SLOVENSKI STANDARD
SIST ISO 4695:1998

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Železove rude – Določanje stisljivosti

Iron ores -- Determination of reducibility

Minerais de fer -- Détermination de la réductibilité

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INTERNATIONAL STANDARD

ISO 4695

Second edition
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Iron ores — Determination of reducibility

Minerais de fer — Détermination de la réductibilité

Reference number
ISO 4695:1995(E)

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 4695 was prepared by Technical Committee ISO/TC 102, *Iron ores*, Subcommittee SC 3, *Physical testing*.

This second edition cancels and replaces the first edition (ISO 4695:1984), clauses 3, 5, 6, 7, 9 and 10 of which have been technically revised.

Annexes A and B of this International Standard are for information only.

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Introduction

The reducibility test method is one of several procedures used to evaluate the behaviour of natural and processed iron ores under specific conditions. The specific conditions involved in this test are: isothermal reduction; reduction in a fixed bed; reduction by means of carbon monoxide; a sample having a specified size range.

The results of this test should be considered in conjunction with the results of other tests, particularly those showing the physical behaviour of materials during reduction.

The mathematical derivation of formulae for reducibility is included for information only in annex A.

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Iron ores — Determination of reducibility

1 Scope

This International Standard specifies a method for determining the reducibility expressed as reduction rate of natural iron ores and agglomerates such as pellets or sinters.

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 method after tin(II) chloride reduction.*

ISO 3310-1:1990, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth.*

ISO 3310-2:1990, *Test sieves — Technical requirements and testing — Part 2: Test sieves of perforated metal plate.*

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 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.*

3 Definitions

For the purpose of this International Standard, the following definition applies.

3.1 reducibility: The ease with which oxygen combined with iron can be removed from natural or processed ores. [ISO 11323]

4 Principle

Isothermal reduction of the test portion at a specified size range in a fixed bed, at a temperature of 950 °C using a reducing gas consisting of CO and N_2.

Weighing of the test portion at specified time intervals.

Calculation of the degree of reduction relative to the iron(III) state and calculation of the rate of reduction at the oxygen/iron ratio of 0,9.

5 Reducing gas

Gas volumes and flow rates used in this International Standard are as measured at a temperature of 0 °C and at atmospheric pressure (101,325 kPa).

5.1 Composition

The reducing gas shall consist of:

CO	40 % (*V/V*) ± 0,5 % (*V/V*)
N_2	60 % (*V/V*) ± 0,5 % (*V/V*)

5.2 Purity

Impurities in the reducing gas shall not exceed:

H_2	0,2 % (*V/V*)
CO_2	0,2 % (*V/V*)
O_2	0,1 % (*V/V*)
H_2O	0,2 % (*V/V*)

Impurities in the heating gas (N_2) shall not exceed 0,1 % (*V/V*).

6 Apparatus

6.1 General

The apparatus shall comprise:

a) a system to supply and regulate the gases;

b) a reduction tube;

c) a weighing device to determine the oxygen loss at regular intervals;

d) a frictionless connection between the gas supply and reduction tube which ensures linearity of weight loss determination;

e) an electrically heated furnace to heat the test portion to the specified temperature;

f) test sieves.

Figure 1 shows a general arrangement of the apparatus.

Figure 2 shows an arrangement of the reduction tube and the furnace.

6.2 Reduction tube, made of non-scaling, heat-resistant metal to withstand temperatures of higher than 950 °C. The perforated plate is mounted in the reduction tube for supporting the test portion. The diameter of the reduction tube shall be 75 mm ± 1 mm.

Figure 3 shows an example of the reduction tube.

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

6.4 Weighing device, capable of weighing the load to an accuracy of 1 g. The weighing device shall be checked for sensitivity at regular intervals.

6.5 Test sieves, conforming to test sieve standard specifications and having square mesh apertures of the following nominal sizes and comforming to ISO 3310-1 or ISO 3310-2:

10,0 mm; 12,5 mm; 16,0 mm.

7 Preparation of test sample

7.1 General

In the case of a commercial test, the test sample shall be prepared according to ISO 10836. The test sample shall be oven dried at 105 °C ± 5 °C to constant mass and cooled to room temperature before testing. A quantity of test sample sufficient to provide at least five 500 g test portions shall be prepared.

7.2 Sample for reducibility test

The gross sample taken for testing is repeatedly divided, extracting masses for the reserve sample, the tumble test sample etc., until a stage is reached where the mass retained just exceeds the minimum required for the preparation of the test sample for reducibility.

7.2.1 Pellets

The division of the gross sample by riffle shall proceed until the mass retained just exceeds 30 kg. This sample shall be sieved on 12,5 mm and 10,0 mm test sieves, discarding the + 12,5 mm − 10,0 mm fractions and retaining the − 12,5 mm + 10,0 mm fraction. A minimum of 2,5 kg shall be used for the reducibility test sample. From the test sample, four test portions and one chemical analysis sample, each having a mass of 0,5 kg shall be obtained.

7.2.2 Sinters and ores

A test sample in the size range − 12,5 mm + 10,0 mm and of mass 30 kg shall be used. This shall be prepared by first sieving on a 12,5 mm sieve then carefully crushing the + 12,5 mm material until it all passes the 16,0 mm sieve. All fractions are combined and removed by sieving the + 12,5 mm − 10,0 mm material from the sample. From the test sample, four test portions and one chemical analysis sample, each having a mass of 0,5 kg shall be obtained.

7.3 Sample for chemical analysis

A 500 g test portion shall be reserved for the determination of total iron content and Fe(II) content.

8 Test conditions

8.1 Flow rate of reducing gas

The reducing gas (clause 5) flow rate shall, during the test period, be maintained at 50 l/min ± 0,5 l/min.

8.2 Temperature of test

The test portion shall be reduced at a temperature of 950 °C.

The reducing gas shall be preheated before entering the test portion to maintain the test portion at 950 °C ± 10 °C during the entire test period.

9 Procedure

9.1 Number of determinations

Carry out the test in duplicate on one ore sample.

9.2 Other determinations

Simultaneously with the test, determine the total iron content in accordance with ISO 2597, ISO 9507 or ISO 9508 and the Fe(II) content in accordance with ISO 9035.

9.3 Test portion

Weigh, to the nearest 1 g, approximately 500 g (± 1 particle) of the test sample (mass m_0).

9.4 Reduction

Place the test portion (9.3) in the reduction tube (6.2) so that the surface is even. In order to achieve a more uniform gas flow, a two-layer bed of porcelain pellets having a size range of 10,0 mm to 12,5 mm may be placed between the perforated plate and the test portion. Place the thermocouple in the centre of the test sample.

Close the top of the reduction tube. Insert the reduction tube into the furnace (6.3) and suspend it centrally from the weighing device (6.4) ensuring that there is no contact with the furnace or heating elements. Connect the gas supply.

Pass a flow of N_2 through the reduction tube at a flow rate of approximately 25 l/min and commence the heating. When the temperature of the test portion approaches 950 °C increase the flow rate of the N_2 to 50 l/min. Continue the heating whilst maintaining the flow of the N_2 until the mass of the test portion is constant (mass m_1) and the temperature is constant at 950 °C ± 10 °C.

CAUTION — Carbon monoxide and the reducing gas, which contains carbon monoxide, are toxic and therefore hazardous. During the following procedure, the testing shall be carried out in a well ventilated area or under a hood. Precautions, according to the safety codes of each country, should be taken for the safety of the operator.

Introduce the reducing gas to replace the N_2 at a flow rate of 50 l/min. Record the mass of the test portion at least every three minutes for the first 15 min and thereafter at 10 min intervals.

Terminate the reduction when the oxygen loss reaches 65 %. If, after 4 hours, this has not been achieved, the test may be stopped.

NOTE 1 If so desired, the test portion may then be cooled under a flow of N_2 to enable sample examination to be carried out.

10 Expression of results

10.1 Calculation of degree of reduction

Calculate the degree of reduction after time t, R_t, relative to the iron (III) state, as a percentage, by the following equation[1]:

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

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[2] is the iron (II) oxide content, as a percentage by mass, of the test sample prior to the test and is calculated from the iron (II) content by multiplying it by a factor of 1,286[3];

1) The derivation of the equation is given in annex A.

2) In the case of a commercial test, it is preferable to adopt w_1 and w_2, not for the 500 g test portion (7.3), but for consignment.

3) Oxide conversion factor FeO/Fe(II) = 1,286.