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BRITISH STANDARD

BS ISO
4698:2007

Iron ore pellets for blast furnace feedstocks — Determination of the free-swelling index

ICS 73.060.10

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British Standards

National foreword

This British Standard is the UK implementation of ISO 4698:2007. It supersedes BS ISO 4698:1994 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee ISE/58, Iron ores.

A list of organizations represented on this committee can be obtained on request to its secretary.

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INTERNATIONAL STANDARD

ISO 4698

Second edition
2007-10-15

Iron ore pellets for blast furnace feedstocks — Determination of the free-swelling index

Boulettes de minerais de fer pour l'alimentation de hauts fourneaux — Détermination de l'indice de gonflement libre

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Reference number
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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 4698 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 4698:1994), 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 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.

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Iron ore pellets for blast furnace feedstocks — Determination of the free-swelling 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 increase in volume of iron ore pellets, when reduced in an unconstrained bed under conditions resembling those prevailing in the reduction zone of a blast furnace. It specifies the determination of the free-swelling index.

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 3082:2000[1)], *Iron ores — Sampling and sample preparation procedures*

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 volume of pellets in the test portion is measured at room temperature, using a volumetric apparatus. The test portion is isothermally reduced under unconstrained conditions in a fixed bed at 900 °C, using a reducing gas consisting of CO and N_2, for 60 min. The volume of the reduced pellets is measured at room temperature. The swelling index is calculated as the difference between the volume of the pellets after and before reduction, expressed as a percentage.

[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 − 12,5 mm + 10,0 mm.

A test sample of at least 1 kg, on a dry basis, of whole-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

At least 4 test portions, each one made up of 18 whole pellets, shall be obtained from the test sample by random selection.

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;

c) a furnace;

d) a test portion holder;

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

f) volumetric apparatus.

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 900 °C and resistant to deformation. The internal diameter shall be 75 mm ± 1 mm.

6.3 Test portion holder, a wire basket made of non-scaling, heat-resistant metal to withstand temperatures higher than 900 °C. It shall comprise three levels, each made to receive six pellets, for a total of 18 pellets. Alternatively, the test portion holder may be made by welding a tube to the centre of three perforated plates, mutually parallel and equally spaced. The tube shall be top-opened and bottom-closed to allow the thermocouple insertion up to the middle of the test portion. The set shall be made of non-scaling, heat-resistant metal to withstand temperatures higher than 900 °C. 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 centre distance of 4 mm to 5 mm.

Figure 2 shows an example of a reduction tube with the test portion holder.

6.4 Furnace, having a heating capacity and temperature control able to maintain the entire test portion, as well as the gas entering the bed, at 900 °C ± 10 °C.

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6.5 Gas-supply system, capable of supplying the gases and regulating gas flow rates.

6.6 Volumetric apparatus, capable of measuring the volume of the test portion to an accuracy of 0,2 mL.

Annex B shows examples of volumetric apparatus.

7 Test conditions

7.1 General

Volumes and flow rates of gases are 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 % ± 0,5 % (volume fraction)
N_2	70,0 % ± 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 15 L/min ± 1 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 10 L/min until the test portion reaches 900 °C, and at 15 L/min during the temperature-equilibration period. During cooling, it shall be maintained at 5 L/min.

7.4 Temperature of the test portion

The temperature of the entire test portion shall be maintained at 900 °C ± 10 °C during the entire reducing period and, as such, the reducing gas shall be preheated before entering the test portion.

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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 Reduction

Determine the volume of the test portion (V_0) to an accuracy of 0,2 mL, in accordance with one of the methods specified in Annex B.

Place 6 pellets on each of the 3 levels of the test portion holder (6.3) and place it in the reduction tube (6.2). Close the top of the reduction tube. Connect the thermocouple, ensuring that its tip is in the centre of the test portion. Close the top of the reduction tube and insert it in the furnace.

Connect the gas-supply system (6.5).

Pass a flow of N_2 through the test portion at a rate of at least 5 L/min and commence heating. When the temperature of the test portion approaches 900 °C, increase the flow to 15 L/min ± 1 L/min. After reaching 900 °C ± 10 °C, maintain the test portion under these conditions 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 ventilation hood. Precautions should be taken for the safety of the operator, according to the safety codes of each country.

Introduce the reducing gas at a flow rate of 15 L/min ± 1 L/min to replace the N_2. After 60 min of reduction, turn off the power.

NOTE Some pellets show a higher degree of swelling within a shorter reduction time than 60 min. Therefore, a shorter reduction time may be used as an alternative when appropriate, e.g. 40 min.

Replace the reducing gas with N_2 at a flow rate of 5 L/min. Remove the reduction tube from the furnace (6.4) while maintaining the flow of N_2 until the test portion reaches room temperature (below 50 °C).

8.3 Volume determination

Remove the test portion from the reduction tube and immediately determine and register the total volume of the test portion (V_1), applying the same method used for the determination of V_0.

9 Expression of results

9.1 Calculation of the free-swelling index (V_{FS})

The free-swelling index, V_{FS}, expressed as a percentage, is calculated from the following equation:

$$V_{FS} = \frac{V_1 - V_0}{V_0} \times 100$$

where

V_0 is the volume, in millilitres, of the test portion before reduction;

V_1 is the volume, in millilitres, of the test portion after reduction.

Record the result to one decimal place.

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9.2 Repeatability and acceptance of test results

Follow the procedure in Annex A by using the repeatability value, r = 3,0 (%, absolute). The results shall be reported to one decimal place.

10 Test report

The test report shall include the following information:

a) a reference to this International Standard, i.e. ISO 4698:2007;

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;

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 free-swelling index, V_{FS};

i) the time of reduction, if it is not 60 min;

j) the type of volumetric method employed.

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:

- reduction tube;
- temperature control and measurement devices;
- gas flow meters;
- purity of gases;
- recording system;
- time-control device;
- volumetric apparatus.

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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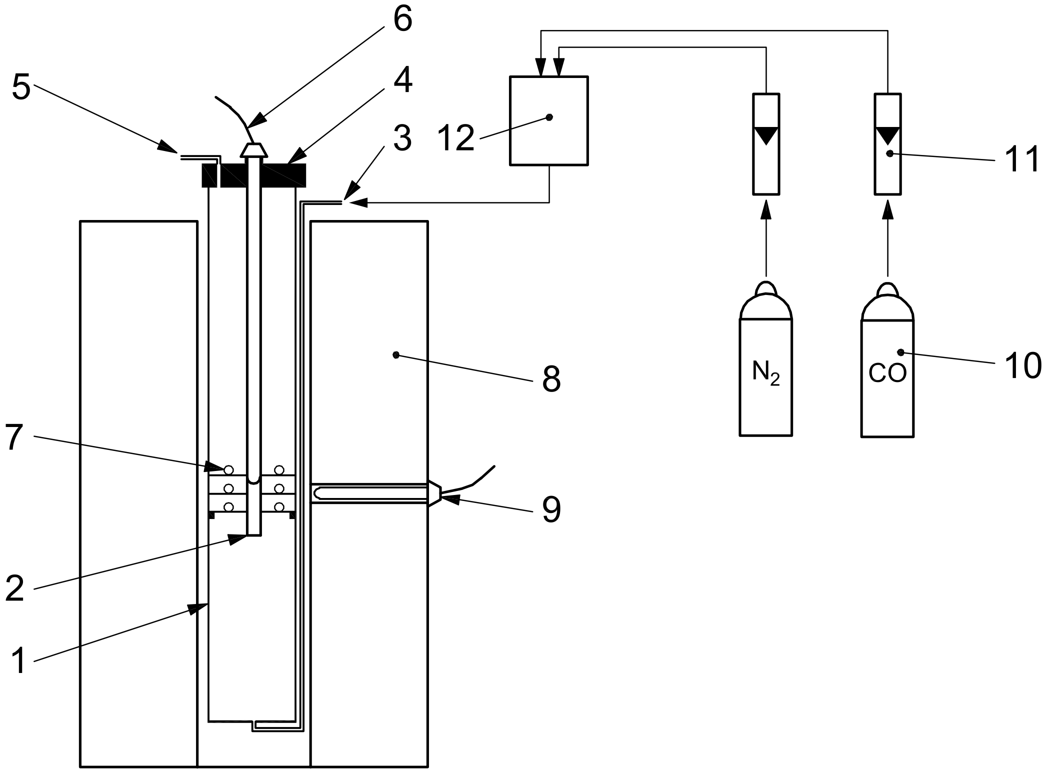

Key

Reduction tube

1 reduction-tube wall
2 test portion holder
3 gas inlet
4 lid
5 gas outlet
6 thermocouple for measuring the reduction temperature
7 test portion

Furnace

8 electrically heated furnace
9 thermocouple for temperature regulation of furnace

Gas-supply system

10 gas cylinder
11 gas flow meter
12 mixing vessel

Figure 1 — Example of test apparatus (schematic diagram)

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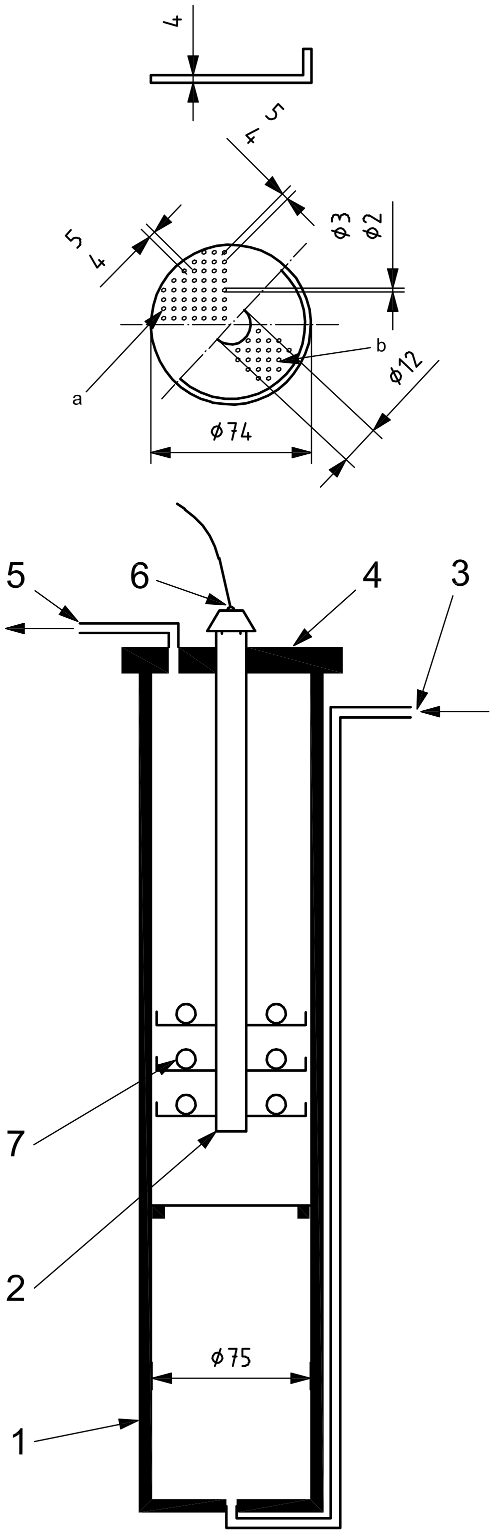
Dimensions in millimetres



Key

1 reduction-tube wall
2 test portion holder
3 opening for gas inlet
4 lid
5 opening for gas outlet
6 opening for thermocouple insertion
7 test portion
[a] Design of perforated plate when a wire basket is used as the test portion holder.
[b] Design of perforated plate when a test portion holder with three levels is used.

NOTE Dimensions not specified in the apparatus Clause are shown for information only.

Figure 2 — Example of a reduction tube with test portion holder (schematic diagram)

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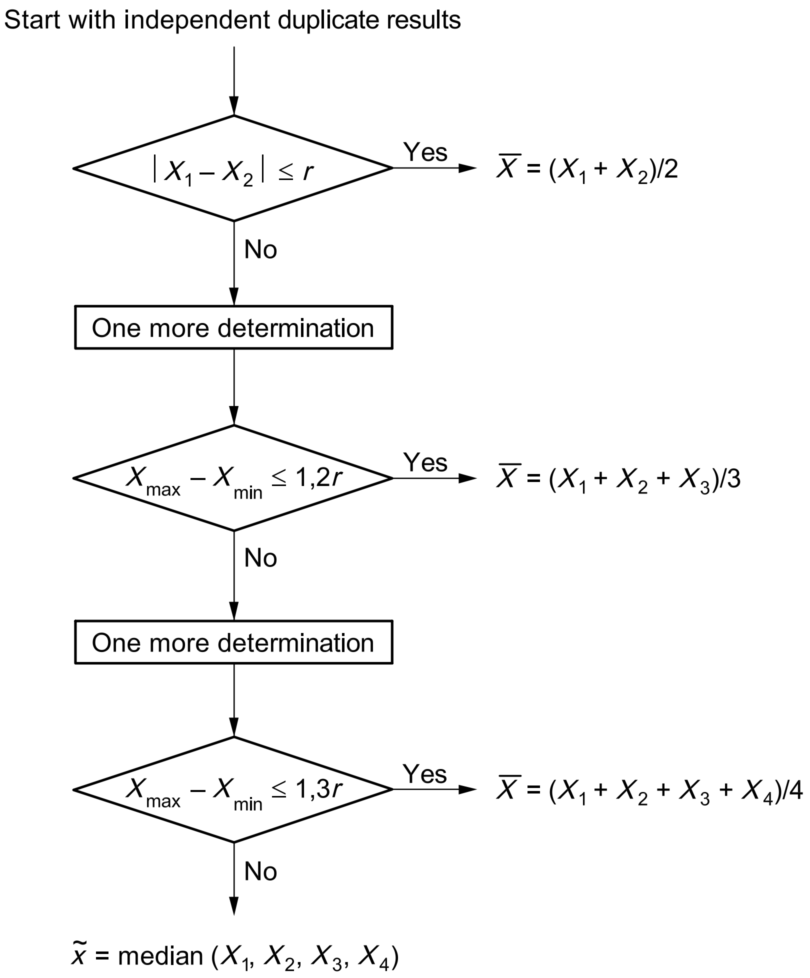
Annex A
(normative)

Flowsheet of the procedure for the acceptance of test results



r: see 9.2

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Annex B
(normative)

Methods for determination of the volume of the test portion

This annex specifies four methods for the determination of the volume of the test portion of hot-bonded pellets.

NOTE As the use of mercury (Hg) should be avoided because of environmental concerns, methods using Hg to determine the volume of the test portion are not recommended. The application of volumetric methods without Hg, such as the ones described in B.2 and B.4 are preferable.

B.1 Mercury volumetric method

B.1.1 Principle

Determination of the volume of the test portion from the volume change of mercury when the test portion is immersed in mercury.

B.1.2 Apparatus

An example of the mercury volumetric apparatus is shown in Figure B.1.

B.1.3 Preparation of the sample

Oven-dry the test portion to constant mass at 105 °C ± 5 °C and cool it to room temperature.

B.1.4 Procedure

B.1.4.1 Set the volumetric apparatus as follows.

Clamp the empty portion holder in the apparatus.

Raise the level of the mercury with the plunger driven by the handle, so that the mercury surface is level with the zero mark of the measuring tube.

Fix the stopper so that the level of the mercury cannot be raised any more with the handle.

Check that the stopper prevents further movement of the plunger when the mercury has reached the zero mark of the tube.

Lower the mercury into the container.

B.1.4.2 After the apparatus has been set, carry out the determination as follows.

Take the test portion consisting of 18 pellets. Remove the test portion holder from the apparatus and place the test portion in the holder. Clamp the test portion holder in the volumetric apparatus and raise the mercury level until the fixed stopper prevents further movement of the plunger.

Read the volume on the measuring tube. Repeat the determination and ensure that the same value is obtained.

Ensure that no mercury remains when the mercury is allowed to flow down through the test portion for the last time.

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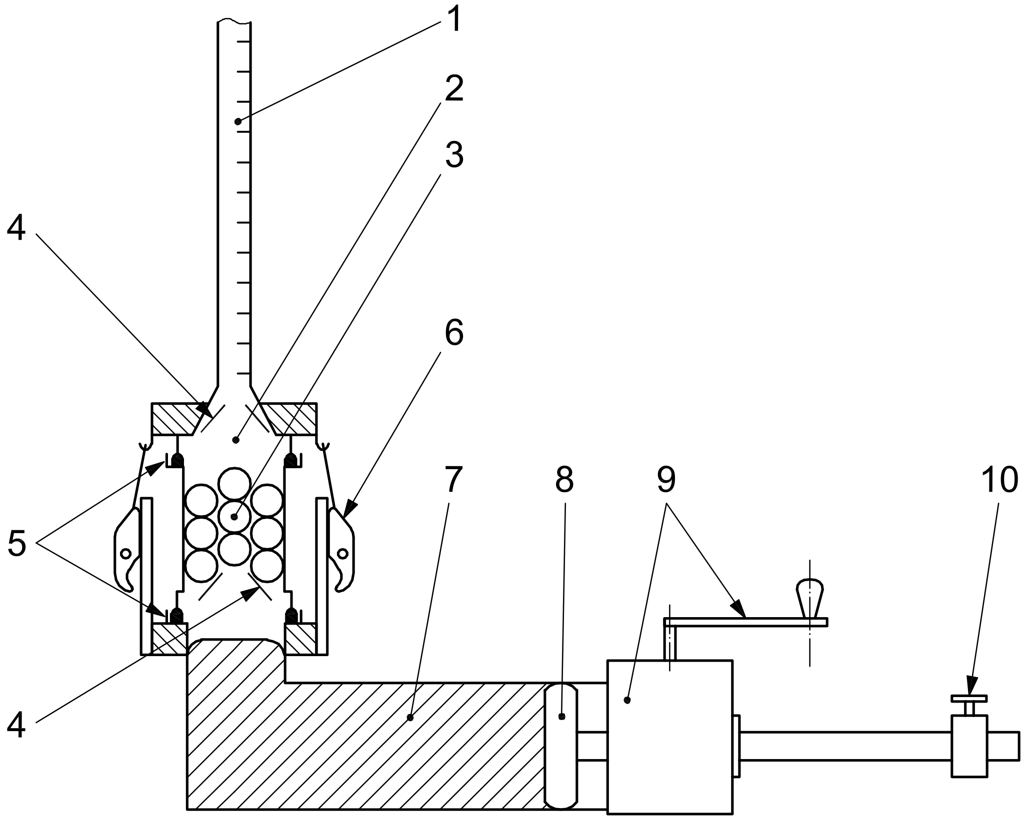
Remove the test portion holder and carefully pour the test portion into a bowl.

Check the pellets one by one, to ensure that there is no mercury on them, and transfer them to another bowl. Then pour any mercury left in the first bowl back into the container.

When the determination has been carried out, carry out a careful check to ensure that there is no free mercury on or near the apparatus. Collect any mercury that has been spilled and pour it into the mercury waste-collection vessel in the laboratory.



Key

1 measuring tube graduated in 1/10 mL
2 test portion holder
3 test portion
4 devices to retain test portion holder within the sample holder
5 o-rings
6 clamp for test portion holder and measuring tube
7 container with mercury
8 movable plunger
9 handle and gear box for moving plunger
10 stopper

Figure B.1 — Example of the mercury volumetric apparatus

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B.2 Oleate-kerosine volumetric method

B.2.1 Principle

Determination of the volume of the test portion from the difference of the masses obtained both in air and in water, after forming a thin hydrophobic film on the surface of the pellets with sodium oleate aqueous solution and stabilizing the film with kerosine. Calculate the volume relative to the density of water.

B.2.2 Test liquids

Prepare all reagents and water freshly as required.

B.2.2.1 **Water**, distilled or ion-exchange water.

B.2.2.2 **Sodium oleate aqueous solution**, $c(C_{17}H_{33}COONa) = 0,1$ mol/L

B.2.2.3 **Kerosine**, of reagent grade.

B.2.3 Apparatus

B.2.3.1 **Container for test liquids** (sodium oleate aqueous solution and kerosine), designed to allow free movement within it of the cage containing the pellets. It shall be made of materials, such as glass, which do not react with sodium oleate aqueous solution or kerosine.

B.2.3.2 **Cage for immersion in test liquids**, to hold the pellets during immersion in the reagents. It shall be made of materials which do not react with sodium oleate aqueous solution or kerosine, and shall be constructed so that the test portion can be stacked in the cage in 2 or 3 layers.

B.2.3.3 **Cage for immersion in water**, to hold the pellets during immersion in water. It shall be made of material to which air bubbles will not adhere.

Figure B.2 shows an example of a cage for immersion in water.

B.2.3.4 **Absorbent sponges**, consisting of 2 pairs of sponges whose surfaces are covered with gauze to absorb any reagent froth on the surface of the pellets.

Figure B.3 shows an example of absorbent sponges.

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Dimensions in millimetres

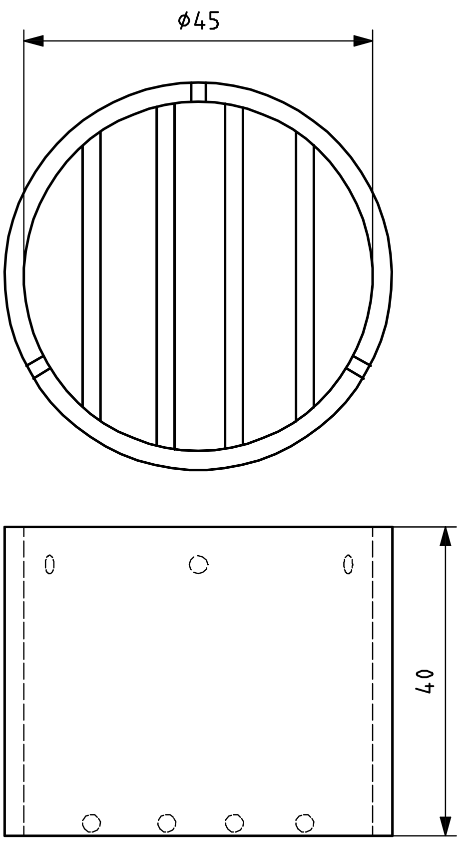
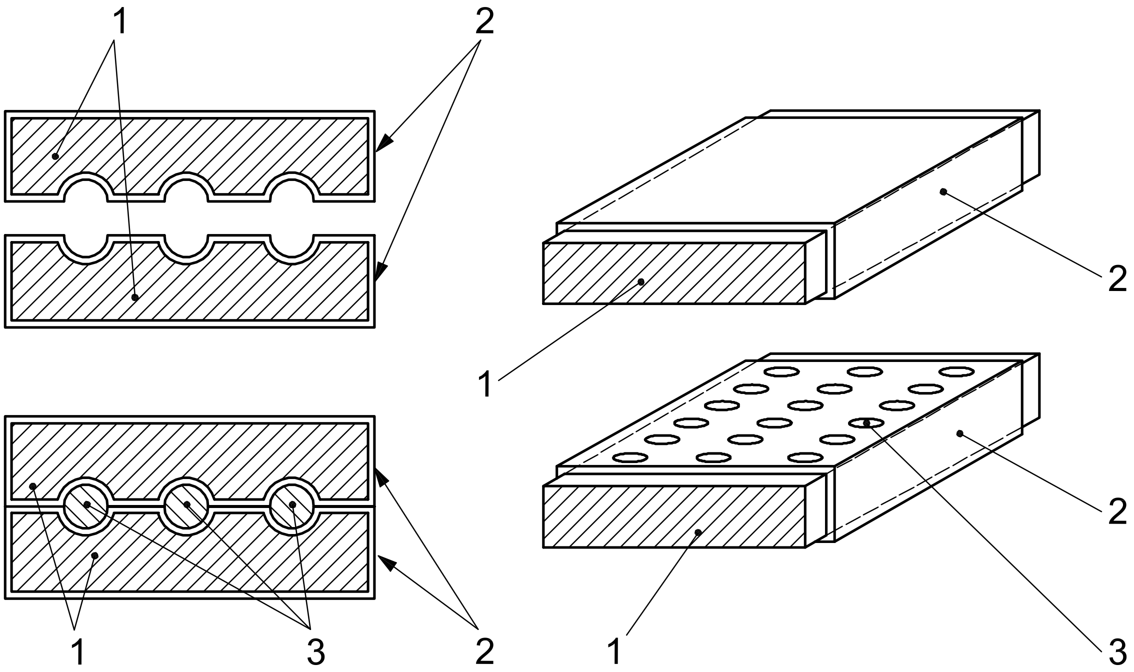


Figure B.2 — Example of a cage for immersion in water



Key

1 sponge
2 gauze (four-fold)
3 pellets

Figure B.3 — Absorbent sponges

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B.2.3.5 **Weighing device**, capable of weighing the test portion to an accuracy of 0,001 g.

B.2.3.6 **Suspensory line**, a suitable line, approximately 0,3 mm in diameter, for suspending the cage in water.

B.2.3.7 **Beaker**, for containing the pellets to be weighed in water, with a suitable capacity which does not contact the cage. The base-plane area shall be large enough to not significantly change the water level when weighing the test portion.

B.2.3.8 **Stand for the beaker**, either wooden or metal.

Materials that generate static electricity, such as acrylic resin, should not be used.

B.2.3.9 **Thermometer**, graduated in units of 0,5 °C or less.

B.2.4 Procedure

B.2.4.1 Place the test portion in the cage (B.2.3.2) and immerse the cage with the test portion in the sodium oleate solution (B.2.2.2) and leave for 30 min. Immediately before raising the cage, repeatedly shake it to remove air bubbles adhering to the pellets.

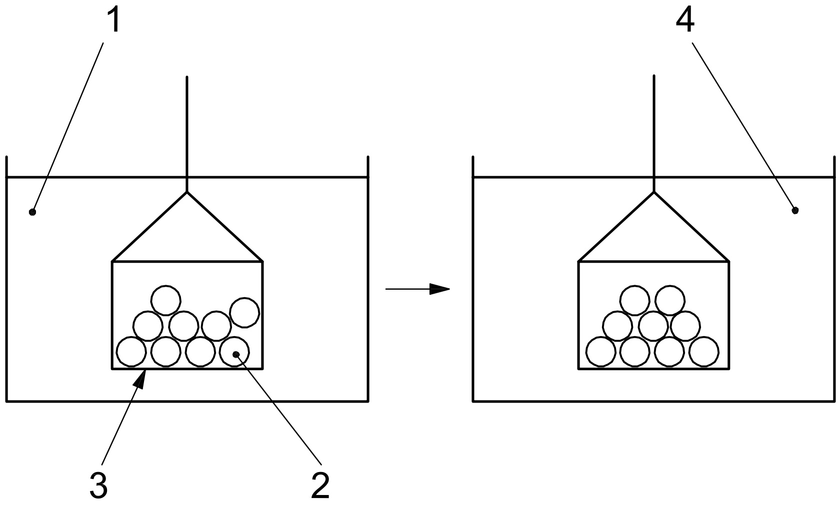
Figure B.4 shows a general view of immersion in reagents.

B.2.4.2 Remove the test portion from the solution and place the pellets, separated from each other, in one pair of sponges (see B.2.3.4). Then press the sponges slightly by hand for 10 s, to absorb the froth of sodium oleate adhering to the test portion.

Next, rotate the test portion vertically to about 90°, line up another pair of sponges and press the containers down again for 10 s to ensure that air bubbles are removed from the whole surface of the test portion.

Use the fresh surface of the gauze.

B.2.4.3 Place the test portion, from which froth has been removed, in the cage (B.2.3.2). Then immerse this cage containing the pellets in kerosine for about 10 s (see Figure B.4).



Key

1 sodium oleate solution

2 pellets

3 cage

4 kerosine

Figure B.4 — General view of immersion in reagents

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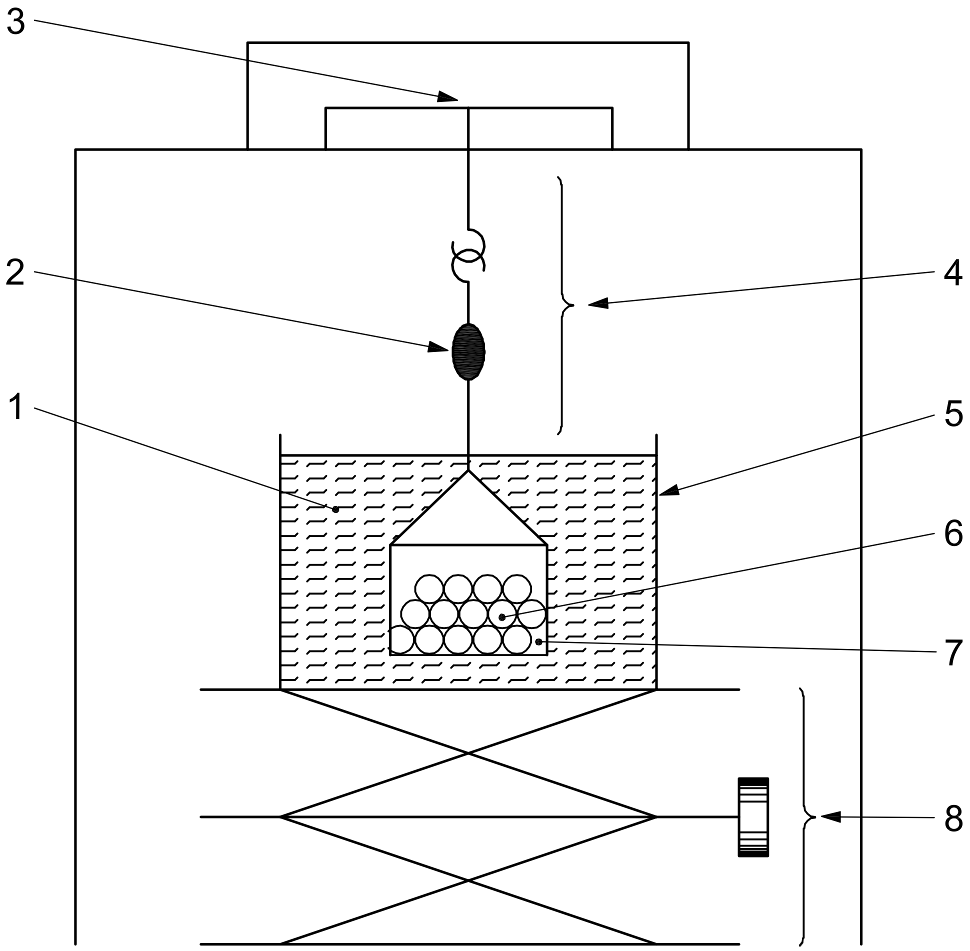
B.2.4.4 Remove the test portion from the kerosine and put it in a pair of sponges to absorb the kerosine froth as described in B.2.4.2.

B.2.4.5 Place the test portion, from which froth has been removed, in the cage for immersion in water (B.2.3.3). Then suspend the cage containing the test portion from the balance, and shake the cage in the water to remove air bubbles adhering to the surface of the test portion (see Figure B.5).

Record the mass of the test portion and the cage (m_1) to 3 decimal places after immersing them in water for 1 min.

NOTE Preliminary immersion of the cage in water will help prevent air bubbles from adhering to it.



Key

1 water
2 sinker
3 balance
4 fishing line
5 beaker
6 pellets
7 cage
8 stool

Figure B.5 — General view of mass determination in water

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B.2.4.6 Remove the test portion from the cage, and put it in the pair of sponges to absorb the water adhering to the surface.

B.2.4.7 Record the mass of the test portion (m_2) in air to 3 decimal places.

B.2.4.8 Suspend the cage from which the test portion was removed and immerse it in water. Obtain the mass of the cage immersed in water (m_3) to 3 decimal places.

B.2.4.9 Measure the temperature of water in the container, to obtain the density (ρ_1) of water at that temperature using relevant tables.

B.2.5 Calculation of volume

Calculate the volume V of the test portion, in millilitres, to 3 decimal places using the following equation:

$$V = \frac{m_2 - (m_1 - m_3)}{\rho_1}$$

where

m_1 is the mass, in grams, of the test portion plus the cage in water in B.2.4.5;

m_2 is the mass, in grams, of the test portion in air in B.2.4.7;

m_3 is the mass, in grams, of the cage in water in B.2.4.8;

ρ_1 is the density, in grams per millilitre, of water expressed to 4 decimal places.

B.3 Mercury immersion method

B.3.1 Principle

The volume is determined by immersing the test portion in mercury. The mass of mercury displaced is measured by a weighing device, and equals the mass of the test portion plus the force required to press the test portion below the surface of the mercury.

B.3.2 Apparatus

Figure B.6 shows an example of the mercury immersion apparatus.

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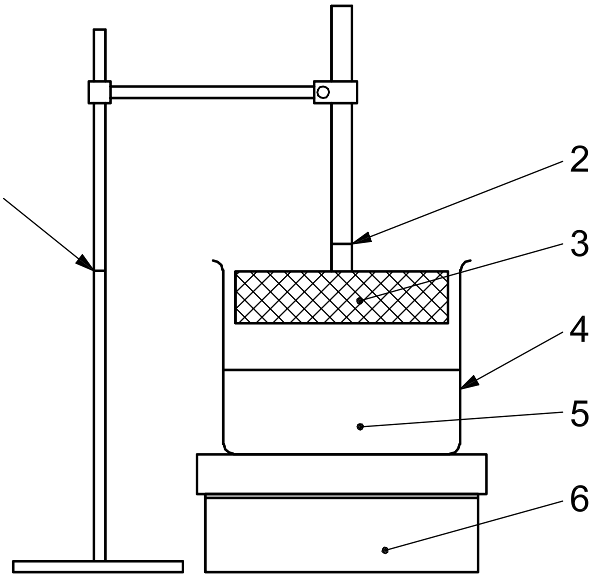

Key

1 reference mark
2 immersion mark
3 basket (0,6 mm sieve)
4 beaker (500 mL)
5 mercury (200 mL)
6 balance

Figure B.6 — Example of the mercury immersion apparatus

B.3.3 Preparation of the test portion

Dry the test portion at 110 °C for 8 h.

B.3.4 Procedure

Add 200 mL of mercury to a 500 mL beaker.

Weigh and record the masses of the test portions to be tested, each consisting of 18 pellets.

Place the beaker on a balance and position the immersion screen in the mercury, adjusting it by loosening the ring-stand clamp, while moving the screen up or down until it is just covered by mercury. Mark this position on the ring-stand; the mark serves as the reference setting used in the following steps.

a) Weigh and record the mass of the mercury and beaker with the screen immersed to the reference mark, and then tare the balance by calibrating the balance reading to zero. Now remove the screen from the mercury, being careful to remove as little mercury as possible on the screen. (Do not tare the balance again.)

b) Place the test portion on top of the mercury and lower the screen to the reference setting, making sure to immerse the entire test portion. Weigh and record the mass (m_1).

c) Determine the test portion volume by dividing the total mass found in step b) by the density of mercury.

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B.3.5 Calculation

Calculate the volume V of the test portion, in millilitres, using the following equation:

$$V = \frac{m_1}{\rho_2}$$

where

m_1 is the mass, in grams, of the test portion in mercury;

ρ_2 is the density of mercury, in grams per millilitre, at the temperature at which the determination is carried out.

B.4 Water immersion method

B.4.1 Principle

Determination of the volume of the test portion from the mass differences obtained both in air and water, after pouring water into the open pores of pellets. Calculation of the volume relative to the density of water.

B.4.2 Water

Prepare distilled or ion-exchange water freshly as needed.

B.4.3 Apparatus

B.4.3.1 **Cage**, to hold the test portion during immersion in water. It shall be made of materials to which air bubbles do not adhere.

Figure B.2 shows an example of a cage for immersion in water.

B.4.3.2 **Suspensory line**, suitable line, approximately 0,3 mm in diameter, for suspending the cage in water.

B.4.3.3 **Container for the water**, designed so that the water surface shall not change significantly when the test portion is immersed in water.

B.4.3.4 **Stand for a beaker**, either wooden or metal.

Materials that generate static electricity, such as acrylic resin, should not be used.

B.4.3.5 **Absorbent sponges**, consisting of a pair of sponges whose surfaces are covered with gauze.

Figure B.3 shows an example of absorbent sponges.

B.4.3.6 **Weighing device**, capable of weighing the test portion to an accuracy of 0,001 g.

B.4.3.7 **Thermometer**, graduated in units of 0,5 °C or less.

B.4.4 Procedure

Immerse the cage (B.4.3.1) containing the test portion in water and shake it to remove air bubbles. Leave it in water for 20 min and record the combined mass of the test portion and the cage to 3 decimal places (m_1).

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Remove the test portion from the cage.

Carefully pack each pellet from the test portion in the absorbent sponges (B.4.3.5). Then repeat the procedure after changing the gauze. (Use the fresh surface of the gauze when packing.)

Record the mass of the test portion (m_2) immediately after removal from the absorbent sponges.

Re-immerse the cage from which the test portion was removed and determine its mass in water (m_3) to three decimal places.

Measure the temperature of water in the beaker, to obtain the density (ρ_1) of water at the test temperature using relevant tables. Record the density to four decimal places.

B.4.5 Calculation of results

Calculate the volume V of the test portion, in millilitres, using the following equation:

$$V = \frac{m_2 - \left(m_1 - m_3\right)}{\rho_1}$$

where

m_1 is the mass, in grams, of the test portion plus the cage in water;

m_2 is the mass, in grams, of the test portion in air after immersion in water;

m_3 is the mass, in grams, of the cage in water;

ρ_1 is the density, in grams per millilitre, of water at the test temperature.

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