BS ISO 10203:2017

BSI Standards Publication

Iron ores — Determination of calcium — Flame atomic absorption spectrometric method

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National foreword

This British Standard is the UK implementation of ISO 10203:2017. It supersedes BS ISO 10203:2015 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee ISE/58, Iron ores.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Iron ores — Determination of calcium — Flame atomic absorption spectrometric method

Minerais de fer — Dosage du calcium — Méthode par spectrométrie d'absorption atomique dans la flamme

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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.

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 documents 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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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 2, *Chemical analysis*.

This fourth edition cancels and replaces the third edition (ISO 10203:2015), of which it constitutes a minor revision with the following changes:

— in 5.11, minor editorial/grammatical amendments have been incorporated;

— in 6.1, "with a lid" has been inserted after "crucible";

— in 6.3 b), "the ratio between" has been inserted before "the slope" and minor editorial/grammatical amendments have been incorporated;

— in 7.2, reference to ISO 2596 has been incorporated and 7.2 has been rearranged in a more appropriate layout/format;

— in 8.2, reference to ISO 2596 has been incorporated;

— "predried" has been deleted where it is inappropriate in 8.1, 8.2, and 8.3.2;

— some references to the reagents in 8.4.2, 8.4.3, and Table 2 have been corrected;

— in the last line of 9.2.3, the formula has been corrected from $\left|\mu_1 - A_c\right| \leq P$ to $\left|\mu_1 - \mu_2\right| \leq P$;

— in 9.2.4, Formula (7) and the relevant descriptions have been modified to harmonize this subclause across all International Standards for which ISO/TC 102/SC 2 is responsible.

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INTERNATIONAL STANDARD ISO 10203:2017(E)

Iron ores — Determination of calcium — Flame atomic absorption spectrometric method

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1 Scope

This document specifies a flame atomic absorption spectrometric method for the determination of the mass fraction of calcium in iron ores.

This method is applicable to mass fractions of calcium between 0,010 % and 8,00 % in natural iron ores, iron ore concentrates, and agglomerates, including sinter products.

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 648, *Laboratory glassware — Single-volume pipettes*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 2596, *Iron ores — Determination of hygroscopic moisture in analytical samples — Gravimetric, Karl Fischer and mass-loss methods*

ISO 3082, *Iron ores — Sampling and sample preparation procedures*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 7764, *Iron ores — Preparation of predried test samples for chemical analysis*

ISO 80000-1:2009, *Quantities and units — Part 1: General*

3 Terms and definitions

No terms and definitions are listed in this document.

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

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

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

4 Principle

The test portion is decomposed by the treatment with hydrochloric acid and a small amount of nitric acid, and then evaporated to dehydrate silica, followed by dilution and filtration.

The residue is ignited and silica is removed by evaporation with hydrofluoric and sulfuric acids. The residue is fused with sodium carbonate and the cooled melt is dissolved in the filtrate.

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The solution obtained is aspirated into the flame of an atomic absorption spectrometer using a dinitrogen oxide-acetylene burner.

The absorbance values obtained for calcium are compared with those obtained from calibration solutions.

5 Reagents

During the analysis, use only reagents of recognized analytical grade and only water that complies with Grade 2 of ISO 3696.

5.1 Sodium carbonate, (Na_2CO_3), anhydrous.

5.2 Hydrochloric acid, (ρ 1,19 g/ml).

5.3 Hydrochloric acid, ρ 1,19 g/ml, diluted 1 + 9.

5.4 Nitric acid, ρ 1,4 g/ml.

5.5 Hydrofluoric acid, ρ 1,13 g/ml, 40 % (mass fraction) or ρ 1,185 g/ml, 48 % (mass fraction).

5.6 Sulfuric acid, ρ 1,84 g/ml, diluted 1 + 1.

5.7 Pure iron, minimum purity 99,9 % (mass fraction), of mass fraction of calcium less than 0,000 2 %.

5.8 Background solution.

Dissolve 5,0 g pure iron (5.7) in 50 ml of hydrochloric acid (5.2) and oxidize by adding nitric acid (5.4) drop by drop. Evaporate until a syrupy consistency is obtained. Add 20 ml of hydrochloric acid (5.2) and dilute to 200 ml with water. Dissolve 17 g of sodium carbonate (5.1) in water, add carefully to the iron solution, and heat to remove carbon dioxide. Transfer the cooled solution to a 1 000 ml one-mark volumetric flask, dilute to volume with water, and mix.

5.9 Lanthanum chloride solution.

Dissolve 50 g of lanthanum chloride ($LaCl_3 \cdot 7H_2O$) (of mass fraction of calcium less than 0,002 %) in 50 ml of hydrochloric acid (5.2) and 300 ml of hot water. Cool and dilute to 1 l.

5.10 Calcium standard solution, 25 µg Ca/ml.

Dissolve 1,248 7 g of dried calcium carbonate ($CaCO_3$) [minimum purity 99,9 % (mass fraction), dried at 105 °C for 1 h] in 100 ml of hydrochloric acid (5.2), diluted 1 + 3. When dissolved, cool, transfer to a 1 000 ml one-mark volumetric flask, dilute to volume with water, and mix. Transfer 10 ml of this solution to a 200 ml one-mark volumetric flask, dilute to volume with water, and mix.

5.11 Calcium calibration solutions.

Using pipettes, transfer 2,0 ml, 5,0 ml, 10,0 ml, 15,0 ml, and 20,0 ml portions of calcium standard solution (5.10) to 200 ml volumetric flasks (Clause 6). Add 6 ml of hydrochloric acid (5.2), 60 ml of background solution (5.8), and 40 ml of lanthanum chloride solution (5.9) to each flask. Dilute the solution to volume with water and mix. Prepare a zero calcium calibration solution by transferring 60 ml of the background solution to a 200 ml volumetric flask. Then add 6 ml of hydrochloric acid (5.2) and 40 ml of lanthanum chloride solution (5.9). Dilute the solution to volume with water and mix.

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