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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

Reference number
ISO 11256:2007(E)

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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 11256 was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 3, *Physical testing*.

This second edition cancels and replaces the first edition (ISO 11256:1998), which has been revised to homogenise with other physical test standards.

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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 direct-reduction 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.

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Iron ore pellets for shaft direct-reduction feedstocks — Determination of the clustering 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 to 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 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 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 %. 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.

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

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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 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 2 000 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, 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 center 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.

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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 a rigid perforated footplate, so as to distribute it evenly to the surface of the porcelain balls placed on top of the test portion. The footplate 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 center 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 in 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 accumulation of material between the 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 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 30,0 % ± 1,0 % (volume fraction)

CO_2 15,0 % ± 1,0 % (volume fraction)

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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:

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 the 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 the iron(II) oxide content (w_1) in accordance with ISO 9035 and total iron content (w_2) in accordance with ISO 2597-1 or ISO 9507.

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.

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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.

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 mass of the test portion is constant and the temperature is constant at 850 °C for 10 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, in accordance with the safety codes of each country.

Record the mass of the test portion (m_1) and the time. Immediately introduce the reducing gas at a flow rate of 40 L/min to replace the N_2. Record the mass of the test portion (m_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:

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

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, 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 95 %, stop the flow of the reducing gas, remove the load and record the time.

Cool the reduced test portion to below 50 °C, under N_2, at a flow rate of 20 L/min.

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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 as 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 7 disaggregation operations of 5 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.

9 Expression of results

9.1 Calculation of the clustering index (CI)[2)]

The clustering index, CI, expressed as a percentage, is calculated from the following equation:

$$\mathrm{CI} = \frac{100}{8 \times m_r} \times \sum_{i=1}^{8} m_{c,i}$$

where

m_r is the total mass, in grams, of the test portion after reduction;

$m_{c,i}$ is the mass, in grams, of the clusters after the i^{th} disaggregation operations.

Record the result to one decimal place.

9.2 Repeatability and acceptance of test results

Follow the procedure of Annex A, by using the repeatability, $r = 0{,}27\ \overline{\mathrm{CI}}\,(\%)$, where $\overline{\mathrm{CI}}$ is the mean value of the clustering index of replicated determinations. The results shall be reported rounded off to the nearest whole number.

10 Test report

The test report shall include the following information:

a) a reference to this International Standard, i.e. ISO 11256:2007;

b) all details necessary for the identification of the sample;

c) the name and address of the test laboratory;

d) the date of the test;

2) Annex B (informative) gives an example of a calculation of the clustering index.

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e) the date of the test report;

f) the signature of the person responsible for the test;

g) 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;

h) the clustering index, CI;

i) the time to reach 95 % of reduction.

11 Verification

Regular checking of the apparatus is essential to ensure test result reliability. The frequency of checking is a matter for each laboratory to determine.

The conditions of the following items shall be checked:

— sieves;

— weighing device;

— reduction tube;

— temperature control and measurement devices;

— loading device;

— gas flow meters;

— purity of gases;

— time-control device;

— cleanliness of porcelain balls;

— tumble drum;

— drum-rotation equipment.

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

Appropriate records of verification activities shall be maintained.

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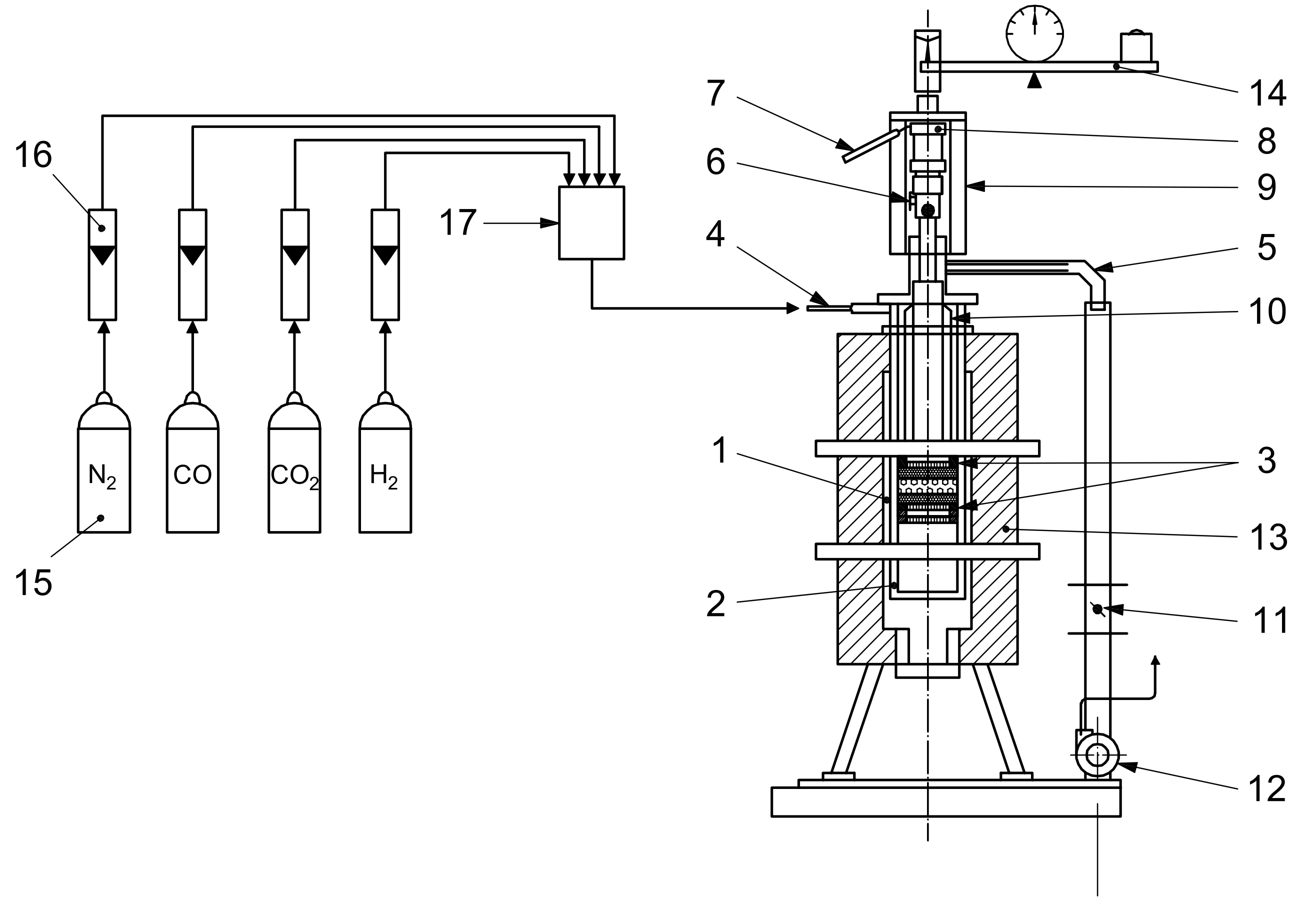

Key

Reduction tube

1 outer reduction tube

2 inner reduction tube

3 upper and lower perforated plates comprising test portion

4 gas inlet

5 gas outlet

6 thermocouple exit

Loading device

7 compressed air inlet

8 pressure cylinder

9 frame for pressure cylinder

10 loading ram

Waste gas

11 throttle valve

12 waste-gas fan

Furnace

13 electrically heated furnace

14 balance

Gas-supply system

15 gas cylinders

16 gas flowmeters

17 mixing vessel

Figure 1 — Example of test apparatus (schematic diagram)

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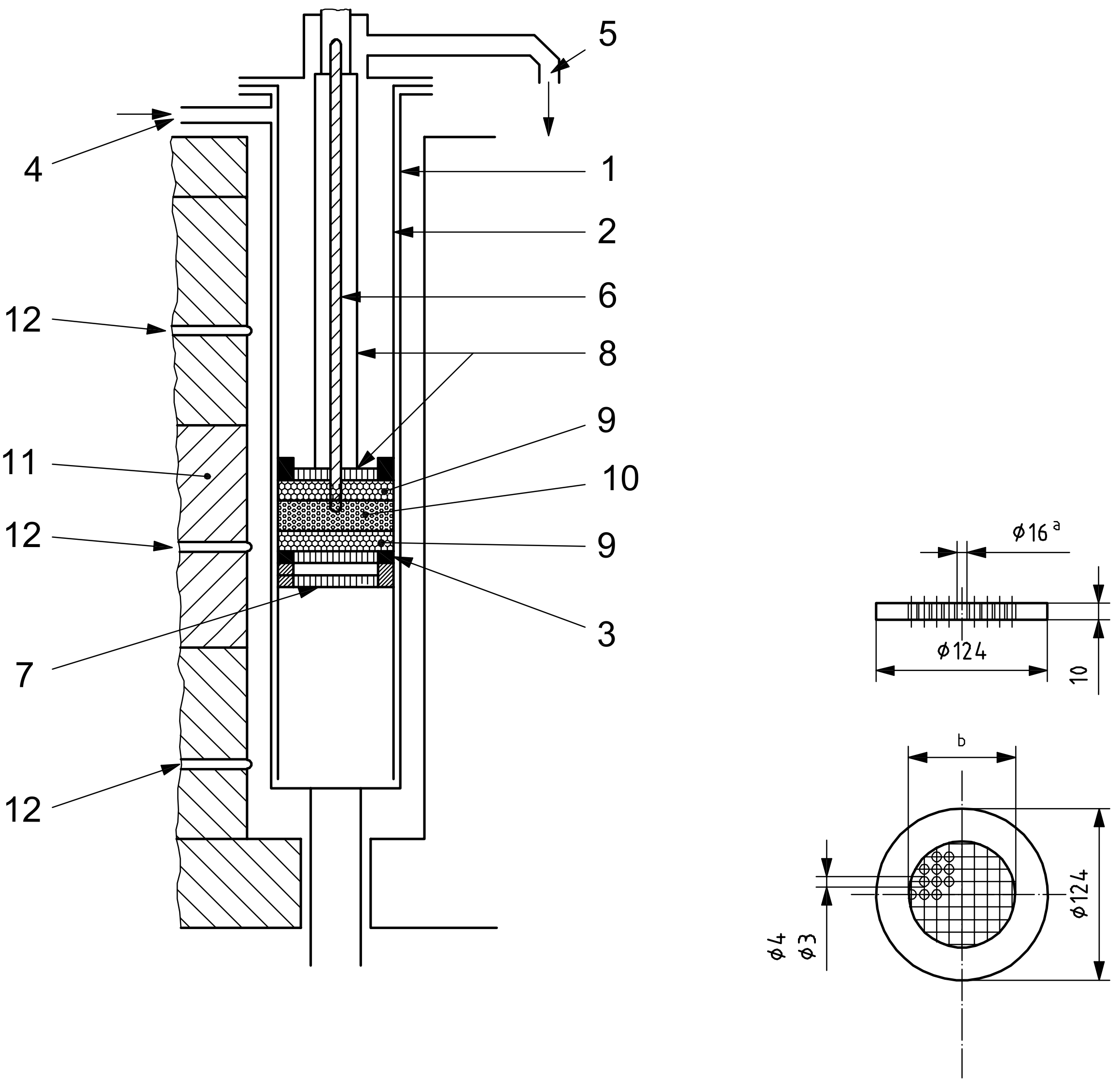
Dimensions in millimetres



Key

1 outer reduction tube
2 inner reduction tube (∅ 125 mm)
3 removable perforated plate
4 opening for gas inlet
5 opening for gas outlet
6 thermocouple for measuring reduction temperature
7 perforated support
8 loading ram with rigid perforated footplate
9 porcelain balls (2 layers)
10 test portion (2 000 g)
11 furnace wall
12 furnace-wall thermocouples (upper, medial and lower)

NOTE Key numbers do not coincide with the ones in Figure 1.

[a] For thermocouple entrance.

[b] 14 holes × 5 or 6 pitch.

Figure 2 — Example of reduction tube (schematic diagram)

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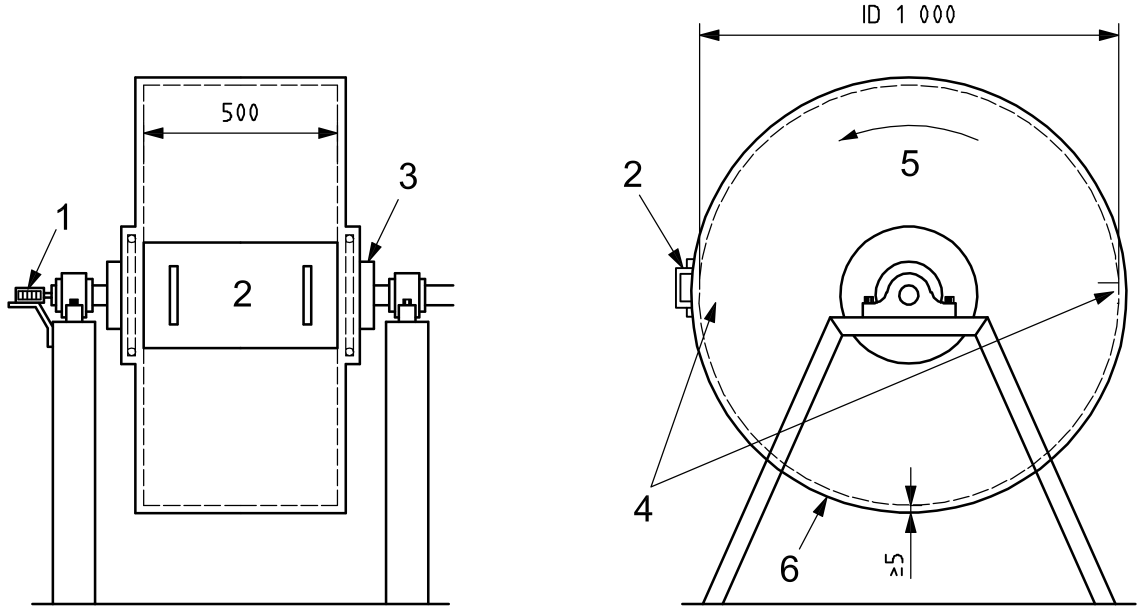
Dimensions in millimetres



a) Front view

b) Side view

Key

1 revolution counter
2 door with handle
3 stub axle (no through shaft)
4 two lifters (50 × 50 × 5)
5 direction of rotation
6 plate
ID internal diameter

Figure 3 — Example of test apparatus (schematic diagram)

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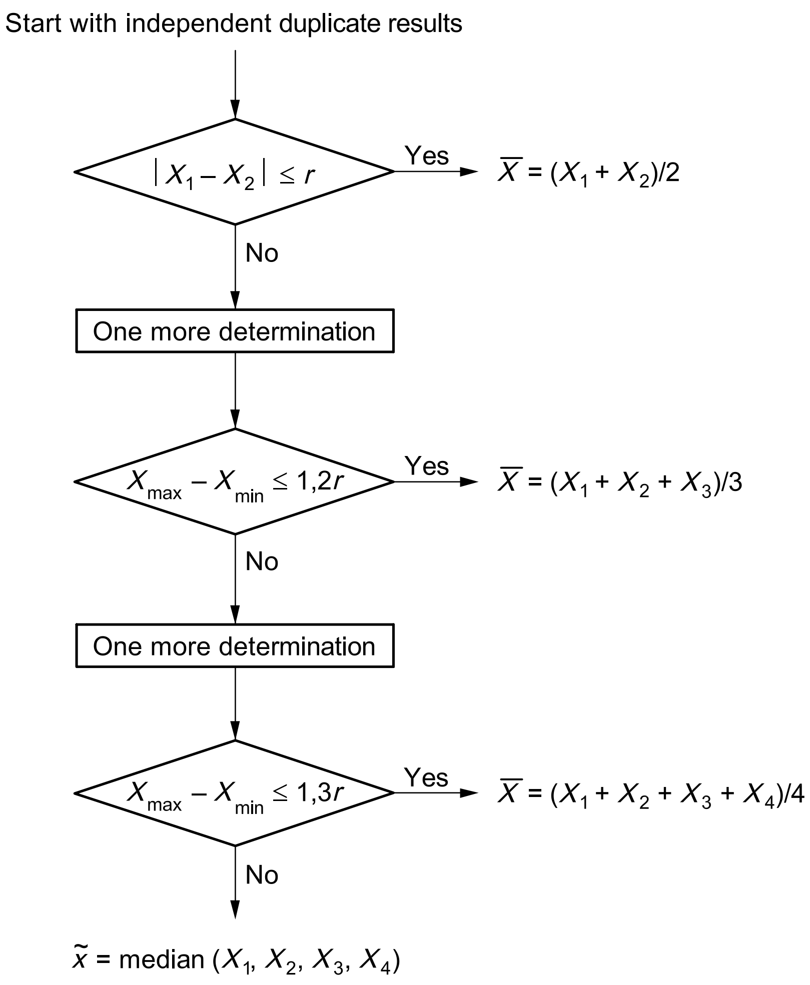
Annex A
(normative)

Flowsheet of the procedure for the acceptance of test results



r: see 9.2

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Annex B
(informative)

Example of a calculation of the clustering index

B.1 Suppose that the mass of a reduced test portion is $m_r = 1\ 442$ g, and that after the removal of the individual pellets separated from the clusters, the mass of the remaining cluster is $m_{c,1} = 1\ 430$ g.

Also suppose that after the disaggregation operations, the following set of results was obtained:

Table B.1

	Number of disaggregation operations, i	**Number of revolutions**	**Mass of clusters**, $m_{c,i}$ g
	1	zero	1 430
	2	5	700
	3	5	300
	4	5	150
	5	5	110
	6	5	80
	7	5	60
	8	5	40
TOTAL	8	35	2 870

The equation for the calculation of the clustering index (CI) is as follows:

$$\text{CI} = \frac{100}{8 \times m_r} \times \sum_{i=1}^{8} m_{c,i}$$

The clustering index for this test will be:

$$\text{CI} = \frac{100}{8 \times 1442} \times 2\,870 = 24{,}88 \cong 25\ \%$$

B.2 Suppose that, for the duplicate test, a value of CI = 32 % was obtained.

$$\overline{\text{CI}} = \frac{25 + 32}{2} = 28{,}5\ \%$$

The difference between the paired results is 7 %. According to 9.2, the permissible tolerance r is

$$r = \frac{27 \times 28{,}5}{100} = 7{,}7\ \%$$

The difference between the paired results is smaller than r. The mean of the paired results shall be reported as the clustering index.

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B.3 Suppose that, for the duplicate test, a value of CI = 53 % was obtained.

$$\overline{\text{CI}} = \frac{25 + 53}{2} = 39\ \%$$

The difference between the paired results is 28 %. According to 9.2, the permissible tolerance r is

$$r = \frac{27 \times 39}{100} = 10{,}5$$

The difference between the paired results is greater than the permissible tolerance r, and then further determinations shall be executed according to the procedure in Annex A.

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