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Iron ore pellets for shaft direct-reduction feedstocks — Determination of the clustering index

Boulettes de minerais de fer pour charges utilisées dans les procédés par réduction directe — Détermination du pouvoir collant

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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 on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 3, *Physical testing*.

This third edition cancels and replaces the second edition (ISO 11256:2007), of which it constitutes a minor revision to contemplate the outcomes of the studies on mass definition and minor editorial improvements.

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

This International Standard 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 ore pellets for shaft direct-reduction feedstocks — Determination of the clustering index

CAUTION — This International Standard may involve hazardous operations and equipment. This International 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 formation of clusters of iron ore pellets when reduced under conditions resembling those prevailing in shaft direct-reduction processes.

This International Standard is applicable to hot-bonded pellets.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 9035, *Iron ores — Determination of acid-soluble iron(II) content — 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.

4 Principle

The test portion is isothermally reduced in a fixed bed at 850 °C under static load using a reducing gas consisting of H_2, CO, CO_2, and N_2 until a degree of reduction of 95 % is obtained. The reduced test portion (cluster) is disaggregated by tumbling using a specific tumble drum. The clustering index is calculated as the mass of clustered material accumulated after specified disaggregation operations.

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 range for pellets shall be 50 % - 16,0 mm + 12,5 mm and 50 % - 12,5 mm + 10,0 mm.

A test sample of at least 10 kg, on a dry basis, of sized pellets 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 five test portions, each of approximately 2 000 g (± the mass of one particle), shall be prepared from the test sample: four test portions for testing and one for chemical analysis.

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 the following:

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

b) a reduction tube assembly including a loading device;

c) a furnace equipped with a balance for permitting the mass loss of the test portion to be read at any time during the test;

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

e) a tumble drum;

f) a weighing device.

Figure 1 shows an example of the test apparatus.

6.2 Reduction tube, with a double wall made of non-scaling, heat-resistant metal to withstand temperatures higher than 850 °C and resistant to deformation. The internal diameter of the inner reduction tube shall be 125 mm ± 1 mm. A removable perforated plate made of non-scaling, heat-resistant metal to withstand temperatures higher than 850 °C shall be mounted in the reduction tube to support the test portion and to ensure uniform gas flow through it. The perforated plate shall be 10 mm thick with diameter 1 mm less than the internal diameter of the tube. The holes in the plate shall be 3 mm to 4 mm in diameter at a pitch centre distance of 5 mm to 6 mm. The internal diameter of the outer reduction tube shall be large enough to allow gas flow preheating before entering the inner reduction tube.

Figure 2 shows an example of a reduction tube.

6.3 Loading device, capable of supplying a total static load of 147 kPa ± 2 kPa evenly to the test portion. The load shall be transferred by means of a ram with rigid perforated foot plate so as to distribute it evenly to the surface of the porcelain balls placed on top of the test portion. The foot plate shall be 10 mm thick and its diameter shall be 1 mm less than the internal diameter of the tube. The holes in the plate shall be 3 mm to 4 mm in diameter at a pitch centre distance of 5 mm to 6 mm.

6.4 Porcelain balls, having a size range between 10,0 mm and 12,5 mm and of sufficient quantity to form two double-layer beds on the perforated plate.

6.5 Furnace, having a heating capacity and temperature control able to maintain the entire test portion as well as the gas entering the test portion at 850 °C ± 5 °C.

6.6 Balance, capable of weighing the reduction tube assembly, including the test portion, to an accuracy of 1 g. The balance shall have an appropriate device to suspend the reduction tube assembly.

6.7 Gas-supply system, capable of supplying the gases and regulating gas flow rates. It shall be ensured that a frictionless connection between the gas-supply system and the reduction tube does not affect the weight loss determination during reduction.

6.8 Tumble drum, made of steel plate at least 5 mm thickness, having an internal diameter of 1 000 mm, and an internal length of 500 mm. Two equally spaced L-shaped steel lifters 50 mm flat × 50 mm high × 5 mm thick and 500 mm long shall be solidly attached longitudinally inside the drum by welding so as to prevent the accumulation of material between lifter and drum. Each lifter shall be fastened so that it points towards the axis of the drum with its attached leg pointing away from the direction of rotation, thus providing a clear unobstructed shelf for lifting the sample. The door shall be so constructed as to fit into the drum to form a smooth inner surface. During the test, the door shall be rigidly fastened and sealed to prevent loss of the sample. The drum shall be rotated on stub axles attached to its ends by flanges welded so as to provide smooth inner surfaces. The drum shall be replaced in any case when the thickness of the plate is reduced to 3 mm in any area. The lifters shall be replaced when the height of the shelf is reduced to less than 47 mm.

Figure 3 shows an example of a tumble drum.

6.9 Drum rotation equipment, capable of ensuring that the drum attains full speed in one revolution, rotates at a constant speed of 25 r/min ± 1 r/min, and stops within one revolution. The equipment shall be fitted with a revolution counter and with an automatic device for stopping the drum after a predetermined number of revolutions.

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

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 (1,01 325 bar).

7.2 Reducing gas

7.2.1 Composition

The reducing gas shall consist of the following:

CO	30,0 % ± 1,0 % (volume fraction);
CO_2	15,0 % ± 1,0 % (volume fraction);
H_2	45,0 % ± 1,0 % (volume fraction);
N_2	10,0 % ± 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 40 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 20 L/min until the test portion reaches 850 °C and at 40 L/min during temperature-equilibration period. During cooling, it shall be maintained at 20 L/min.

7.4 Temperature of the test portion

The temperature of the entire test portion shall be maintained at 850 °C ± 5 °C during the entire reducing period and as such, the reducing gas shall be preheated before entering the test portion.

7.5 Loading of the test portion

After 60 min of reduction, the test portion shall be subjected to a constant load of 147 kPa ± 2 kPa applied over the surface of the bed.

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 Chemical analysis

Take, at random, one of the test portions prepared in 5.2 and use it for the determination of iron(II) oxide content (w_1) in accordance with ISO 9035 and the total iron content (w_2) in accordance with ISO 2597-1 or ISO 2597-2.

8.3 Reduction

In order to achieve a more uniform gas flow, place a double-layer bed of porcelain balls (6.4) in the reduction tube (6.2) on the perforated plate and level its surface. Place a ceramic-fibre wool along the inner wall of the reduction tube to avoid the test portion sticking to the wall.

Take, at random, another test portion prepared in 5.2 and record its mass (m_0). Place it in the reduction tube (6.2) and level its surface.

Place a further double layer of the porcelain balls upon the test portion.

Close the top of the reduction tube with the loading device (6.3). Insert the reduction tube assembly into the furnace (6.5) and suspend it centrally from the balance (6.6) ensuring that there is no contact with the furnace wall or heating elements.

Connect the thermocouple ensuring that its tip is in the centre of the test portion as shown in Figure 2.

Connect the gas-supply system (6.7) and the compressed air to the loading device.

Pass a flow of N_2 through the test portion at a rate of at least 20 L/min and commence heating. When the temperature of the test portion approaches 850 °C, increase the flow rate to 40 L/min. Continue heating while maintaining the flow of N_2 until the balance reading is constant and the temperature is constant at 850 °C for 10 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.

Tare the balance, start the time control device, and immediately introduce the reducing gas at a flow rate of 40 L/min to replace the N_2. Record the mass loss of the test portion (Δm_t) continuously or at least every 3 min for the first 15 min and thereafter at 10 min intervals.

After 60 min of reduction, apply a load of 147 kPa ± 2 kPa evenly to the test portion.

Calculate the degree of reduction, R_t, relative to the iron(III) state after t min as follows:

$$R_t = \left(\frac{0{,}111\, w_1}{0{,}430\, w_2} + \frac{\Delta m_t}{m_0 \times 0{,}430\, w_2} \times 100 \right) \times 100 \qquad (1)$$

where

m_0 is the mass, in grams, of the test portion;

Δm_t is the mass loss, in grams, of the test portion after reduction time, t;

w_1 is the iron(II) oxide content, as a percentage by mass, of the test sample prior to the test determined in accordance with ISO 9035. It is calculated from the iron(II) content by multiplying it by the oxide conversion factor, FeO/Fe(II) = 1,286;

w_2 is the total iron content, as a percentage by mass, of the test portion prior to the test determined in accordance with ISO 2597-1 or ISO 2597-2.

When the degree of reduction reaches 95 %, turn off the power and stop the flow of the reducing gas. Remove the load and record the time.

Introduce N_2 at a flow rate of 20 L/min until the test portion is below 50 °C.

8.4 Disaggregation

Carefully remove all the material from the reduction tube. Determine the mass of the reduced material (m_r). During this operation, some individual pellets usually separate from the clustered material. Remove the pellets and record the mass of the clustered material ($m_{c,1}$). This step is considered the first disaggregation operation.

The removal of the test portion from the reduction tube is a critical step and care should be taken to avoid its untimely disaggregation.

Place the clustered material inside the tumble drum (6.8) and rotate it for a total of 35 revolutions divided into seven disaggregation operations of five revolutions each. After each disaggregation operation, the mass of the remaining clusters is measured and recorded as a series ($m_{c,2}$, $m_{c,3}$, ... $m_{c,8}$). Any individual pellets that are separated from the clustered material shall be removed prior to the next disaggregation operation.