen keramischen Roh- und Werkstoffen - Teil 2: Optische Emissionsspektrometrie mit induktiv gekoppeltem Plasma (ICP-OES) oder Ionenchromatographie (IC) nach Verbrennung im Sauerstoffstrom (ISO/DIS 14720-2:2025)

Essai des matières céramiques - Détermination de la teneur en soufre des matières premières céramiques non oxydes et des matériaux céramiques non oxydes - Partie 2: Spectrométrie d'émission optique avec plasma couplé inductif (ICP-OES) ou chromatographie ionique (IC) après combustion dans le courant d'oxygène (ISO/DIS 14720-2:2025)

Ta slovenski standard je istoveten z: **prEN ISO 14720-2**

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81.060.10	Surovine	Raw materials

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Testing of ceramic materials - Determination of sulfur in non-oxidic ceramic raw materials and ceramic materials —

Part 2: inductively coupled plasma optical emission spectrometry (ICP-OES) or ion chromatography (IC) after burning in the oxygen flow

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Partie 2: Spectrométrie d'émission optique avec plasma couplé inductif (ICP-OES) ou chromatographie ionique (IC) après combustion dans le courant d'oxygène

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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 14720-2 was prepared by Technical Committee ISO/TC 33, *Refractories*.

ISO 14720 consists of the following parts, under the general title *Testing of ceramic materials — Determination of sulfur in non-oxidic ceramic raw materials and ceramic materials*:

— *Part 1: Infrared measurement methods*

— *Part 2: Inductively coupled plasma optical emission spectrometry (ICP-OES) or ion chromatography (IC) after burning in the oxygen flow*

Testing of ceramic materials - Determination of sulfur in non-oxidic ceramic raw materials and ceramic materials —

Part 2:
inductively coupled plasma optical emission spectrometry (ICP-OES) or ion chromatography (IC) after burning in the oxygen flow

1 Scope

This part of ISO 14720 defines a method for the determination of sulfur in non-oxidic ceramic raw materials and ceramic materials, which are completely oxidized at a higher temperature in an oxygen atmosphere, for example carbon and graphite materials.

For materials which are not completely oxidizable under these conditions, it is possible to determine sulfur that can be released under these conditions, for example the adherent sulfur.

This part of ISO 14720 is applicable for materials with mass fractions of sulfur ≤ 10 % and mass fractions of ash < 20 %, The defined method is limited for materials with mass fractions of barium < 10 mg/kg, because the sulfur bonded in barium sulfate is not detectable with this method.

For the lower detection limit of this method, a mass fraction of sulfur of 0,5 mg/kg in the case of inductively coupled plasma optical emission spectrometry (ICP-OES) and 5 mg/kg in the case of ion chromatography (IC) has to be considered as a recommended value.

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 11885, *Water quality — Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES)*

ISO 10304-1, *Water quality — Determination of dissolved anions by liquid chromatography of ions — Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
sulfur content
mass fraction of inorganic and organic bound sulfur

4 Principle

The dried sample is oxidized in a flow of oxygen at a temperature of 1 100 °C using a porcelain crucible. The resulting sulfur oxides are absorbed in a solution of sodium hydroxide and hydrogen peroxide. The remaining material (ash) is dissolved and combined with the absorption liquid in the case of calcium-sulfatecontaining sample material. The concentration of sulfur in the sample solution is determined by inductively coupled plasma optical emission spectrometry (ICP-OES) according to ISO 11885 or by ion chromatography (IC) as sulfate according to ISO 10304-1 .

5 Interferences

5.1 Inductively coupled plasma optical emission spectrometry (ICP-OES)

5.1.1 Spectral interferences

The compensation of spectral interferences is performed according to the manual of the manufacturer of the spectrometer.

NOTE 1 Spectral interferences can be caused by:

a) Overlapping with the emission line of another element;

b) Overlapping with molecular bands;

c) Unspecific background (for example scattering, recombinations).

Overlapping of lines can be compensated for by arithmetical correction of the raw data if applicable.

Correction factors are determined by measuring at least one undisturbed additional emission line of the interfering element and considering the relation of the intensity of this emission line to the emission line which interferes with the analyte element.

In the case of overlapping with molecular bands, an alternative emission line has to be chosen.

NOTE 2 Unspecific background is usually compensated for by measuring the background signal in the surroundings of the analyte emission line.

5.1.2 Physical interferences

To compensate physical interferences, suitable measures shall be taken.

NOTE Physical interferences occur as plasma interferences or as transport interferences. Both are caused by a different behaviour of the calibration solution compared to the sample solution based on a different chemical composition. Plasma interferences are caused by changes in plasma conditions, for example temperature distribution and electron density, leading to a changed excitation of emission lines. Transport interferences are caused by differences in physical characteristics of the calibration solution and sample solution, mainly density, viscosity and surface tension.

Some of the transport interferences can be reduced by using appropriate (peristaltic) pumps. Plasma interferences as well as transport interferences can be reduced significantly by using suitable reference lines of a reference element with the same concentration in both the calibration and analyte solution (internal standard). Other possibilities are to equalize the chemical composition of the calibration and sample solution as much as possible (matrix matching) or to use a standard addition procedure or a standard addition calibration procedure instead of the standard calibration procedure.

5.2 Ion chromatography (IC)

To avoid cross-interferences by additional anions, suitable measures shall be taken.

NOTE Anions like chloride, bromide, fluoride, nitrite, nitrate, formiate and acetate in the absorption liquid may lead to cross-interferences. By using the chromatographic parameters given in Annex B, the sulfate signal usually can be separated completely from the signals of the other anions.

6 Apparatus

6.1 Combustion device, adjustable to (1 100 ± 20) °C, suitable for oxidizing the sample in an oxygen flow and suitable to absorb the reaction gases completely.

NOTE For an example of a suitable device see Annex A.

6.2 Porcelain boat, unglazed.

6.3 Gas-washing bottles, standard type, nominal volume 100 ml, filled with 25 ml of absorption solution (7.6 and Annex A).

6.4 Filtration adaptor, with a membrane filter with a pore width of 0,45 µm, connectable to the outlet of the disposable syringe (6.5).

6.5 Disposable syringe, nominal volume 5 ml.

6.6 Ultrasonic bath, preferably with a volume of ≥ 1 l and an ultrasonic power of ≥ 150 W.

6.7 Inductively coupled plasma optical emission spectrometer, sequential or simultaneous spectrometer with a lower wavelength limit of at least 180 nm.

6.8 Ion chromatograph with a column for anions and conductivity detector, if available with suppressor technique.

7 Reagents

7.1 General

Reagents of known analytical grade shall be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Hydrogen peroxide solution, H_2O_2, $w(H_2O_2)$ = 30 %.

7.3 Sodium hydroxide solution, NaOH, c(NaOH) = 0,001 mol/l.

7.4 Hydrochloric acid, HCl, w(HCl) = 18 %.

7.5 Ultrapure water, with a specific resistance of 18 MΩ · cm (at 25 °C).

7.6 Absorption solution, sodium hydroxide solution (7.3) and hydrogen peroxide solution (7.2) mixed in a ratio of 9:1.

7.7 Sulfate standard stock solution, certified single- or multi-element standard stock solution, preferably with a concentration of 1 000 µg/ml for sulfur.

7.8 Calibration solution, at least two calibration solutions have to be prepared by dilution of the standard stock solution (7.7) with water (7.5) according to the expected sulfur concentrations.

7.9 Eluent, exclusively for IC; for its use the instructions of the manufacturer have to be followed.

NOTE Depending on the column different eluents are used. For a suitable eluent see Annex B.

7.10 Oxygen, from a compressed gas cylinder, purity ≥ 99,998 % *V*/*V*.

8 Sampling and sample preparation

Sampling shall be performed in such a way that the sample to be analysed is representative for the total amount of material, for example according to ISO 8656-1[1].

The sample material shall have a particle size of ≤ 160 µm; if necessary, it shall be crushed and homogenized. Samples which incinerate completely can be used without crushing provided they fit into the porcelain boat (6.2).

In an unknown drying state, the sample shall be dried at (110 ± 5) °C to constant mass. The sample is cooled down to ambient temperature in a desiccator and stored therein.

It is important to avoid any contamination of the sample by improper handling (for example touching by fingers).

9 Preparation

9.1 Combustion device

The temperature of the combustion zone has to be adjusted to (1 100 ± 20) °C.

9.2 Oxygen (7.10)

Oxygen from the compressed gas cylinder is taken out by using a pressure-reduction valve. The oxygen flow is adjusted to (20 ± 10) l/h with a gas flow controller.

9.3 Inductively coupled plasma optical emission spectrometer (6.7)

Use the procedures recommended by the manufacturer of the instrument as set down in the operating procedure. The wavelength for sulfur at 180,731 nm and 182,034 nm should be used.

NOTE For recommendations for operating the inductively coupled plasma optical emission spectrometer (6.7), see Annex C.

9.4 Ion chromatograph (6.8)

Use the procedures recommended by the manufacturer of the instrument as set down in the operating procedure.

NOTE For recommendations for operating the ion chromatograph (6.8), see Annex B.

10 Calibration

10.1 Inductively coupled plasma optical emission spectrometer

The calibration shall be performed according to the manufacturer's manual using the calibration solutions according to 7.8. The concentration of the calibration solutions shall be adjusted according to the sulfur concentration in the sample solutions. The concentration of the sample solution shall be in the (quasi-)linear range of the calibration curve. If necessary the analysis solution shall be diluted.

10.2 Ion chromatograph

The calibration shall be performed according to the manufacturer's manual. At least a two-point calibration with the calibration solutions according to 7.8 shall be carried out.