INTERNATIONAL STANDARD

ISO 11257

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Iron ores — Determination of disintegration and metallization of feedstock for direct reduction by gas reforming processes

Minerais de fer — Détermination de la dégradation et de la métallisation des charges utilisées dans les procédés de reforming par réduction directe

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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.

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.

International Standard ISO 11257 was prepared by Technical Committee ISO/TC 102, *Iron ores*, Subcommittee SC 5, *Physical testing for direct reduction*.

Annex A forms an integral part of this International Standard. Annex B is for information only.

Introduction

Direct reduction processes are intended to reduce iron ores partially, or almost completely by thermal processes to form high grade feedstocks for iron- and steelmaking. Several kinds of direct reduction processes are in operation worldwide and others are still under development. The behaviour of the iron ores, as feedstock, may vary from process to process. ISO 11257 was prepared addressing specificaly the direct reduction by gas reforming processes.

The obtained proportion of generated fines is a relative measure of the disintegration behaviour and the degree of metallization is a measure of the metallization behaviour of the iron ore.

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

Iron ores — Determination of disintegration and metallization of feedstock for direct reduction by gas reforming processes

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 safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 Scope

This International Standard specifies a test method for evaluating the disintegration and metallization behaviour of iron ore pellets and lumps under conditions that resemble the ones prevailing in direct reduction by gas reforming processes, such as the Midrex-type process. [1)]

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 2597-1:1994, *Iron ores — Determination of total iron content — Part 1: Titrimetric methods after tin(II) chloride reduction.*

ISO 3310-1:— [2)], *Test sieves — Requirements and tests — Part 1: Metal wire cloth sieves.*

ISO 4701:— [3)], *Iron ores — Determination of size distribution by sieving.*

ISO 5416:1997, *Direct reduced iron — Determination of metallic iron content — Bromine-methanol titrimetric method.*

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

ISO 9508:1990, *Iron ores — Determination of total iron content — Silver reduction titrimetric method.*

ISO 10836:1994, *Iron ores — Method of sampling and sample preparation for physical testing.*

ISO 11323:1996, *Iron ores — Vocabulary.*

3 Definitions

For the purposes of this International Standard the definitions given in ISO 11323 and the following apply:

3.1 reduction-disintegration: Size degradation of lump ores or agglomerates resulting under reduction test conditions that resemble the ones prevailing in the direct reduction processes.

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1) Midrex is a trade-name used by Midrex International B.V. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the method named. Equivalent methods may be used if they can be shown to lead to the same results.

2) To be published. (Revision of ISO 3310-1:1990)

3) To be published. (Revision of ISO 4701:1985)

4 Principle

Isothermal reduction of the test portion in a rotation tube by reducing gas consisting of H_2, CO, CO_2 and CH_4 at temperature of 760 °C, over 300 min.

Heating and cooling in an inert atmosphere.

Sieving with test sieve having square opening of 3,15 mm.

Calculation of the reduction-disintegration index as the percentage mass of material less than 3,15 mm.

Calculation of the degree of metallization from the chemical analysis of the reduced material.

5 Apparatus

See figure 1.

Dimensions in millimetres

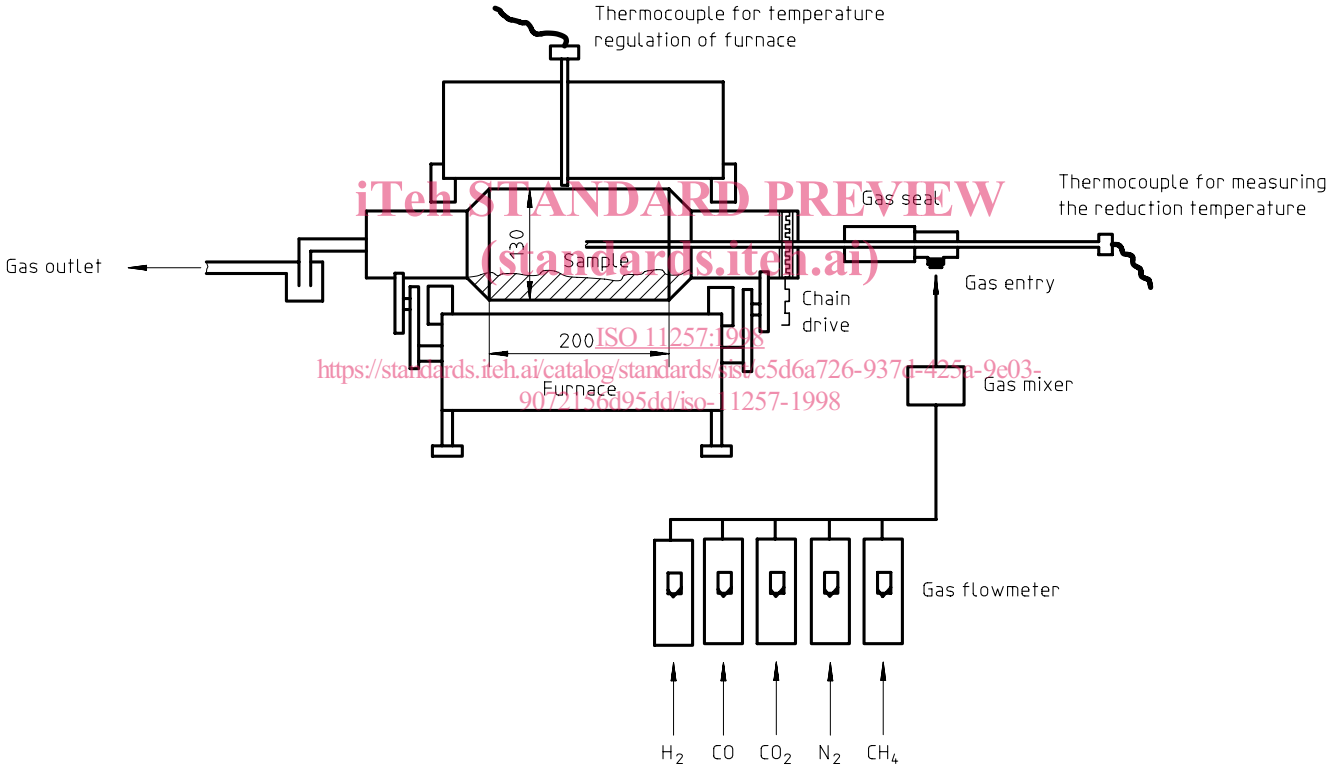

Figure 1 — Reduction-disintegration test apparatus

5.1 Gas supply system, capable of supplying the gases and regulating the gas flow rates.

5.2 Reduction tube, without lifters, made of non-scaling, heat-resisting metal to withstand a temperature greater than 760 °C. The internal diameter of the reduction tube shall be 130 mm ± 1 mm and the internal length 200 mm.

5.3 Furnace, having a heating capacity and controls sufficient to reach the test temperature within 90 min and to maintain the entire test portion at 760 °C ± 5 °C.

5.4 Rotation equipment, to permit rotation of the reduction tube at a constant rate of $0{,}167\ s^{-1} \pm 0{,}017\ s^{-1}$ (10 rpm ± 1 rpm).

5.5 Dust collector, to trap any fine solid particles carried in the gas stream out of the tube during the test.

5.6 Test sieves, conforming to ISO 3310-1 and having square openings of the following nominal aperture size: 20 mm; 16 mm; 12,5 mm; 10 mm and 3,15 mm.

5.7 Weighing device, capable of weighing the test portion and the reduced material to an accuracy of 0,1 g.

6 Test conditions

6.1 General

Volumes and flow rates of gases used in this International Standard are as measured at a temperature of 0 °C and at an atmospheric pressure of 101,325 kPa.

6.2 Composition of reducing gas

The reducing gas shall consist of:

H_2	55 % ± 1 % (*V*/*V*)
CO	36 % ± 1 % (*V*/*V*)
CO_2	5 % ± 1 % (*V*/*V*)
CH_4	4 % ± 1 % (*V*/*V*)

6.3 Purity of reducing gas

Impurities in the reducing gas shall not exceed:

O_2	0,1 % (*V*/*V*)
H_2O	0,2 % (*V*/*V*).

6.4 Flow rate of reducing gas

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

6.5 Purity of heating and cooling gas

Impurities in the nitrogen shall not exceed 0,1% (*V*/*V*).

6.6 Temperature of test

The reducing gas shall be preheated while entering the reduction tube to maintain the temperature within the reduction tube and hence the entire test portion at 760 °C ± 5 °C, during the entire reduction period.

7 Sampling and sample preparation

The sampling and the preparation of test samples and test portions shall be in accordance with ISO 10836. [4)]

The test sample shall be oven dried at 105 °C ± 5 °C and cooled to room temperature before the preparation of the test portions.

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4) ISO 10836:1994 does not yet include test sample preparation for this test method. Subclause 7.2.3 can be applied with the sieves adjusted accordingly.

At least six test portions, each of approximately 500 g mass shall be prepared.

Collect each test portion by taking ore particles at random. The target mass of the test portion is 500 g ± the mass of one particle.

NOTE - If the mass of the test portion deviates from 500 g, either add or remove particles one by one at random to reach a mass as close as possible to 500 g.

For example, if the mass of the test portion is 490 g and one more particle has a mass of 25 g, then the choice lies between 490 g and 515 g. The last particle should not be included in the test portion because the lower mass (490 g) is closer to the target mass (500 g) than the greater mass (515 g).

The size range for pellets shall be 10 mm to 16 mm, being 50 % between 10 mm and 12,5 mm and 50 % between 12,5 mm and 16 mm.

The size range for lumps shall be 10 mm to 20 mm, being 50 % of the mass between 10 mm and 16 mm and 50 % between 16 mm and 20 mm.

8 Procedure

8.1 Number of determinations

Carry out the test in duplicate on one ore sample.

8.2 Test portion

Take at random one test portion prepared as in clause 7.

8.3 Reduction

Place the test portion in the reduction tube (5.2) and close it. Connect the gas supply system (5.1).

Insert the reduction tube (5.2) in the furnace (5.3).

Pass a flow of nitrogen through the reduction tube at a rate of 10 l/min.

By means of the rotation equipment (5.4) commence rotation of the reduction tube at 0,167 s^{-1} (10 rpm) and immediately start the heating. The heating rate shall be such that the test portion reaches 760 °C within 90 min. When the temperature of the test portion approaches 760 °C, increase the flow rate of nitrogen to 13 l/min and continue heating at 760 °C for 30 min.

WARNING — Hydrogen, carbon monoxide and the reducing and waste gas which contains hydrogen and carbon monoxide are toxic and therefore hazardous. During the reduction the testing shall be carried out in a well ventilated area or under a hood. Precautions, according to local or national safety codes, shall be taken for safety of personnel and installations.

Introduce the reducing gas to replace the nitrogen, at a flow rate of 13 l/min. Perform the reduction for 300 min.

At the end of 300 min of reduction, stop rotation, turn off the power. Replace the reducing gas by nitrogen, at a flow rate of 10 l/min, and cool the reduced test portion to room temperature.

Remove the reduced test portion carefully from the reduction tube, scraping if necessary to remove any material adhering to the inside of the tube and separate any free carbon deposited during reduction (a magnet can be used).

Determine the mass of the reduced test portion and hand sieve it through 10,0 mm and 3,15 mm sieves (5.6), in accordance with ISO 4701. Determine and record the mass of each fraction to the nearest 0,1 g. The dry weight of dust trapped in the dust collector (5.5) and material lost during sieving shall be considered to be minus 3,15 mm fraction.

NOTE - Equivalent mechanical sieving may be used provided that preliminary test results give similar result to hand sieving within the permissible tolerance of 2 % absolute.

Pulverise the entire reduced test portion and determine its total iron in accordance with ISO 2597-1, ISO 9507 or ISO 9508, and its metallic iron, in accordance with ISO 5416.

NOTE - If desired, the mass percentage of carbon can be determined applying ISO 9686, to the reduced test portion.

9 Expression of results

9.1 Reduction-disintegration index (RDI_{DR})

The reduction-disintegration index RDI_{DR}, as a percentage, is calculated by the following formula:

$$RDI_{DR} = \frac{m_0 - (m_1 + m_2)}{m_0} \times 100 \quad ..(1)$$

where

m_0 is the mass, in grams, of the sieved reduced test portion (the material collected in the dust collector included);

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

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

9.2 Degree of metallization (M)

The degree of metallization M, is calculated by the following formula:

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

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.

The result shall be expressed to one decimal place.

9.3 Number of tests and permissible tolerance

The test shall be carried out in duplicate. The permissible tolerance, r, between the paired results (RDI_{DR1} and RDI_{DR2}) depends on their mean value ($\overline{RDI}_{DR}$; $\overline{M}$), as given by the equations in table 1.

Table 1 — Permissible tolerance (r)

Index	(95 % probability)
RDI_{DR}	$r = 0{,}86 + 0{,}20\ \overline{RDI}_{DR}$
M	$r = 7{,}22 - 0{,}06\ \overline{M}$