INTERNATIONAL STANDARD

ISO 5072

First edition
1997-07-01

Brown coals and lignites — Determination of true relative density and apparent relative density

Charbons bruns et lignites — Détermination de la densité relative vraie et de la densité relative apparente

Reference number
ISO 5072:1997(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 5072 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 5, *Methods of analysis*.

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Printed in Switzerland

Brown coals and lignites — Determination of true relative density and apparent relative density

1 Scope

This International Standard describes methods for the determination of true relative density and the apparent relative density of brown coals and lignites.

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 1015:1992, *Brown coals and lignites — Determination of moisture content — Direct volumetric method.*

ISO 5068:1983, *Brown coals and lignites — Determination of moisture — Indirect gravimetric method.*

ISO 5069-2:1983, *Brown coals and lignites — Principles of sampling — Part 2: Sample preparation for determination of moisture content and for general analysis.*

3 Definitions

3.1 True relative density: Ratio of the mass of a sample of dry coal ground to pass through a 212 µm sieve to the mass of an equal volume of water at a specified temperature.

3.2 Apparent relative density: Ratio of the mass of a dry coal to the mass of a volume of water equal to the apparent volume of the coal at a specified temperature.

4 Determination of the true relative density by the water method

4.1 Principle

The true relative density is determined pycnometrically by water displacement, with the inclusion of a wetting agent to ensure dispersion of the solid in the displacement medium.

NOTE — The use of other displacement media such as methanol is not recommended because of possible swelling of some brown coals and lignites in such liquids.

4.2 Reagents

4.2.1 Distilled or deionized water, freshly boiled.

4.2.2 Wetting agent, 5 % (*V*/*V*) solution of detergent (such as sodium dodecyl sulfate) is suitable.

4.2.3 Potassium dichromate-sulfuric acid mixture, for cleaning pycnometers.

4.3 Apparatus

4.3.1 Analytical balance, sensitive to 0,1 mg.

4.3.2 Camel hair brush, of such a diameter that bristles can pass completely through the stem of the funnel (4.3.4).

4.3.3 Filter paper, for drying the necks of the pycnometers.

4.3.4 Funnel, with a stem of sufficient length to reach the middle of the pycnometer flask.

4.3.5 Glass cloth, fibre free for polishing the pycnometers prior to weighing.

4.3.6 Glass syringe, with a needle to bring the pycnometer liquid to the mark of the pycnometer.

4.3.7 Earthing point, to remove static charge from pycnometers.

4.3.8 Mercury thermometer, capable of measuring the temperature in the range of 20 °C to 30 °C with a minimum scale spacing of 0,1 °C.

4.3.9 Pycnometers, of capacity 50 ml with capillary-bored ground stoppers, internal diameter of the neck no greater than 5 mm.

4.3.10 Vacuum desiccator, with protective cage.

NOTE — The use of vacuum desiccator is preferable for the purpose of degassing samples.

4.3.11 Vacuum pump, capable of attaining a vacuum of residual pressure from 0 kPa to 5 kPa.

4.3.12 Water bath, thermostatically controlled, the temperature of which shall be maintained at 25 °C ± 0,1 °C as measured by a thermometer (4.3.8) permanently placed in the water bath.

4.3.13 Weighing boat, with a capacity of not less than 2 g.

4.4 Sample

The sample for the determination of true relative density shall be the general analysis sample (ground to pass through a 212 µm sieve) prepared in accordance with ISO 5068-2.

4.5 Procedure

4.5.1 Calibration of the mass of the pycnometer(s)

Pipette 10 ml of wetting agent into the pycnometer(s) (4.3.9). Pipette 10 ml of water (4.2.1) into the pycnometer(s). Swirl to mix the solution. Place the pycnometer(s) in the vacuum desiccator (4.3.10) and evacuate to 0 Pa to 500 Pa for 15 min. Release the vacuum and fill the pycnometer(s) with water (4.2.1) using a glass syringe (4.3.6) to 3 mm from the top of the neck.

Place the pycnometer(s) in a water bath (4.3.12) thermostatically controlled at 25 °C ± 0,1 °C. Allow to equilibrate for at least 1 h 45 min. Top up the pycnometer(s) to the meniscus using a glass syringe without removing them from the water bath. Leave the pycnometer(s) in the water bath for a further 5 min.

Remove the pycnometer(s) from the water bath and insert the capillary-bored stopper(s) such that no air is trapped in the pycnometer(s). Wipe the outside of the pycnometer(s) gently using filter paper (4.3.3) to remove excess water. Polish the pycnometer(s) using fibre-free glass cloth (4.3.5) to remove any visible film. Touch the pycnometer(s) to the earthing point (4.3.7). Weigh the pycnometer(s) and record the mass(es) to the nearest 0,1 mg.

The mean of three replicate determinations of the mass of each pycnometer which fall within the range of 1 mg is taken as the calibration mass of the pycnometer.

4.5.2 Determination of true relative density

Weigh 2 g ± 0,1 g of sample, to the nearest 0,1 mg, into a wighing boat (4.3.13). Transfer the sample to a pycnometer using a funnel (4.3.4) and a camel hair brush (4.3.2). Ensure that no material is lost during the transfer.

Pipette 10 ml of wetting agent (4.2.2) into the pycnometer allowing the wetting agent to run down the inner surface of the pycnometer. Pipette 10 ml of water (4.2.1) into the pycnometer allowing the water to run down the inner surface of the pycnometer. Swirl gently to wet the sample with the minimum production of bubbles. Place the pycnometer in the vacuum desiccator and evacuate to 0 Pa to 500 Pa for 15 min. To ensure that no sample containing froth is expelled from the pycnometer, regulate the vacuum supply to remove any froth produced below the neck of the pycnometer. Release the vacuum and fill the pycnometer with water (4.2.1) to 3 mm from the top of the neck using a glass syringe.

Place the pycnometer in a water bath thermostatically controlled at 25 °C ± 0,1 °C. Allow to equilibrate for at least 1 h 45 min. Top up the pycnometer to the meniscus using a glass syringe without removing from the water bath. Leave the pycnometer in the water bath for a further 5 min.

Remove the pycnometer from the water bath and insert the capillary bored stopper such that no air is trapped in the pycnometer. Wipe the outside of the pycnometer gently using filter paper to remove excess water. Polish the pycnometer using fibre-free glass cloth to remove any visible film. Touch the pycnometer to the earthing point. Weigh the pycnometer and record the mass to the nearest 0,1 mg.

Determine the moisture content M on a separate test portion of the sample (4.4) in accordance with ISO 1015 or ISO 5068:1983, subclause 6.3.

4.6 Calculation of results

Calculate the true relative density of the dry coal TRD_d according to the formula:

$$TRD_d = \frac{m \times (100 - M_{ad})}{m \times (100 - M_{ad}) + 100 \times (m_1 - m_2)}$$

where

m is the mass, in grams, of the analysis sample;

m_1 is the mass, in grams, of the pycnometer and water;

m_2 is the mass, in grams, of the pycnometer, sample and water;

M_{ad} is the moisture, as a percentage by mass, of the analysis sample.

4.7 Precision of the method

4.7.1 Repeatability limit

The results of duplicate determinations, carried out in the same laboratory by the same operator using the same apparatus on the same sample within short intervals of time, shall not differ by more than the value given in table 1.

4.7.2 Reproducibility limit

The means of the results of duplicate determinations, carried out in each of two laboratories on representative test portions taken from the same sample, shall not differ by more than the value given in table 1.

Table 1 — Precision of the method

Repeatability	Reproducibility
% of result	% of result

4.8 Test report

The test report shall contain the following information:

a) an identification of the sample;

b) reference to this International Standard;

c) the result and the method of expression used;

d) any unusual features noted during the determination.

5 Determination of the apparent relative density

5.1 Principle

The apparent relative density is determined by weighing a sample suspended in water, allowing the sample to drain to remove surface liquid and then reweighing the sample in air.

5.2 Reagents

5.2.1 Water, distilled or deionized.

5.3 Apparatus

5.3.1 Beam balance, sensitive to 10 mg, modified as in figure 1.

5.3.2 Evaporating dishes, 160 mm diameter, 60 mm deep.

5.3.3 Paper towels or **filter papers**

5.3.4 Top pan balance, capacity of 1 kg, sensitive to 10 mg.

5.3.5 Vacuum pump, capable of attaining 250 Pa pressure.

5.3.6 Vacuum desiccator, 200 mm diameter.

5.4 Sample

The determination of apparent relative density shall be carried out on air-dried sample of particle size 10 mm to 30 mm.

5.5 Procedure

Set up the beam balance (5.3.1) as shown in figure 1, with a minimum of 50 mm of water (5.2.1) covering the gauge basket. Tare the beam balance so that at the balance point approximately 5 g is added to the weight pan. Record the tare mass.

NOTE — Check the beam balance tare at least once every 5 to 10 determinations.

Dimensions in millimetres

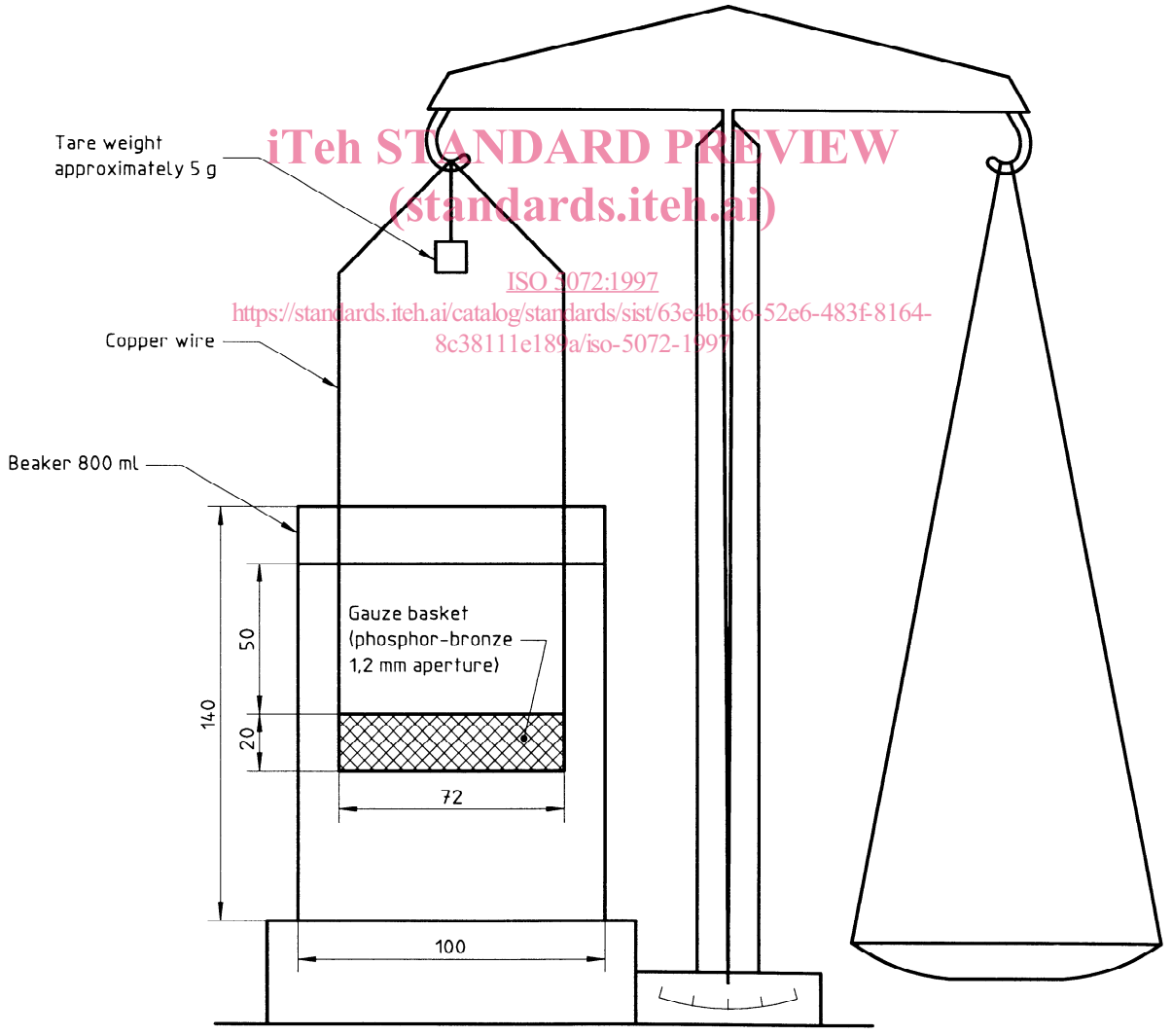

Figure 1 — Modified beam balance

Weigh from 30 g to 35 g of sample (5.4) into an evaporating dish (5.3.2). Add water (5.2.1) to the dish to completely immerse the sample. Place the evaporating dish plus sample in a vacuum desiccator (5.3.6). Evacuate the desiccator to 0 Pa to 250 Pa for 5 min. Release the vacuum and allow to stand for a further 5 min.

Transfer the sample to a pad of paper towels (5.3.3), using the paper towels to remove excess water. Immediately place the sample in the gauze basket of the beam balance ensuring that the sample is totally covered with water. Allow the balance to attain equilibrium and record the mass of the sample in water to the nearest 10 mg.

Transfer the sample from the gauze basket to a pad of paper towels, using the paper towels to remove any visible surface moisture. Immediately transfer the sample to a top pan balance (5.3.4) and record its mass in air to the nearest 10 mg.

NOTE — Do not allow excessive drying when transferring the sample.

5.6 Calculation of results

Calculate the apparent relative density of the dry coal ARD_d according to the formula:

$$ARD_d = \frac{m \times (100 - M_t)}{100 \times (m - m_1 + m_2)}$$

where

m is the mass, in grams, of the sample in air;

m_1 is the mass, in grams, of the sample suspended in water;

m_2 is the mass, in grams, of the tare weight;

M_t is the total moisture, as a percentage by mass, determined on the sample after density determination.

5.7 Precision of the method

5.7.1 Repeatability limit

The results of duplicate determinations, carried out in the same laboratory by the same operator using the same apparatus on the same sample within short intervals of time, shall not differ by more than the value given in table 2.

5.7.2 Reproducibility limit

The means of the results of duplicate determinations, carried out in each of two laboratories on representative test portions taken from the same sample, shall not differ by more than the value given in table 2.

Table 2 — Precision of the method

Repeatability	Reproducibility
2 % of the mean result	5 % of the mean result

5.8 Test report

The test report shall contain the following information:

a) an identification of the sample;

b) reference to this International Standard;

c) the result and method of expression used;

d) any unusual features noted during the determination.

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