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Iron ores for shaft direct-reduction feedstocks — Determination of the low-temperature reductiondisintegration 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

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 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 <u>www.iso.org/members.html</u>.

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.

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Iron ores for shaft direct-reduction feedstocks — Determination of the low-temperature reductiondisintegration 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 11257:2022

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 <u>https://www.electropedia.org/</u>

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, CO₂ and CH₄, 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 \pm 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 standards.iteh.ai)

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 \pm 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 \pm 5 °C.

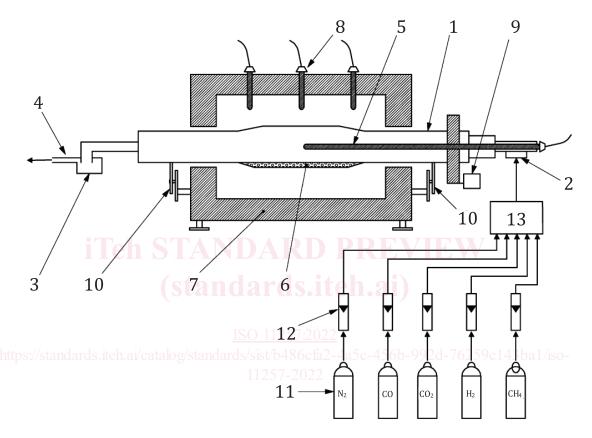
6.4 Rotation equipment, capable of rotation the reduction tube at a constant rate of 10 r/min ± 1 r/min.

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.



Кеу

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)
- CO₂ 5,0 % ± 1,0 % (volume fraction)
- H₂ 55,0 % ± 1,0 % (volume fraction)
- CH₄ 4,0 % ± 1,0 % (volume fraction)

7.2.2 Purity

Impurities in the reducing gas shall not exceed the following:

0₂ 0,1 % (volume fraction) (standards.iteh.ai

 H_2O 0,2 % (volume fraction)

7.2.3 Flow rate

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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 (N₂) 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 \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 <u>Annex A</u>.

8.2 Reduction

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