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Iron ores for blast furnace feedstocks — Determination of the reducibility by the rate of reduction index

Minerais de fer pour charges de hauts fourneaux — Détermination de la réductibilité à partir de la vitesse de réduction

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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.

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 4695 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 4695:1995), 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 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 the reducibility by the rate of reduction index

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 extent to and ease with which oxygen can be removed from iron ores, when reduced under conditions resembling those prevailing in the reduction zone of a blast furnace.

This International Standard is applicable to lump ores, sinters 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 9035:1989, *Iron ores — Determination of acid-soluble iron(II) content — 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*

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 950 °C, using a reducing gas consisting of CO and N_2, and is weighed at specified time intervals until its degree of reduction reaches 65 %. The rate of reduction is calculated at the oxygen/iron ratio of 0,9.

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

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, sinters and lump ores shall be − 12,5 mm + 10,0 mm.

A test sample of at least 2,5 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 5 test portions, each of approximately 500 g (± the mass of 1 particle) shall be prepared from the test sample: 4 test portions for testing and 1 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:

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

b) a reduction-tube assembly;

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 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 950 °C and resistant to deformation. The internal diameter shall be 75 mm ± 1 mm. A removable perforated plate, made of non-scaling, heat-resistant metal to withstand temperatures higher than 950 °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 4 mm thick, with its diameter 1 mm less than the tube internal diameter. The holes in the plate shall be 2 mm to 3 mm in diameter, at a pitch center distance of 4 mm to 5 mm.

Figure 2 shows an example of a reduction tube.

6.3 Furnace, having a heating capacity and temperature control able to maintain the entire test portion, as well as the gas entering the bed, at 950 °C ± 10 °C.

6.4 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 or support the reduction-tube assembly.

6.5 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.

6.6 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 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 40,0 % ± 0,5 % (volume fraction)

N_2 60,0 % ± 0,5 % (volume fraction)

7.2.2 Purity

Impurities in the reducing gas shall not exceed:

H_2 0,2 % (volume fraction)

CO_2 0,2 % (volume fraction)

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 50 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 25 L/min until the test portion reaches 950 °C, and at 50 L/min during the temperature-equilibration period.

7.4 Temperature of the test portion

The temperature of the entire test portion shall be maintained at 950 °C ± 10 °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 Chemical analysis

Take, at random, one of the test portions prepared in 5.2 and use it for the determination of the 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 9507.

8.3 Reduction

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

NOTE In order to achieve a more uniform gas flow, a double-layer bed of porcelain balls sized between 10,0 mm and 12,5 mm can be placed between the perforated plate and the test portion.

Close the top of the reduction tube. Connect the thermocouple, ensuring that its tip is in the centre of the test portion.

Insert the reduction tube into the furnace (6.3) and suspend or support it centrally from the balance (6.4) ensuring that there is no contact with the furnace wall or heating elements.

Connect the gas-supply system (6.5).

Pass a flow of N_2 through the test portion at a rate of 25 L/min and commence the heating. When the temperature of the test portion approaches 950 °C, increase the flow rate to 50 L/min. Continue heating while maintaining the flow of N_2, until the mass of the test portion is constant and the temperature is constant at 950 °C ± 10 °C for 15 min.

DANGER — Carbon monoxide and the reducing gas, which contains carbon monoxide, are toxic 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 according to the safety codes of each country.

Record the mass of the test portion (m_1) and immediately introduce the reducing gas at a flow rate of 50 L/min ± 0,5 L/min to replace the N_2. Record the mass of the test portion (m_t) at least every 3 min for the first 15 min and thereafter at 10 min intervals.

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

$$R_t = \left(\frac{0{,}111\, w_1}{0{,}430\, w_2} + \frac{m_1 - m_t}{m_0 \times 0{,}430\, w_2} \times 100 \right) \times 100$$

where

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

m_1 is the mass, in grams, of the test portion immediately before starting the reduction;

m_t is the mass, 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 9507.

When the degree of reduction reaches 65 %, turn off the power and stop the flow of the reducing gas. If, after 4 h, 65 % of oxygen loss has not been achieved, the reduction may be stopped. Introduce N_2 at a flow rate of 5 L/min for 5 min or more, to purge the reducing gas from the tube.

9 Expression of results

9.1 Calculation of the reducibility index $\left(\frac{dR}{dt}\right)$ (O/Fe = 0,9)

Prepare the reduction curve by plotting the degree of reduction R_t against time t.

Read off, from the reduction curve, the time, in minutes, to attain degrees of reduction of 30 % and 60 %.

The reducibility index, expressed as the rate of reduction at the atomic ratio of O/Fe of 0,9[2)] in %/min, is calculated from the following equation[3)]:

$$\frac{dR}{dt}(\text{O/Fe} = 0{,}9) = \frac{33{,}6}{t_{60} - t_{30}}$$

where

t_{30} is the time, in minutes, to attain a degree of reduction of 30 %;

t_{60} is the time, in minutes, to attain a degree of reduction of 60 %;

33,6 is a constant.

Record the result to 2 decimal places.

NOTE If a degree of reduction of 60 % is not attained in the test, lower values may be accommodated by the following equation:

$$\frac{dR}{dt}(\text{O/Fe} = 0{,}9) = \frac{k}{t_y - t_{30}}$$

where

t_y is the time, in minutes, to attain a degree of reduction of y %;

k is a constant depending on y.

If y = 50 % then the value of k = 20,2; if y = 55 % then the value of k = 26,5.

2) The atomic ratio O/Fe = 0,9 means a 40 % degree of reduction.

3) The derivation of the equation is given in Annex B.