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Iron ores for blast furnace feedstocks — Determination of low-temperature reduction-disintegration indices by dynamic method

Minerais de fer pour charges de hauts fourneaux — Détermination des indices de désintégration par réduction à basse température, par méthode dynamique

[Revision of second edition (ISO 13930:2007)]

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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 13930 was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 3, *Physical testing*.

This third edition cancels and replaces the second edition (ISO 13930:2007) to contemplate the care needed during hand sieving, to introduce the mechanical sieving and to exclude the reference to ISO 4701.

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 blast furnace 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 blast furnace feedstocks — Determination of low-temperature reduction-disintegration indices by dynamic method

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 of iron ores when reduced under conditions resembling those prevailing in the low-temperature reduction zone of the blast furnace.

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 3082:2009, *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*

ISO 11323:2010, *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.

4 Principle

The test portion is isothermally reduced in a rotating tube bed, at 500 °C, using a reducing gas consisting of CO, CO_2, H_2 and N_2, for 60 min. The reduced product is sieved with sieves having square openings of 6,30 mm, 3,15 mm and 500 µm. Three low-temperature reduction-disintegration indices (LTD) are calculated as the mass percentage of material greater than 6,30 mm, less than 3,15 mm and less than 500 µm.

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 either - 16,0 mm + 12,5 mm or - 12,5 mm + 10,0 mm.

The size ranges for lump ores shall be - 12,5 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.

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 the test apparatus.

6.2 Reduction tube, made of non-scaling, heat-resistant metal to withstand temperatures higher than 500 °C and resistant to deformation. The internal diameter shall be 150 mm and the internal length shall be 540 mm. Four equally spaced steel angle lifters, 540 mm long × 20 mm high × 4 mm thick, shall be solidly attached longitudinally inside the tube by welding, in such a manner as to prevent accumulation of material between the lifter and tube. 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. The tube shall be replaced, in any case, when its wall thickness is reduced to 3 mm in any area, and the lifters when their height is reduced to less than 18 mm.

Figure 2 shows an example of a reduction tube.

6.3 Furnace, having a heating capacity and temperature control able to reach the test temperature within 45 min and to maintain the entire test portion, as well as the gas entering the test portion, at 500 ^{o}C ± 5 ^{o}C.

6.4 Rotation equipment, capable of rotation the reduction tube at a constant rate of 10 r/min ± 0,2 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 square apertures of the following nominal sizes: 6,30 mm; 3,15 mm; 500 µm.

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	20,0 % ± 0,5 % (volume fraction)
CO_2	20,0 % ± 0,5 % (volume fraction)
H_2	2,0 % ± 0,2 % (volume fraction)
CH_4	58,0 % ± 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 20 L/min ± 1 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 20 L/min until the test portion reaches 500 °C, during the temperature-equilibration period and during cooling.

7.4 Temperature of the test portion

The temperature of the entire test portion shall be maintained at 500 °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 ± 0,2 r/min.

Pass a flow of N_2 through the test portion at a rate of 20 L/min. Heat the test portion, bring the temperature inside the reduction tube to 500 °C within 45 min and stabilise the temperature within the next 15 min. If this requirement is not met, discontinue the test and start a new one.

DANGER — Carbon monoxide, hydrogen and the 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 20 L/min ± 1 L/min to replace the N_2. After 60 min of reduction, stop the flow of the reducing gas, stop the rotation of the reduction tube and cool the test portion to a temperature below 350 °C in the reduction tube under N_2 at a flow rate of 20 L/min. Then lift the reduction tube from the furnace and cool the test portion to below 100 °C, still under the flow of inert gas.

8.3 Sieving

Remove all the material carefully from the reduction tube, scraping, if necessary, to remove any material adhering to the tube wall.

Determine and register the mass of the reduced material (m_0) and sieve it with care on 6,30 mm, 3,15 mm and 500 µm sieves. Determine and record the mass of each fraction retained on the 6,30 mm (m_1), 3,15 mm (m_2) and 500 µm (m_3) sieves. The dry weight of the dust trapped in the dust collector, and material lost during sieving, shall be considered to be part of the − 500 µm fraction.

NOTE1 Equivalent mechanical sieving may be used provided that preliminary test programme is carried out according to ISO 3086, being the hand sieving the reference method.

NOTE2 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.

9 Expression of results

9.1 Calculation of the low-temperature disintegration indices ($LTD_{+6,3}$, $LTD_{-3,15}$ and $LTD_{-0,5}$)

The low-temperature disintegration indices $LTD_{+6,3}$, $LTD_{-3,15}$ and $LTD_{-0,5}$, expressed as percentages by mass, are calculated from the following equation:

$$LTD_{+6,3} = \frac{m_1}{m_0} \times 100$$

$$LTD_{-3,15} = \frac{m_0 - (m_1 + m_2)}{m_0} \times 100$$

$$LTD_{-0,5} = \frac{m_0 - (m_1 + m_2 + m_3)}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion after reduction, including the dust trapped in the dust collector;

m_1 is the mass, in grams, of the fraction retained on the 6,30 mm sieve;

m_2 is the mass, in grams, of the fraction retained on the 3,15 mm sieve;

m_3 is the mass, in grams, of the fraction retained on the 500 μm sieve.

Record each result to one decimal place.

9.2 Repeatability and acceptance of test results

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

Table 1 — Repeatability (*r*)

Mean value of *LTD* (%)		***r***
Over	Up to and including	%, absolute
98		—
93	98	2,0
88	93	2,5
12	88	3,0
7	12	2,5
2	7	2,0
0	2	—