
**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*

Sample Document

get full document from standards.iteh.ai



Sample Document

get full document from standards.iteh.ai



COPYRIGHT PROTECTED DOCUMENT

© ISO 2017, Published in Switzerland

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

Page

Foreword	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Principle	1
5 Reagents	2
6 Apparatus	3
7 Sampling and samples	3
7.1 Laboratory sample	3
7.2 Preparation of predried test samples	4
7.2.1 General	4
7.2.2 Method specified in ISO 2596	4
7.2.3 Method specified in ISO 7764	4
8 Procedure	4
8.1 Number of determinations	4
8.2 Test portion	4
8.3 Blank test and check test	5
8.3.1 Blank test	5
8.3.2 Check test	5
8.4 Determination	5
8.4.1 Decomposition of the test portion	5
8.4.2 Treatment of the residue	5
8.4.3 Preparation of the test solution	6
8.4.4 Adjustment of the atomic absorption spectrometer	6
8.4.5 Atomic absorption measurements	7
9 Expression of results	7
9.1 Calculation of mass fraction of calcium	7
9.2 General treatment of results	7
9.2.1 Repeatability and permissible tolerance	7
9.2.2 Determination of analytical result	8
9.2.3 Between-laboratories precision	8
9.2.4 Check for trueness	8
9.2.5 Calculation of final result	10
9.3 Oxide factor	10
10 Test report	10
Annex A (normative) Flowsheet of the procedure for the acceptance of analytical values for test samples	11
Annex B (informative) Derivation of repeatability and permissible tolerance formulae	12
Annex C (informative) Precision data obtained by international analytical trials	13
Bibliography	14

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 $|\mu_1 - A_c| \leq P$ to $|\mu_1 - \mu_2| \leq P$;
- 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.

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

WARNING — This document might involve hazardous materials, operations, and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

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.

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 ($\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$) (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.

6 Apparatus

Ordinary laboratory apparatus, including one-mark pipettes and one-mark volumetric flasks complying with the specifications of ISO 648 and ISO 1042, respectively, and the following.

All glassware, volumetric flask lids, and the platinum crucibles shall first be washed in hydrochloric acid (diluted 1 + 1) and then in water.

6.1 Platinum crucible with a lid, of minimum capacity 30 ml.

6.2 Muffle furnace, capable of maintaining a temperature of approximately 1 100 °C.

6.3 Atomic absorption spectrometer, equipped with a dinitrogen oxide/acetylene burner.

WARNING — Follow the manufacturer's instructions for igniting and extinguishing the dinitrogen oxide-acetylene flame to avoid possible explosion hazards. Wear tinted safety glasses whenever the flame is burning.

The atomic absorption spectrometer used in this method shall meet the following criteria.

- a) Minimum sensitivity: The absorbance of the most concentrated calcium calibration solution (see 5.11) shall be at least 0,3.
- b) Graph linearity: The ratio between the slope of the calibration graph covering the top 20 % of the concentration range (expressed as a change in absorbance) and the value of the slope for the bottom 20 % of the concentration range determined in the same way shall not be less than 0,7.
- c) Minimum stability: The standard deviation of the absorbance of the most concentrated calibration solution and that of the zero calibration solution, each being calculated from a sufficient number of repetitive measurements, shall be less than 1,5 % and 0,5 %, respectively, of the mean value of the absorbance of the most concentrated calibration solution.

The use of a strip chart recorder and/or digital readout device is recommended to evaluate criteria a), b), and c) and for all subsequent measurements.

NOTE Instrument parameters can vary with each instrument. The following parameters were successfully used in several laboratories and they can be used as guidelines. Solutions were aspirated into a dinitrogen oxide-acetylene flame of a premix burner.

In systems where the values shown below for gas flow rates do not apply, the ratio of the gas flow rates may still be a useful guideline.

— Hollow cathode lamp, mA	15
— Wavelength, nm	422,7
— Dinitrogen oxide flow rate, l/min	13,8
— Acetylene flow rate, l/min	6,6

7 Sampling and samples

7.1 Laboratory sample

For analysis, use a laboratory sample of –100 µm particle size which has been taken and prepared in accordance with ISO 3082. In the case of ores having significant contents of combined water or oxidizable compounds, use a particle size of –160 µm.

NOTE A guideline on significant contents of combined water and oxidizable compounds is incorporated in ISO 7764.