TECHNICAL REPORT

ISO/TR 18230

First edition
2015-08-15

Iron ores — Determination of loss on ignition — Non-oxidised ores

Minerais de fer — Détermination de la perte au feu — Minerais non oxydés

Reference number
ISO/TR 18230:2015(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 such as water vapour, carbon dioxide and sulphides (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 — Non-oxidised ores

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

1 Scope

This Technical Report describes a gravimetric method for the determination of the loss in mass of non-oxidized iron ores, when ignited at 1 000 °C.

This method is applicable to a concentration range of a mass fraction of −3,0 % to 7,0 % loss on ignition in natural iron ores, iron ore concentrates and agglomerates, and sinters.

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 a mass fraction of 0,2 %;

c) internationally traded ores with combined water greater than 2,5 %.

NOTE 1 Loss on ignition can be used as an estimate of combined water.

NOTE 2 This method is intended for in-house use and is not intended for referee purposes.

2 Principle

A test portion is heated in a muffle furnace at 1 000 °C for 60 min and cooled in a desiccator. The decreased mass of the test portion is then measured.

3 Reagents

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

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

4 Apparatus

Ordinary laboratory apparatus and the following:

4.1 Silica, **porcelain** or **platinum crucibles**, 15 ml to 25 ml capacity, with lids.

The crucibles should be pre-conditioned in the muffle furnace at 1 000 °C for 60 min. Crucibles and lids should be stored in the desiccator (4.4) 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.

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

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

4.4 Desiccator, of borosilicate glass, 150 mm to 250 mm internal diameter with a vacuum stopcock that will allow the evacuation of air.

The desiccator plate should be metal or ceramic or similar that will not break when in contact with a crucible at 1 000 °C. The rim of the desiccator should be lightly greased with silicon grease or petroleum jelly. 150 g to 200 g of silica gel (3.1) should be replaced daily.

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

5 Sampling and samples

5.1 Laboratory sample

For analysis, use a laboratory sample of −100 µm particle size which has been taken and prepared in accordance with ISO 3082.

5.2 Preparation of test samples

Analyses may be performed using either predried test samples (prepared in accordance with ISO 7764).

6 Procedure

6.1 Number of determinations

Carry out the analysis at least in duplicate, using the procedure given in 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 are to be carried out either by the same operator at a different time, or by a different operator, including appropriate recalibration in either case.

6.2 Test portion

Taking several increments, weigh to the nearest 0,000 1 g, approximately 2,0 g ± 0,2 g of the test sample (5.2).

6.3 Determination

6.3.1 Pre-treatment of crucible

Using crucible tongs, place the crucible (4.1) into the muffle furnace (4.3) 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 to a desiccator (4.4), place the lid on the crucible immediately, and cool under vacuum 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 (m_1).

6.3.2 Determination of LOI

Transfer the test portion (6.2) to the crucible and re-weigh the crucible and lid to the nearest 0,000 1 g (m_2).

Place the crucible containing the test portion (m_2) into the muffle furnace (4.3) at 1 000 °C for 60 min ± 10 min.

Remove the crucible from the furnace to a vacuum desiccator (4.4), place the lid on the crucible immediately, and cool under vacuum 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 (m_3).

7 Expression of results

7.1 Calculation of loss on ignition

The loss on ignition *LOI*, expressed as a percentage by mass, is calculated Formula (1):

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

where

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

m_2 is the mass of the crucible, lid and dry test portion, in grams;

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

7.2 General treatment of results

7.2.1 Repeatability and permissible tolerances

The precision of this analytical method is expressed by the following formulae (see also Annex A):

$$S_d = 0{,}040 \quad (2)$$

$$S_L = 0{,}029\ 1\ X + 0{,}051\ 7 \quad (3)$$

$$R_d = 0{,}114 \quad (4)$$

$$P = 0{,}042\ 7\ X + 0{,}026\ 2 \quad (5)$$

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.