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Second edition
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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

Analyse des matériaux céramiques — Dosage du soufre dans les produits et les matières premières céramiques non oxydes —

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

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC *33, Refractories*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 187, *Refractory products and materials,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 14720-2:2013), which has been technically revised.

The main changes are as follows:

— title of this document revised to better describe its field of application;

— Clause 1 adapted to the new title;

— subclause 9.3 provides additional information on suitable sulfur emission lines;

— in 9.3, 9.4, 11.1 and 11.2, NOTES were transferred into normative paragraphs;

— subclause 11.1 provides additional information on blank measurement;

— in 11.2, the error in the analysis process regarding the extraction of the residue after combustion has been corrected;

— in Clause 12, symbols for variables have been revised according to ISO/IEC requirements.

A list of all parts in the ISO 14720 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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 document specifies 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, e.g. 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, e.g. the adherent sulfur.

This document 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) can be considered as a practical value.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 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*

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

3 Terms and definitions

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

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

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-sulfate-containing sample material. The concentration of sulfur in the sample solution shall be determined by inductively coupled plasma optical emission spectrometry (ICP-OES) in accordance with ISO 11885 or by ion chromatography (IC) as sulfate in accordance with 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 shall be 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 shall be 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 shall 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 can 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 Figure A.1.

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) (see Figure A.1).

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