INTERNATIONAL STANDARD

ISO 14720-1

First edition
2013-03-01

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

Part 1: Infrared measurement methods

Essais des matières premières pour produits réfractaires — Dosage du soufre dans les matières premières non oxydantes sous forme de poudre et de granulés —

Partie 1: Méthodes d'essai par infrarouge

Reference number
ISO 14720-1:2013(E)

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Published in Switzerland

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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 14720-1 was prepared by Technical Committee ISO/TC 33, *Refractories.*

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

— *Part 1: Infrared measurement methods*

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

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

Part 1: Infrared measurement methods

1 Scope

This part of ISO 14720 defines a method for the determination of sulfur in powdered and granular non-oxidic ceramic raw materials and materials, such as silicon carbides, silicon nitrides, graphites, carbon blacks, cokes, carbon powders. If proved by the recovery rate, this method can also be applied for other non-metallic powdered and granular materials, e.g. silicon dioxide.

This part of ISO 14720 is applicable for materials with mass fractions of sulfur from 0,005 % to 2 %.

This part of ISO 14720 can also be applied for materials with higher mass fractions of sulfur after verification of the particular case.

2 Principle

The sample and added combustion accelerators (mostly tungsten- or iron-granules) are heated in an inductive furnace under oxygen atmosphere. The high-frequency field of the furnace couples with electrically conductive components of sample and combustion accelerators. The sample is heated to temperatures above 1 800 °C and the total sulfur content of the sample is released as sulfur dioxide. The reaction gas is transferred to the infrared absorption cell of the analyser. The molecular absorption of sulfur dioxide is measured by using a narrow-band optical filter which is translucent for the wavelength of the characteristic infrared absorption of sulfur dioxide. The mass fraction of sulfur dioxide in the reaction gas is proportional to peak-height and peak-area, respectively, of the transient absorption signal. The mass fraction of sulfur in the sample is calculated by using a calibration function established by suitable calibration standards measured under comparable conditions.

3 Apparatus

3.1 Device with induction furnace or alternatively **resistance furnace and infrared cell.**

NOTE The correctness of the analysis result can be proved by using matrix-analogous reference materials or by comparing with an independent alternative test method. If determining mass fractions below 100 mg/kg, it has to be considered that some analytical devices may deliver incorrect results.

3.2 Analytical balance, capable of reading to the nearest 0,01 mg.

3.3 Ceramic crucible, e.g. mullite or alumina.

3.4 Crucible lid with hole, e.g. mullite or alumina.

4 Reagents

4.1 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.

4.2 Tungsten granules

NOTE Depending on the material, the decomposition of the sample in the furnace may be improved by partially replacing tungsten granules by tin granules. Tungsten/tin-mixtures are commercially available.

4.3 Iron granules

4.4 Calibration standards, preferably certified reference materials with composition and sulfur content similar to the analysed material.

NOTE See Annex C. Also suitable are primary substances, preferably sulfates.

4.5 Oxygen, purity ≥ 99,998 % *V/V*.

5 Sampling and sample preparation

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

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

The sample material shall have a particle size of ≤ 160 µm. If necessary, it shall be crushed and homogenized.

6 Calibration

The calibration shall be performed according to the manufacturer's manual. It shall be ensured that the mass of sulfur in the calibration sample and test sample are within the same order of magnitude.

NOTE This is achieved by choosing a suitable calibration substance (4.4) and adapted masses.

For a mass fraction below 100 mg/kg, the trueness of the result shall be verified using a suitable reference material, preferably a certified reference material.

The calibration shall be carried out according to the procedure in 7.3.

7 Performance

7.1 Preparation of analysis

Ceramic crucibles (3.3) and lids (3.4) have to be pre-cleaned by heating to 1 200 °C for 1 h prior to analysis, preferably using a muffle furnace. The sulfur content of the iron granules (4.3) shall be determined batch-wise. In the case of a too high and irreproducible sulfur content of the iron granules in relation to the expected sulfur content in the sample, they shall be pre-cleaned by the following procedure: weigh 1 g of iron granules into each ceramic crucible used for analysis. Cover with a lid and melt the iron granules under oxygen atmosphere in the inductive furnace of the analytical device. If the sulfur content of the sample is less than 100 mg/kg, the iron granules can be alternatively purified by heating in hydrogen atmosphere at approximately 800 °C for 1 h.