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Iron ores for shaft direct-reduction feedstocks — Determination of the low-temperature reduction-disintegration index and degree of metallization

Minerais de fer pour charges utilisées dans les procédés par réduction directe — Détermination de l'indice de désintégration par réduction à basse température et du degré de métallisation

Reference number
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Contents

Foreword

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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 of 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 www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 3, *Physical testing*.

This fourth edition cancels and replaces the third edition (ISO 11257:2015), which has been technically revised to include ISO 16878 as a reference for the metallic iron determination.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

This document concerns one of a number of physical test methods that have been developed to measure various physical parameters and to evaluate the behaviour of iron ores, including reducibility, disintegration, crushing strength, apparent density, etc. This method was developed to provide a uniform procedure, validated by collaborative testing, to facilitate comparisons of tests made in different laboratories.

The results of this test have to be considered in conjunction with other tests used to evaluate the quality of iron ores as feedstocks for direct reduction processes.

This document can be used to provide test results as part of a production quality control system, as a basis of a contract, or as part of a research project.

Iron ores for shaft direct-reduction feedstocks — Determination of the low-temperature reduction-disintegration index and degree of metallization

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

1 Scope

This document specifies a method to provide a relative measure for evaluating the degree of size degradation and degree of metallization of iron ores, when reduced under conditions resembling those prevailing in shaft direct-reduction processes.

This document is applicable to lump ores and hot-bonded pellets.

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 2597-1, *Iron ores — Determination of total iron content — Part 1: Titrimetric method after tin(II) chloride reduction*

ISO 2597-2, *Iron ores — Determination of total iron content — Part 2: Titrimetric methods after titanium(III) chloride reduction*

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

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

ISO 3310-2, *Test sieves — Technical requirements and testing — Part 2: Test sieves of perforated metal plate*

ISO 5416, *Direct reduced iron — Determination of metallic iron — Bromine-methanol titrimetric method*

ISO 16878, *Iron ores — Determination of metallic iron content — Iron(III) chloride titrimetric method*

ISO 11323, *Iron ore and direct reduced iron — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11323 apply.

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

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

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

4 Principle

The test portion is isothermally reduced in a rotating tube bed, at 760 °C, using a reducing gas consisting of H_2, CO, CO_2 and CH_4, for 300 min. The reduced product is sieved with a sieve having square openings of 3,15 mm. Then, it is analysed for total iron and metallic iron. The reduction-disintegration index is calculated as the mass percentage of material less than 3,15 mm. The degree of metallization is calculated from the chemical analysis results.

5 Sampling, sample preparation, and preparation of test portions

5.1 Sampling and sample preparation

Sampling of a lot and preparation of a test sample shall be in accordance with ISO 3082.

The size ranges for pellets shall be 50 % – 16,0 mm + 12,5 mm and 50 % – 12,5 mm + 10,0 mm.

The size ranges for lump ores shall be 50 % – 20,0 mm + 16,0 mm and 50 % – 16,0 mm + 10,0 mm.

A test sample of at least 2,0 kg, on a dry basis, of the sized material shall be obtained.

Oven-dry the test sample to constant mass at 105 °C ± 5 °C and cool it to room temperature before preparation of the test portions.

NOTE Constant mass is achieved when the difference in mass between two subsequent measurements becomes less than 0,05 % of the initial mass of the test sample.

5.2 Preparation of test portion

Collect each test portion by taking ore particles at random.

NOTE Manual methods of division recommended in ISO 3082, such as riffling, can be applied to obtain the test portions.

At least four test portions, each of approximately 500 g (±the mass of 1 particle), shall be prepared from the test sample.

Weigh the test portions to the nearest 1 g and register the mass of each test portion on its recipient label.

6 Apparatus

The usual laboratory apparatus and, in particular, the following shall be used.

6.1 Ordinary laboratory equipment, such as an oven, hand tools, time-control device and safety equipment.

6.2 Reduction tube, without lifters, made of non-scaling, heat-resistant metal to withstand temperatures higher than 760 °C and resistant to deformation.

The internal diameter shall be 130 mm ± 1 mm and its internal length shall be 200 mm. A dust collector shall be connected to the tube to trap any fine particles carried in the gas stream out of the tube during the test.

6.3 Furnace, having a heating capacity and temperature control able to reach the test temperature within 90 min and to maintain the entire test portion, as well as the gas entering the bed, at 760 °C ± 5 °C.

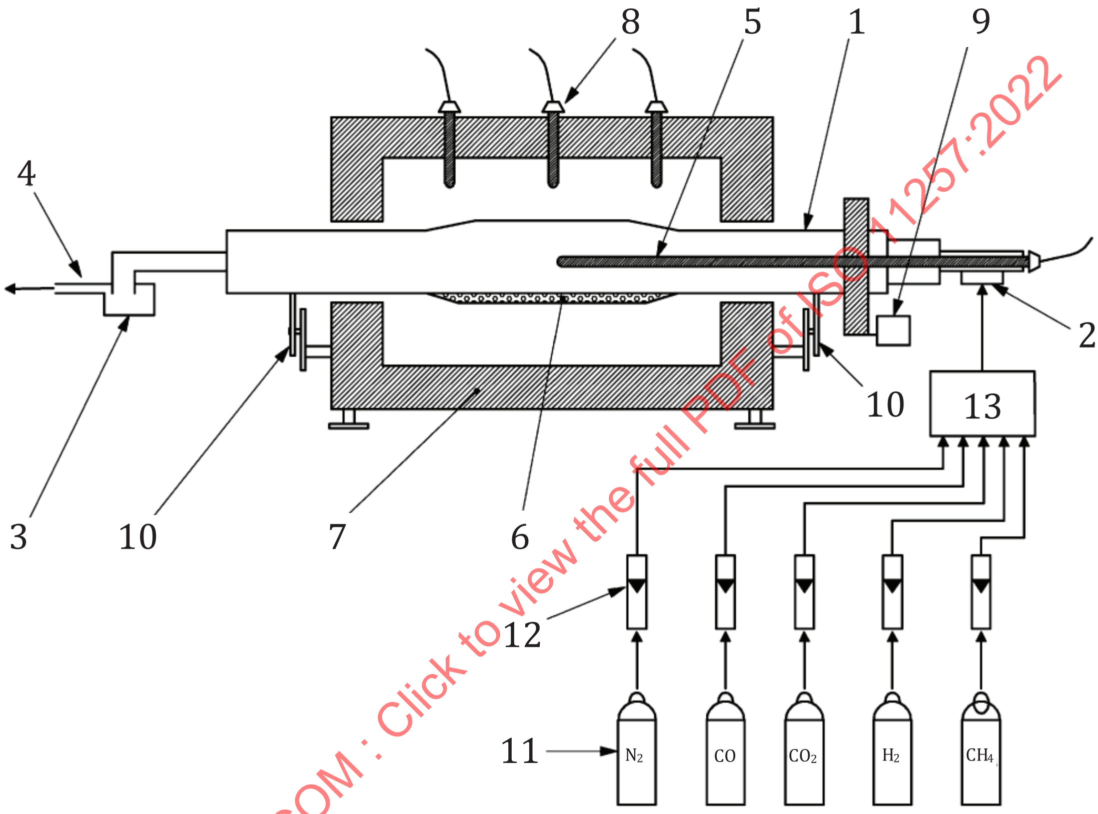
6.4 Rotation equipment, capable of rotation the reduction tube at a constant rate of 10 r/min ± 1 r/min.

6.5 **Gas-supply system**, capable of supplying the gases and regulating gas flow rates.

6.6 **Test sieves**, conforming to ISO 3310-1 or ISO 3310-2 and having mesh apertures of the following nominal sizes: 10,0 mm and 3,15 mm.

6.7 **Weighing device**, capable of weighing the test sample and test portions to an accuracy of 0,1 g.

Figure 1 shows an example of the test apparatus.



Key

Reduction tube

1 reduction tube
2 gas inlet
3 dust collector
4 gas outlet
5 thermocouple for measuring the reduction temperature
6 test portion

Furnace

7 electrically heated furnace
8 thermocouple for temperature regulation of furnace
9 rotation equipment (electric motor)
10 tube-support wheels

Gas supply system

11 gas cylinders
12 gas flow meters
13 mixing vessel

Figure 1 — Example of test apparatus (schematic diagram)

7 Test conditions

7.1 General

Volumes and flow rates of gases used are as measured at a reference temperature of 0 °C and at a reference atmospheric pressure of 101,325 kPa (1,013 25 bar).

7.2 Reducing gas

7.2.1 Composition

The reducing gas shall consist of the following:

CO	36,0 % ± 1,0 % (volume fraction)
CO_2	5,0 % ± 1,0 % (volume fraction)
H_2	55,0 % ± 1,0 % (volume fraction)
CH_4	4,0 % ± 1,0 % (volume fraction)

7.2.2 Purity

Impurities in the reducing gas shall not exceed the following:

O_2	0,1 % (volume fraction)
H_2O	0,2 % (volume fraction)

7.2.3 Flow rate

The flow rate of the reducing gas, during the entire reducing period, shall be maintained at 13 l/min ± 0,5 l/min.

7.3 Heating and cooling gas

Nitrogen (N_2) shall be used as the heating and cooling gas. Impurities shall not exceed 0,1 % (volume fraction).

The flow rate of N_2 shall be maintained at 10 l/min until the test portion reaches 760 °C and at 13 l/min during temperature-equilibration period. During cooling, it shall be maintained at 10 l/min.

7.4 Temperature of the test portion

The temperature of the entire test portion shall be maintained at 760 °C ± 5 °C during the entire reducing period and, as such, the reducing gas shall be preheated before entering the test portion.

8 Procedure

8.1 Number of determinations for the test

Carry out the test as many times as required by the procedure in Annex A.

8.2 Reduction

Take, at random, one of the test portions prepared in 5.2 and place it in the reduction tube (6.2).

Insert the reduction tube into the furnace (6.3). Close the reduction tube, connect the thermocouple, ensuring that its tip is in the middle of the reduction tube, and connect the gas supply system.

By means of the rotation equipment (6.4), commence rotation of the reduction tube at 10 r/min ± 1 r/min.

Pass a flow of N_2 through the test portion at a rate of at least 10 l/min and immediately start heating. The heating rate shall be such that the test portion reaches 760 °C within 90 min. When the temperature approaches 760 °C, increase the flow rate of N_2 to 13 l/min and continue heating at 760 °C ± 5 °C for 30 min.

DANGER — Carbon monoxide, hydrogen and reducing gas, which contains carbon monoxide and hydrogen, are toxic and explosive, and therefore hazardous. Testing shall be carried out in a well-ventilated area or under a hood. Precautions should be taken for the safety of the operator, taking into account the safety codes of each country.

Introduce the reducing gas at a flow rate of 13 l/min ± 0,5 l/min to replace the N_2. After 300 min of reduction, stop the flow of the reducing gas, stop the rotation of the reduction tube and cool the reduced test portion to room temperature under N_2 at a flow rate of 10 l/min.

8.3 Sieving

Remove all the material carefully from the reduction tube, scraping, if necessary, to remove any material adhering to the tube wall. Remove from the reduced material any free carbon deposited during reduction (a magnet can be used).

Determine the mass of the reduced material (m_0) and hand sieve it with care on 10,0 mm and 3,15 mm sieves (6.6). Determine and record the mass of each fraction retained on 10,0 mm (m_1) and 3,15 mm (m_2) sieves to the nearest 0,1 g. The dry mass of dust trapped in the dust collector, and material lost during sieving, shall be considered to be part of the −3,15 mm fraction.

NOTE Equivalent mechanical sieving can be used provided that preliminary test programme is carried out according to ISO 3086, having as reference the hand sieving method.

Sieving results are influenced by the sieve shaker characteristics. Therefore, in cases in which two or more laboratories need to compare their results for commercial or research purposes, they should adjust the sieving conditions until they obtain identical results for the same test sample.

8.4 Chemical analysis

Pulverise the entire reduced material and determine its total iron content (w_t) in accordance with ISO 2597-1 or ISO 2597-2, and its metallic iron content (w_0) in accordance with ISO 5416 or ISO 16878.

NOTE 1 ISO 16878 is a more environmentally friendly and safer method than ISO 5416, which uses mercury chloride and bromo methanol as reagents.

NOTE 2 If desired, the mass percentage of combined carbon in the reduced test portion can be determined by applying ISO/TR 9686.

9 Expression of results

9.1 Calculation of the reduction-disintegration index (D_{DR})

The reduction-disintegration index, D_{DR}, expressed as a mass percentage of material less than 3,15 mm, is calculated from Formula (1):

$$D_{DR} = \frac{m_0 - (m_1 + m_2)}{m_0} \times 100 \qquad (1)$$

where

m_0 is the mass, in grams, of the reduced test portion before sieving, including the dust trapped in the dust collector;

m_1 is the mass, in grams, of the fraction of the reduced test portion retained on the 10 mm sieve;

m_2 is the mass, in grams, of the fraction of the reduced test portion retained on the 3,15 mm sieve.

Record the result to one decimal place.

9.2 Calculation of the degree of metallization (*M*)

The degree of metallization, *M*, expressed as a percentage by mass, is calculated from Formula (2):

$$M = \frac{w_0}{w_t} \times 100 \quad (2)$$

where

w_0 is the metallic iron content, expressed as a percentage by mass, of the reduced test portion;

w_t is the total iron content, expressed as a percentage by mass, of the reduced test portion.

Record the result to one decimal place.

9.3 Repeatability and acceptance of test results

Follow the procedure in Annex A for each index, by using the repeatability value given in Table 1. The results shall be reported to one decimal place.

Table 1 — Repeatability (*r*)

Index	*r* %
D_DR	$0{,}86 + 0{,}20\ \bar{D}_\mathrm{DR}$
M	$7{,}22 - 0{,}06\ \bar{M}$
NOTE $\bar{D}_\mathrm{DR}$ and $\bar{M}$ are the mean values of the results of D_DR and M, respectively.	

10 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 11257:—;

b) all details necessary for the identification of the sample;

c) the name and address of the test laboratory;

d) the date of the test;

e) the date of the test report;

f) the signature of the person responsible for the test;

g) the details of any operation and any test conditions not specified in this document or regarded as optional, as well as any incident which could have had an influence on the results;