
**Iron ores — Dynamic test for
low-temperature reduction-disintegration**

*Minerais de fer — Essai dynamique de désagrégation par réduction
à basse température*



Content

1 Scope	1
2 Normative references	1
3 Definitions and abbreviations	1
4 Principle	2
5 Apparatus	2
6 Test conditions	3
7 Sampling and sample preparation	3
8 Procedure	4
9 Expression of results	5
10 Test report	5
11 Verification	6
Annex A (normative) Repeatability and number of tests	9
Annex B (informative) Bibliography	10

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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 13930 was prepared by Technical Committee ISO/TC 102, *Iron ores*, Subcommittee SC 3, *Physical testing*.

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

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Introduction

This International Standard describes a test method for evaluating the disintegration behaviour of iron ores reduced in a rotating tube under specific conditions. The proportions of coarse fines, generated fines and dust remaining are a measure of the degree of reduction-disintegration. The specific features of this test are:

- a) the placing of a test portion of a specified size in a rotating tube;
- b) isothermal reduction at a temperature of 500 °C;
- c) measurement of the disintegration of the test portion by sieving after the reduction.

The method provides an indication of the physical stability of blast furnace burden materials under conditions resembling those at the beginning of reduction under a weakly reducing atmosphere.

The results of this test should be considered in conjunction with the results of other reduction tests, particularly those conducted at high temperatures.

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Iron ores — Dynamic test for low-temperature reduction-disintegration

WARNING — This standard may involve hazardous materials, operations or equipment. It does not purport to address all the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of any regulatory limitations prior to use.

1 Scope

This International Standard specifies a method for testing the disintegration of iron ores during reduction in a rotating tube at a temperature of 500 °C.

The method is applicable to lump ores and pellets as blast furnace burden materials.

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 3082:1998, *Iron ores — Sampling and sample preparation procedures*.

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

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

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

3 Definitions and abbreviations

3.1 Definitions

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

3.1.1

low-temperature disintegration indices

Relative indications of the size degradation of pellets and lump ores under conditions prevailing in the upper part of a blast furnace shaft and obtained by a reduction test performed in a rotating tube. The indices are expressed as the percentages represented by the +6,3 mm sieve fraction, the –3,15 mm sieve fraction and the –0,5 mm sieve fraction, respectively.

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

3.2 Abbreviations

For the purposes of this International Standard, the following abbreviations apply:

LTD _{+6,3}	the disintegration index expressed in terms of the mass % of the +6,3 mm sieve fraction (the so-called disintegration strength)
LTD _{-3,15}	the disintegration index expressed in terms of the mass % of the -3,15 mm sieve fraction (the so-called disintegration index)
LTD _{-0,5}	the disintegration index expressed in terms of the mass % of the -0,5 mm sieve fraction (the so-called disintegration abrasion)

4 Principle

A test portion with a specified size range is reduced in a rotating tube at a temperature of 500 °C using reducing gas consisting of carbon monoxide (CO), carbon dioxide (CO₂), hydrogen (H₂) and nitrogen (N₂).

After 60 min reduction time, the test portion is cooled to a temperature below 100 °C and sieved with test sieves having square mesh apertures of 6,3 mm, 3,15 mm and 0,5 mm.

The low-temperature disintegration (LTD) values are calculated as a quantitative measure of the degree of disintegration of an iron ore that has been reduced while tumbling: the percentage masses of material greater than 6,3 mm, less than 3,15 mm and less than 0,5 mm, respectively, are related to the total mass of the test portion after reduction.

5 Apparatus

Figure 1 shows a schematic example of the low-temperature disintegration test apparatus.

Figure 2 shows an example of an installation which could be used to measure the temperature and the temperature distribution within the gas stream and the test portion in the reduction tube.

The test apparatus shall consist of the following main components:

5.1 Gas supply system, capable of supplying the gases and regulating the gas flow rates (flow meters indicated by 1 in figure 1).

5.2 Reduction tube (6 in figure 1), made of non-scaling, heat-resisting metal to withstand a temperature higher than 500 °C. The inside diameter of the reduction tube shall be 150 mm and its inside length 540 mm. Four equally spaced lifters 540 mm long, 20 mm wide and 4 mm thick shall be mounted longitudinally inside the tube.

5.3 Thermocouples, for measuring the furnace temperature (4 in figure 1) and the temperature within the reduction tube (2 in figure 1).

5.4 Furnace (7 in figure 1), having a heating capacity sufficient to reach the test temperature within 45 min and to maintain the entire test portion at 500 °C ± 5 °C.

5.5 Dust collector (11 in figure 1), to trap any fine particles carried out of the tube in the gas stream during the test.

5.6 Test sieves, having square mesh apertures of the following nominal sizes in accordance with ISO 3310-1:

16,0 mm; 12,5 mm; 11,2 mm; 10,0 mm; 6,3 mm; 3,15 mm and 0,5 mm.

5.7 Weighing device, capable of weighing the load to an accuracy of 0,1 g.

6 Test conditions

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)²⁾.

6.1 Composition of reducing gas

The reducing gas shall consist of:

CO	20 % ± 0,5 % (V/V)
CO ₂	20 % ± 0,5 % (V/V)
H ₂	2 % ± 0,2 % (V/V)
N ₂	58 % ± 1,0 % (V/V)

Impurities in the reducing gas shall not exceed:

O ₂	0,1 % (V/V)
H ₂ O	0,2 % (V/V)

6.2 Flow rate of reducing gas

The reducing-gas flow rate shall, during the test period, be maintained at 20 l/min ± 1 l/min.

6.3 Temperature of test

By appropriate design of the test rig, the reducing gas shall be preheated before entering the reduction tube to maintain the temperature of the test portion at 500 °C ± 5 °C during the entire test period.

7 Sampling and sample preparation

7.1 Sampling

The sample and test sample shall be taken in accordance with ISO 3082 and ISO 10836.

7.2 Test sample preparation

Oven-dry the test samples at 105 °C ± 5 °C for 2 h and cool to room temperature.

The total mass of the test sample shall be approximately 2 kg on a dry basis, being prepared as follows:

- a) Pellets, size 10,0 mm to 12,5 mm

The test sample in the size range of 10,0 mm to 12,5 mm shall be obtained by sieving and, after sieving, only pellets taken at random, e.g. by riffing, shall be used for the test.

- b) Pellets, size 12,5 mm to 16,0 mm

The test sample in the size range of 12,5 mm to 16,0 mm shall be obtained by sieving and, after sieving, only pellets taken at random, e.g. by riffing, shall be used for the test.

2) 1 mmHg = 0,133 3 kPa; 1 atm = 0,101 325 MPa

c) Lump ores

The test sample in the size range of 10,0 mm to 12,5 mm shall be prepared as follows:

Screen the sample on a 12,5 mm sieve and carefully crush the +12,5 mm material until it all passes the 16,0 mm sieve. Combine all the fractions and remove, by sieving, the +12,5 mm and the –10,0 mm material from the sample.

8 Procedure

8.1 Number of tests

Carry out the test at least in duplicate on each test sample.

8.2 Test portion

Weigh, to the nearest 1 g, approximately 500 g ($\pm$ the mass of one particle) of the test sample.

8.3 Reduction

Place the test portion in the reduction tube (5.2). Insert the reduction tube into the furnace and connect the thermocouple and the gas flow system to the reduction tube. Commence rotation of the reduction tube at $10 \text{ rpm} \pm 0,2 \text{ rpm}$.

Replace the air in the tube with inert gas. Heat the test portion and, while heating, pass a flow of inert gas through the reduction tube at a flow rate of approximately 20 l/min. Bring the temperature inside the reduction tube to 500 °C within 45 min and stabilize the temperature within the next 15 min. If this requirement is not met, discontinue the test and start a new one.

CAUTION — Carbon monoxide, hydrogen and the reducing gas which contains carbon monoxide and hydrogen are toxic and explosive and therefore hazardous. Precautions, in accordance with the safety codes of each country, shall be taken for the safety of the operator.

After a total time of 60 min, start the reduction. Introduce the reducing gas (6.1) at a flow rate of $20 \text{ l/min} \pm 1 \text{ l/min}$ to replace the inert gas and to reduce the test portion. Continue the reduction with the reducing gas for $60 \text{ min} \pm 1 \text{ min}$.

After 60 min reduction time, stop the flow of the reducing gas, stop the rotation of the reduction tube and cool the test portion to a temperature below 350 °C in the reduction tube under a flow (20 l/min) of inert gas. Then lift the reduction tube from the furnace and cool further, still under the flow of inert gas.

8.4 Sieving

At a temperature below 100 °C, which allows further handling, carefully remove all the material from the reduction tube. Add the dust trapped in the dust collector to this material. Determine the mass (m_0) to the nearest 1 g. Sieve mechanically on 6,3 mm, 3,15 mm and 0,5 mm sieves.³⁾ Determine and record the mass of each fraction retained on the 6,3 mm (m_1), 3,15 mm (m_2) and 0,5 mm (m_3) sieve. Material lost during sieving shall be considered to be –0,5 mm.

3) Sieving results are influenced by the sieve shaker characteristics. Therefore in cases in which two or more laboratories need to compare their results for commercial or research purposes, they should adjust the sieving conditions until they obtain identical results for the same test sample.

9 Expression of results

9.1 Calculation

The low-temperature disintegration indices $LTD_{+6,3}$, $LTD_{-3,15}$ and $LTD_{-0,5}$, expressed as a percentage by mass, shall be calculated to the first decimal place from the following equations:

$$LTD_{+6,3} = \frac{m_1}{m_0} \times 100$$

$$LTD_{-3,15} = \frac{m_0 - (m_1 + m_2)}{m_0} \times 100$$

$$LTD_{-0,5} = \frac{m_0 - (m_1 + m_2 + m_3)}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion after reduction, including the dust trapped in the dust collector;

m_1 is the mass, in grams, of the oversize fraction retained on the 6,3 mm sieve;

m_2 is the mass, in grams, of the oversize fraction retained on the 3,15 mm sieve;

m_3 is the mass, in grams, of the oversize fraction retained on the 0,5 mm sieve.

9.2 Permissible limits

If, for each of the LTD indices, the difference between the paired results lies within the permissible limits given in annex A, the test is finished. Report the mean value of the paired results.

If, for any of the LTD indices, the difference between the paired results does not lie within the permissible limits, follow the procedure given in annex A.

10 Test report

The test report shall include the following information:

- a reference to this International Standard;
- all details necessary for identification of the sample;
- the mass of the test portion before and after reduction;
- the sieving conditions, i.e. the type of sieving machine used, the kind of motion and the sieving time;
- the results of the test, i.e. the low-temperature disintegration indices expressed to one decimal place, plus the results of the individual determinations;
- details of any operation and any test conditions not specified in this International Standard or regarded as optional, as well as any incident which may have had an influence on the results;
- the name and address of the test laboratory;
- the signature of the person responsible for the test;
- the date of the test.

11 Verification

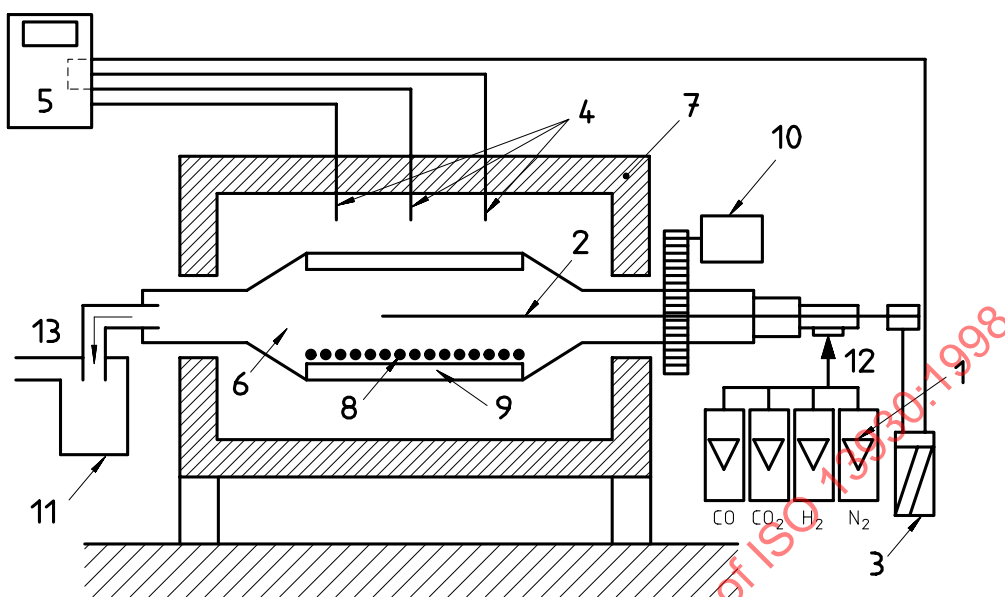
Regular checking of apparatus and procedures is essential to ensure repeatable results. Checks shall be carried out at regular intervals. The frequency of checking is a matter for each laboratory to determine. The following items shall be checked:

- reduction tube:
 - condition of lifters
 - speed of rotation
 - revolution counter
 - temperature and temperature distribution within the rotating tube
 - temperature and temperature distribution within the test portion
- sieves
- weighing device
- gas composition
- gas flow rate
- timer
- recording system

It is recommended that internal reference material is prepared and used periodically to check test repeatability.

Appropriate records of verification activities shall be maintained.

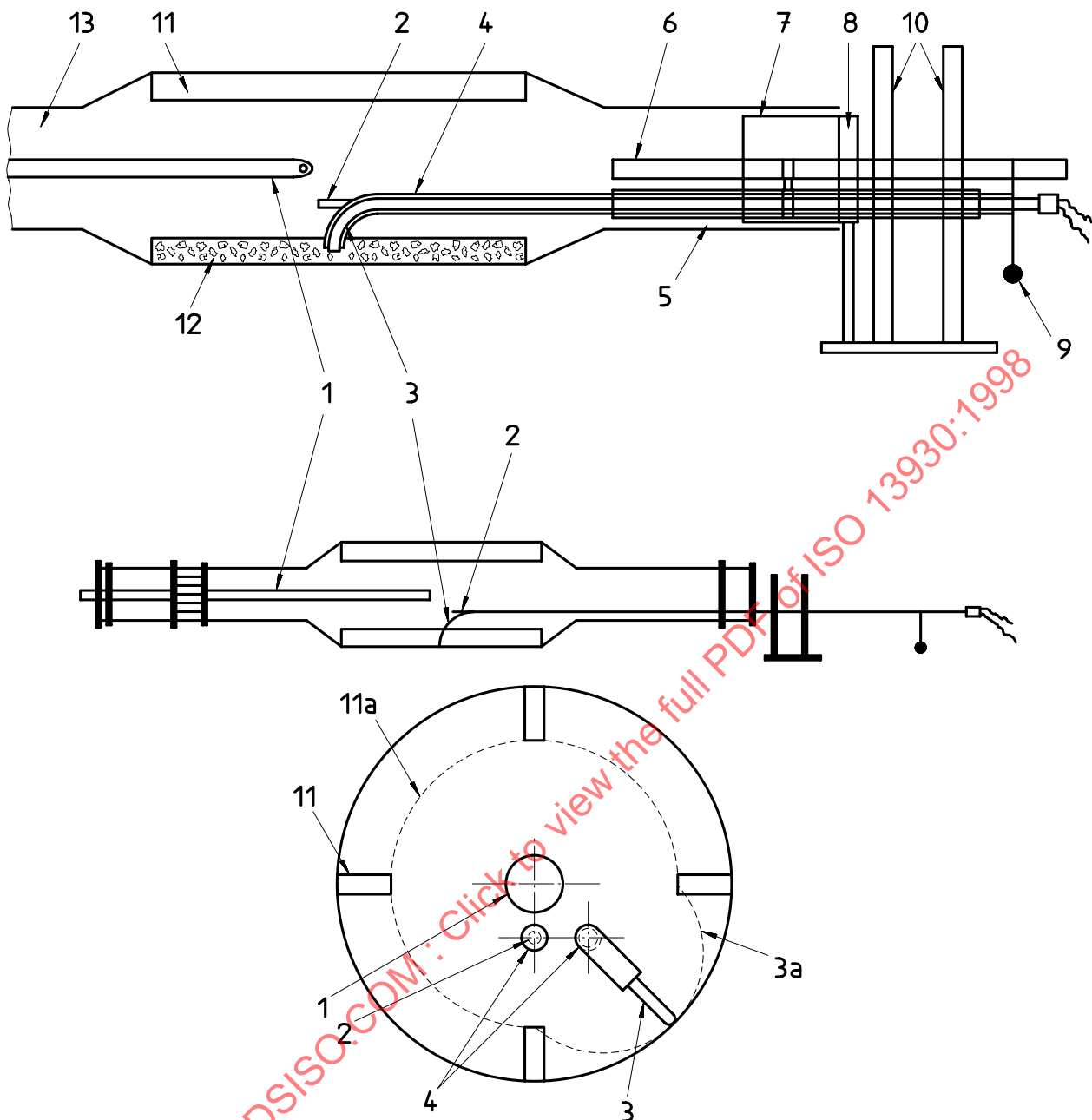
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Key

- 1 Gas flow meters
- 2 Thermocouple for measuring reduction tube temperature
- 3 Temperature recorder
- 4 Thermocouple for measuring furnace temperature
- 5 Temperature control unit
- 6 Reduction tube, 540 mm × 150 mm, four lifters
- 7 Furnace
- 8 Test portion
- 9 Lifter
- 10 Electric motor
- 11 Dust collector
- 12 Gas in
- 13 Gas out

Figure 1 — Example of low-temperature disintegration test apparatus



Key

- 1 Standard thermocouple (thermocouple 2 in figure 1)
- 2 Straight thermocouple, sheathed, 3 mm diameter
- 3 Curved thermocouple, sheathed, 3 mm diameter
- 3a Path traced by curved thermocouple
- 4 Support tubes (diam. 6 mm) for thermocouples 2 and 3
- 5 Sliding mounting (diam. 10 mm) for each support tube 4, designed to move along the reduction tube
- 6 Support tube (diam. 15 mm) for mounting 5, fitted with a scale
- 7 Fixation ring
- 8 Thermal insulation (ceramic wool)
- 9 Arm-mounted counterweight to compensate for thermocouple 3
- 10 Height-adjustable stand with baseplate
- 11 Four lifters
- 11a Path traced by lifters
- 12 Test portion
- 13 Gas inlet

Figure 2 — Example of an installation which could be used to measure the temperature and the temperature distribution within the gas stream and test portion in the reduction tube