INTERNATIONAL STANDARD

ISO 11536

First edition
2015-08-01

Iron ores — Determination of loss on ignition — Gravimetric method

Minerais de fer — Détermination de la perte au feu — Méthode gravimétrique

Reference number
ISO 11536:2015(E)

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

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 meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 2, *Chemical analysis*.

Introduction

The measurement of loss on ignition (LOI) is a technique widely used in the iron ore industry. Ignition loss is the sum of contributions from the mass loss of volatile compounds water vapour, carbon dioxide and sulfides (due to the decomposition of goethite and carbonaceous materials), and the mass gain due to oxidation [Fe(II) to Fe_2O_3]. Its use is complementary to the determination of elemental or oxide concentrations. It serves to allow for an addition of the oxides, generated at the ignition temperature, and the LOI, to arrive at total (oxide + LOI). The determination of LOI is essential in sinter plant and blast furnace balance calculations, as it is used to calculate calcinated elemental concentrations.

Iron ores — Determination of loss on ignition — Gravimetric method

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

1 Scope

This International Standard specifies a gravimetric method for the determination of the loss in mass of fully-oxidized iron ores, when ignited at 1 000 °C.

This method is applicable to a concentration range of 1,0 % (m/m) to 10,0 % (m/m) loss on ignition in natural iron ores, iron ore concentrates and agglomerates.

The method is not applicable to the following:

a) Processed ores containing metallic iron (direct reduced iron);

b) Natural or processed ores in which the sulfur content is higher than 0,2 % (m/m);

c) Natural or processed ores in which the content of partially-oxidized iron is more than 1,0 % (m/m).

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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*

3 Principle

A test portion is heated in a muffle furnace at 1 000 °C for 60 min and cooled in a desiccator (5.4). The mass loss of the test portion is determined, and expressed as a mass fraction percentage relative to the original weight.

4 Reagents

During the analysis, use only reagents of recognized analytical grade.

4.1 Silica gel, dried at 105 °C for 4 h.

5 Apparatus

Ordinary laboratory apparatus and, in particular, the following:

5.1 Silica (porcelain or platinum crucibles), 15 ml to 25 ml capacity, with lids.

The crucibles and their lids shall be pre-conditioned in the muffle furnace at 1 000 °C for 60 min. Crucibles and lids shall be stored in the desiccator (5.4) prior to use.

If platinum crucibles used for LOI analysis are to be used for flux fusions, platinum ware should be thoroughly cleaned to prevent cross-contamination.

5.2 Balance, capable of reading to the nearest 0,1 mg at the mass load of the crucible.

5.3 Muffle furnace, capable of maintaining a temperature of 1 000 °C ± 25 °C, with provision for air circulation adequate to prevent water vapour retention.

5.4 Vacuum desiccator, of borosilicate glass, 150 mm to 250 mm internal diameter with a vacuum stopcock that will allow the equilibrium of air pressure.

The desiccator plate shall be metal, ceramic or similar that will not break when in contact with a crucible at 1 000 °C. The rim of the desiccator shall be lightly greased with silicon grease or petroleum jelly. 150 g to 200 g of silica gel (4.1) shall be replaced daily.

If platinum crucibles are used, metal desiccator plates should not be used.

6 Sampling and samples — Laboratory sample

For analysis, use a laboratory sample of minus 160 µm particle size which has been taken and prepared in accordance with ISO 3082.

7 Procedure

7.1 General

Thoroughly mix the laboratory sample and, taking multiple increments, extract a test sample in such a way that it is representative of the whole contents of the container.

Test samples shall be air-equilibrated.

7.2 Number of determinations

Carry out the analysis at least in duplicate in accordance with Annex A, independently, on each test sample.

NOTE The expression "independently" means that the second and any subsequent result is not affected by the previous result(s). For this particular analytical method, this condition implies that the repetition of the procedure is to be carried out either by the same operator at a different time, or by a different operator, including appropriate recalibration in either case.

7.3 Determination of hygroscopic moisture content

The hygroscopic moisture shall be determined in accordance with ISO 2596. Two test portions shall be taken simultaneously, one for the ignition loss determination and the other for the hygroscopic moisture determination.

7.4 Test portion

Taking several increments, weigh to the nearest 0,000 1 g, approximately (2,0 ± 0,2) g of the test sample (see 7.1).

7.5 Determination

Using crucible tongs, place the crucible (5.1) covered with the lid into the muffle furnace (5.3) at 1 000°C for 60 min ± 10 min, ensuring that there is no loose material on the floor of the muffle furnace that could possibly adhere to the crucible.

Remove the crucible from the furnace and place in the desiccator (5.4), and place the lid on the crucible immediately. Allow the crucible to cool for 30 min. Keep the lid on the crucible throughout the cooling period.

Release the vacuum slowly, then weigh the cooled crucible and lid to the nearest 0,000 1 g.

Transfer the test portion to the crucible and re-weigh the crucible and lid to the nearest 0,000 1 g.

Repeat the determination as described above.

8 Expression of results

8.1 Calculation of loss on ignition

The loss on ignition (*LOI*), expressed as a percentage by mass, is calculated to a dry basis as indicated below.

The total mass loss after ignition (TML_w), including moisture, is calculated from Formula (1):

$$TML_w\ \%\ (\mathrm{m/m}) = \frac{m_2 - m_3}{m_2 - m_1} \times 100 \tag{1}$$

where

m_1 is the mass of the ignited crucible and lid, in grams;

m_2 is the mass of the crucible and lid plus the air-equilibrated test portion, in grams;

m_3 is the mass of the crucible and lid plus test portion after ignition, in grams.

The loss on ignition (*LOI*), moisture corrected, is calculated from Formula (2)

$$LOI_w\ \%\ (\mathrm{m/m}) = \frac{\left(TML_w - A\right) \times 100}{100 - A} \tag{2}$$

where A is the hygroscopic moisture content as a percentage by mass, determined in accordance with ISO 2596.

8.2 General treatment of results

8.2.1 Repeatability and permissible tolerances

The precision of this analytical method is expressed by the following formulae:

$$S_d = 0,018\,25 + 0,001\,14X \tag{3}$$

$$S_L = 0,025 \tag{4}$$

$$R_d = 0,051\,64 + 0,003\,23X \tag{5}$$

$$P = 0,053\,02 + 0,005\,09X \quad (6)$$

where

X is the loss on ignition, expressed as a percentage by mass, of the sample;

S_d is the independent duplicate standard deviation;

S_L is the between-laboratories standard deviation;

R_d is the independent duplicate limit;

P is the permissible tolerance.

8.2.2 Determination of analytical result

Having computed the independent duplicate results according to Formula (1), compare them with the independent duplicate limit (R_d), using the procedure given in Annex A, to obtain the final laboratory result, μ.

8.2.3 Between-laboratories precision

Between-laboratories precision is used to determine the agreement between the final results reported by two laboratories. The assumption is that both laboratories followed the same procedure described in 8.2.2.

Compute the following quantity:

$$\mu_{12} = \frac{\mu_1 + \mu_2}{2} \quad (7)$$

where

μ_1 is the final result reported by laboratory 1;

μ_2 is the final result reported by laboratory 2;

μ_{12} is the mean of final results.

If $|\mu_1 - \mu_2| \leq P$, the final results are in agreement.

8.2.4 Check for trueness

The present method is not suitable to measure any certified reference material, unless it is of the same mineralogical type, and preferably of the same geological origin as the samples used in the test. If that is the case, the trueness of the analytical method may be checked in the same way as in other ISO methods. For this, calculate the analytical result (μ) for the CRM/RM using the procedures in 8.2.2, and compare it with the reference or certified value Ac. There are two possibilities:

a) $|\mu_C - A_C| \leq C$ in which case the difference between the reported result and the certified/reference value is statistically insignificant.

b) $|\mu_C - A_C| > C$ in which case the difference between the reported result and the certified/reference value is statistically significant.

where

μ_C is the final result for the certified reference material;

A_C is the certified/reference value for the CRM/RM;

C is a value dependent on the type of CRM/RM used.

NOTE Certified reference materials used for this purpose are to be prepared and certified in accordance with ISO Guide 35.

For a CRM certified by an inter-laboratory test program

$$C = 2\sqrt{\sigma_L^2 + \frac{\sigma_d^2}{n} + V(A_C)} \quad (8)$$

where

n is the number of replicate determinations carried out on the CRM/RM;

$V(A_C)$ is the variance of the certified value A_C (= 0 for a CRM certified by only one laboratory, see Note below).

NOTE This type of CRM is to be avoided unless it is known to have an unbiased certified value.

8.2.5 Calculation of final result

The final result is the arithmetic mean of the acceptable analytical values for the test sample, or as otherwise determined by the operations specified in Annex A, calculated the nearest 0,001 % and reported to the nearest 0,01 %, as follows:

a) where the figure in the fifth decimal place is less than 5, it is discarded and the figure in the fourth decimal place is kept unchanged;

b) where the figure in the fifth decimal place is 5 and there is a figure other than 0 in the sixth decimal place, or when the figure in the fifth decimal place is greater than 5, the figure in the fourth decimal place is increased by one;

c) where the figure in the fifth decimal place is 5 and the Figure 0 is in the sixth decimal place, the 5 is discarded and the figure in the fourth decimal place is kept unchanged if it is 0, 2, 4, 6, or 8 and is increased by one if it is 1, 3, 5, 7, or 9.

9 Test report

The test report shall include the following information:

a) name and address of the testing laboratory;

b) date of issue of the test report;

c) a reference to this International Standard, i.e. ISO 11536;

d) details necessary for the identification of the sample;

e) reference number of the result;

f) any characteristics noticed during the determination, and any operations not specified in this International Standard which may have an influence on the result, for either the test sample or the certified reference material(s).