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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

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 11257 was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 3, *Physical testing*.

This second edition cancels and replaces the first edition (ISO 11257:1998), which has been revised to homogenise with other physical test standards.

Introduction

This International Standard 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 should be considered in conjunction with other tests used to evaluate the quality of iron ores as feedstocks for direct-reduction processes.

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

1   Scope

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

2   Normative references

The following referenced documents are indispensable for the application 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 2597-1:2006, *Iron ores — Determination of total iron content — Part 1: Titrimetric method after tin (II) chloride reduction*

ISO 3082:2000[1]), *Iron ores — Sampling and sample preparation procedures*

ISO 3310-1:2000, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

ISO 3310-2:1999, *Test sieves — Technical requirements and testing — Part 2: Test sieves of perforated metal plate*

ISO 4701:—[2]), *Iron ores and direct reduced iron — Determination of size distribution by sieving*

ISO 5416:2006, *Direct reduced iron — Determination of metallic iron — Bromine-methanol titrimetric method*

ISO 9507:1990, *Iron ores — Determination of total iron content — Titanium (III) chloride reduction methods*

ISO 11323:2002, *Iron ore and direct reduced iron — Vocabulary*

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1)   Under revision to incorporate ISO 10836, *Iron ores — Method of sampling and sample preparation for physical testing.*

2)   To be published. (Revision of ISO 4701:1999)

3 Terms and definitions

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

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 portions

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 4 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

6.1 General

The test apparatus shall comprise:

a) ordinary laboratory equipment, such as an oven, hand tools, time-control device and safety equipment;

b) a reduction tube assembly;

c) a furnace, with a system to rotate the reduction tube;

d) a system to supply the gases and regulate the flow rates;

e)   test sieves;

f)   a weighing device.

Figure 1 shows an example of a test apparatus.

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 rotating 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 the gas flow rates.

6.6   Test sieves, conforming to ISO 3310-1 or ISO 3310-2 and having square 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.

7   Test conditions

7.1   General

Volumes and flow rates of gases 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:

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:

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 $\pm$ 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 the 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 $\pm$ 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 $\pm$ 1 r/min.

Pass a flow of N_2 through the test portion at a rate of at least 10 L/min and immediately commence 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 $\pm$ 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, in accordance with the safety codes of each country.

Introduce the reducing gas at a flow rate of 13 L/min $\pm$ 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 on 10,0 mm and 3,15 mm sieves (6.6), in accordance with ISO 4701. 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 weight of dust trapped in the dust collector, and material lost during sieving, shall be considered to be part of the $-3,15$ mm fraction.

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 9507, and its metallic iron content (w_0) in accordance with ISO 5416.

NOTE   If desired, the mass percentage of combined carbon in the reduced test portion can be determined by applying ISO 9686:2006, *Direct reduced iron — Determination of carbon and/or sulfur — High-frequency combustion method with infrared measurement.*

9   Expression of results

9.1   Calculation of the reduction-disintegration index (RDI$_{DR}$)

The reduction-disintegration index, RDI$_{DR}$, expressed as a mass percentage of material less than 3,15 mm, is calculated from the following equation:

$$\text{RDI}_{DR} = \frac{m_0 - (m_1 + m_2)}{m_0} \times 100$$

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 the following equation:

$$M = \frac{w_0}{w_t} \times 100$$

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 %
RDI_{DR}	$0{,}86 + 0{,}20\,\overline{RDI_{DR}}$
M	$7{,}22 - 0{,}06\,\overline{M}$
NOTE $\quad \overline{RDI_{DR}}$ and $\overline{M}$ are the mean values of the results of RDI_{DR} and M, respectively.	

10 Test report

The test report shall include the following information:

a)   a reference to this International Standard, i.e. ISO 11257:2007;

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

h)   the reduction-disintegration index, RDI_{DR}, and degree of metallization, M;

i)   the sieving conditions, e.g. the method of sieving and the sieving time.

11 Verification

Regular checking of the apparatus is essential to ensure test result reliability. The frequency of checking is a matter for each laboratory to determine.

The conditions of the following items shall be checked:

— sieves;

— weighing device;

— reduction tube;

— tube-rotation equipment;

— temperature control and measurement devices;

— gas flow meters;