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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

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 27, *Coal and Coke*, Subcommittee SC 5, *Methods of analysis*.

This third edition cancels and replaces the second edition (ISO 5072:2013), of which it constitutes a minor revision. The changes compared to the previous edition are as follows:

— referenced documents have been updated;

— terms and definitions have been added;

— sample has been added;

— calculation and expression of results have been amended;

— precision has been amended;

— test report has been amended.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html

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

1 Scope

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

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 5068-1, *Brown coals and lignites — Determination of moisture content — Part 1: Indirect gravimetric method for total moisture*

ISO 5068-2, *Brown coals and lignites — Determination of moisture content — Part 2: Indirect gravimetric method for moisture in the analysis sample*

ISO 13909-4, *Hard coal and coke — Mechanical sampling — Part 4: Coal — Preparation of test samples*

ISO 18283, *Coal and coke — Manual sampling*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

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 % (volume fraction) 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, with a resolution of 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 cleaning cloth, fibre free for polishing the pycnometers prior to mass determination.

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

4.4 Sample

The sample shall be the general analysis test sample, prepared to a nominal top size of 212 µm by the preparation procedures specified in ISO 13909-4 or ISO 18283.

The sample should be brought in moisture equilibrium with the laboratory atmosphere by exposure in a thin layer on a tray. Exposure time shall be kept to a minimum.

The sample shall be thoroughly mixed immediately before analysis, preferably by mechanical means.

Duplicate determinations of moisture from the same test sample shall be conducted concurrently with the determination of the true relative density by the method specified in ISO 5068-2.

4.5 Procedure

4.5.1 Calibration of the mass of the pycnometer(s)

Pipette 10 ml of wetting agent (4.2.2) 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 cleaning cloth (4.3.5) to remove any visible film. Touch the pycnometer(s) to the earthing point (4.3.7). Determine and record the pycnometer(s) mass(es) to the nearest 0,1 mg.

The difference between the greatest and least value of three replicate determinations of the mass of each pycnometer must be no more than 1 mg. The mean of these three replicates is taken as the calibration mass of the pycnometer

4.5.2 Determination of true relative density

Transfer a mass of 2 g ± 0,1 g of the general analysis test sample, determined to the nearest 0,1 mg, into a sample 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 cleaning cloth to remove any visible film. Touch the pycnometer to the earthing point. Determine and record the pycnometer mass to the nearest 0,1 mg.

4.6 Calculation of results

Calculate the true relative density of the dry coal, TRD_d, according to Formula (1):

$$TRD_d = \frac{m \times (100 - M_{ad})}{m \times (100 - M_{ad}) + 100 \times (m_1 - m_2)} \quad (1)$$

where

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

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

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

M_{ad} is the moisture mass fraction, as a percent, of the general analysis test sample (determined in accordance with ISO 5068-2);

100 is conversion factor from dimensionless mass fraction to percent, in %.

4.7 Precision

4.7.1 Repeatability limit

The results of duplicate determinations, carried out in the same laboratory by the same operator with the same apparatus within a short interval of time on representative portions taken from the same analysis sample, shall not differ by more than the values of the repeatability limit, *r*, shown in Table 1.

4.7.2 Reproducibility limit

The mean of the results of duplicate determinations, carried out in each of the two laboratories, on the representative portions taken from the same sample at the last stage of sample preparation, shall not differ by more than the values of the reproducibility limit, *R*, shown in Table 1.

Table 1 — Repeatability limit and reproducibility limit

Test parameter	Maximum acceptable differences between results absolute, %	
	Repeatability limit r	Reproducibility limit R
TRD_d	0,02	0,04

4.8 Test report

The test report shall include the following information:

a) identification of the sample tested;

b) the method used by reference to this document, i.e. ISO 5072:2021;

c) the date of the determination;

d) the results and the method of expression used.

5 Determination of the apparent relative density

5.1 Principle

The apparent relative density is determined by determining the mass of a sample suspended in water, allowing the sample to drain to remove surface liquid and then redetermining the mass of 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 a pressure of 250 Pa.

5.3.6 Vacuum desiccator, 200 mm diameter.

5.4 Sample

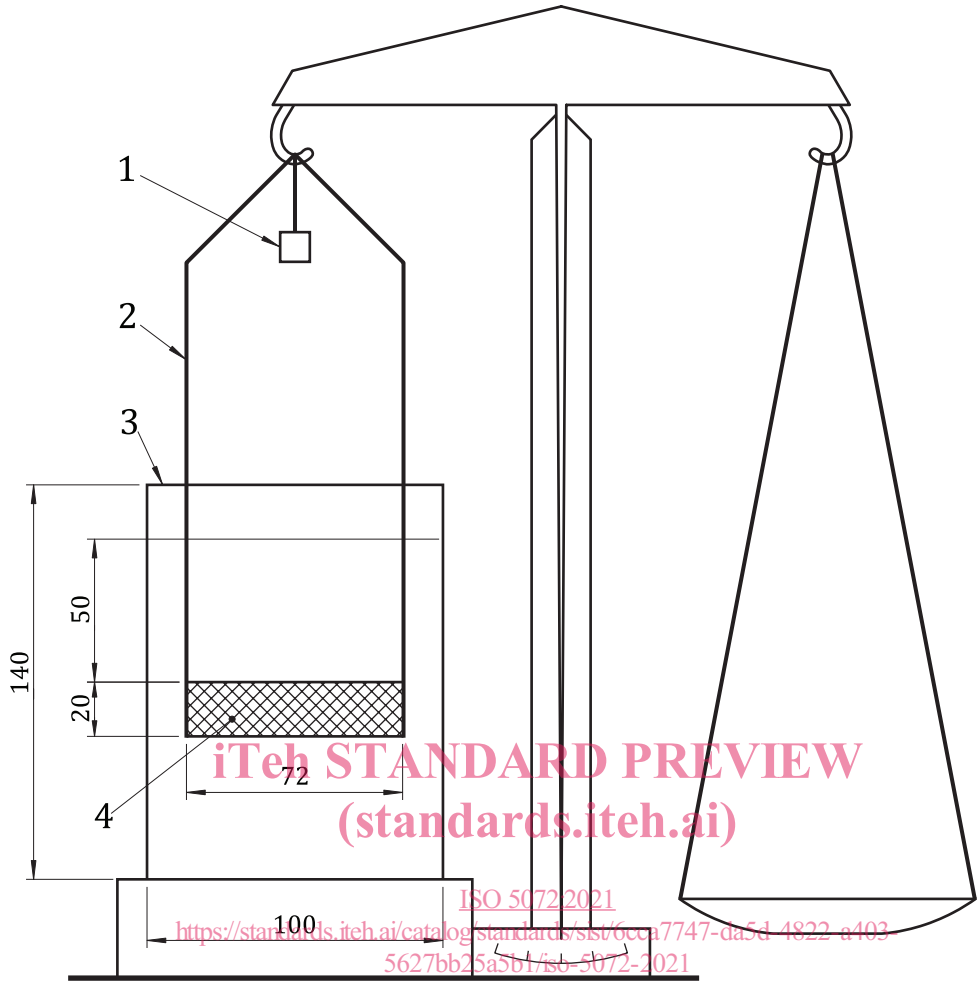
The determination of apparent relative density shall be carried out on sample air-dried in accordance with the preparation procedures specified in ISO 13909-4 or ISO 18283, of particle size 10 mm to 30 mm.

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 mass measurement pan. Record the tare mass.

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

Dimensions in millimetres



Key

1 tare mass, approximately 5 g
2 copper wire
3 beaker, 800 ml
4 gauze basket (phosphor-bronze 12 mm aperture)

Figure 1 — Modified beam balance

Transfer a mass 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.