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Brown coals and lignites — Determination of moisture content —

Part 1: Indirect gravimetric method for total moisture

Charbons bruns et lignites — Détermination de l'humidité —

Partie 1: Méthode gravimétrique indirecte pour l'humidité totale

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 5068-1 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 5, *Methods of analysis*.

This first edition of ISO 5068-1, together with ISO 5068-2, cancels and replaces ISO 5068:1983, which has been technically revised.

ISO 5068 consists of the following parts, under the general title *Brown coals and lignites — Determination of moisture content*:

— *Part 1: Indirect gravimetric method for total moisture*

— *Part 2: Indirect gravimetric method for moisture in the analysis sample*

Brown coals and lignites — Determination of moisture content —

Part 1: Indirect gravimetric method for total moisture

1 Scope

This International Standard specifies two methods for determination of the total moisture content of brown coals and lignites using an indirect gravimetric single-stage method and a two-stage method.

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 1213-2, *Solid mineral fuels — Vocabulary — Part 2: Terms relating to sampling, testing and analysis*

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

3 Definitions

For the purposes of this document, the definitions given in ISO 1213-2 apply.

4 Principle

4.1 Single-stage method

A sample, prepared using a closed mill, is dried to constant mass at a temperature of 105 °C to 110 °C in an atmosphere of nitrogen, and the total moisture content is calculated from the loss in mass of the sample.

4.2 Two-stage method

A sample is coarsely ground and is then allowed to dry, either at ambient temperature or at a higher temperature not exceeding 40 °C, to reach equilibrium with the atmosphere. The sample is further crushed and then dried to a constant mass at a temperature of 105 °C to 110 °C in an atmosphere of nitrogen. The total moisture content is calculated from the losses in mass during the two drying stages.

5 Reagent

5.1 Desiccant, fresh or freshly regenerated and preferably self-indicating.

Suitable desiccants are magnesium perchlorate and silica gel.

WARNING — Magnesium perchlorate is a strong oxidizing agent. Do no attempt to regenerate the absorbent. Do not permit contact with organic materials or reducing agent.

5.2 Nitrogen, high-purity, dry with a maximum oxygen content of 30 µl/l.

NOTE Commercial nitrogen of this purity is available. See Annex A for details.

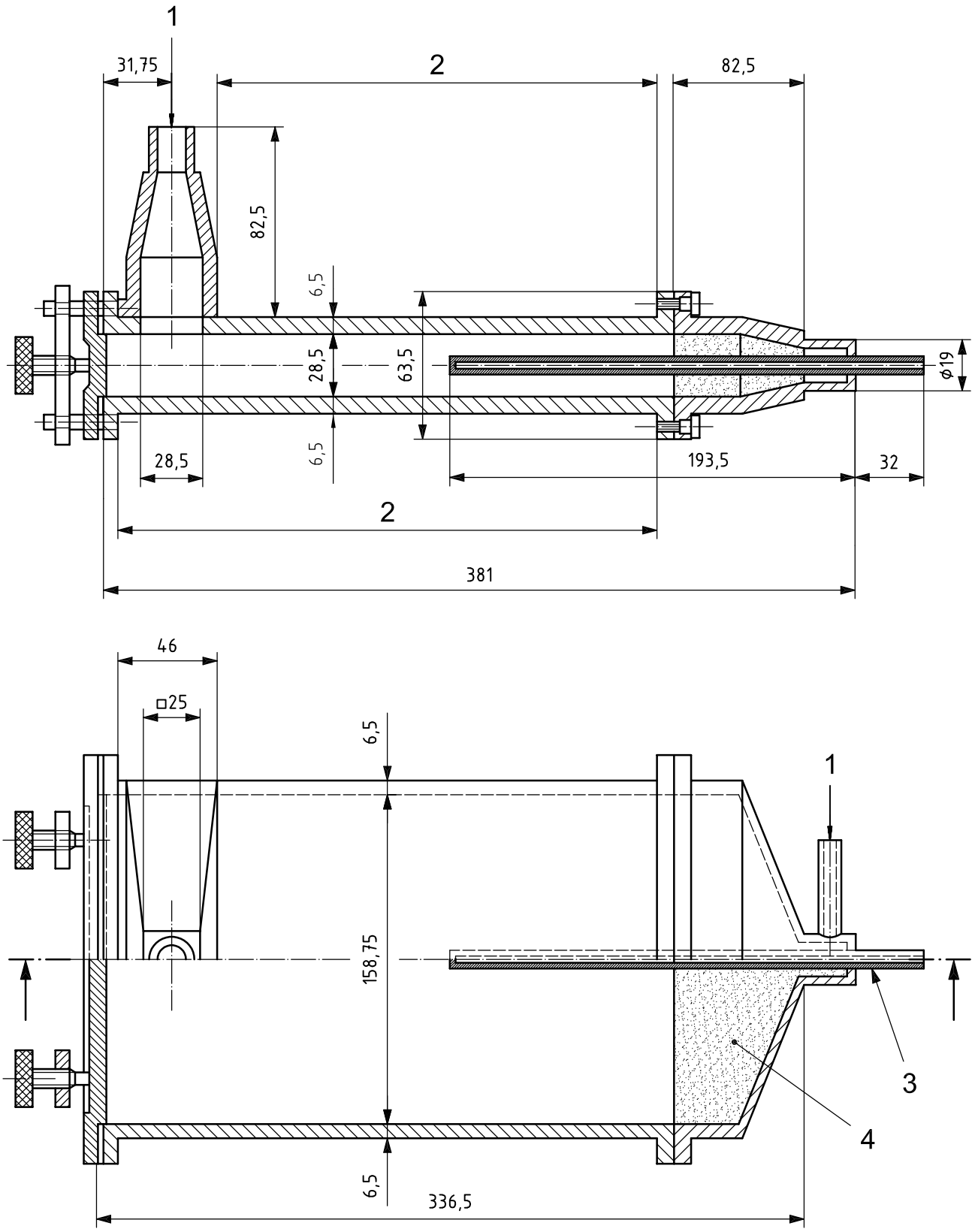
6 Apparatus

6.1 Oven, nitrogen-flushed, capable of being controlled by a temperature of 105 °C to 110 °C and with the additional provision for passing a current of dry nitrogen through it at a flow rate about 15 times the oven volume per hour.

Before using a new oven, carry out a temperature profile through the interior and take care to insert samples only where the temperature is known to be 105 °C to 110 °C.

A suitable oven is illustrated in Figure 1.

Dimensions in millimetres



Key

1 nitrogen outlet
2 space for heating element
3 thermometer tube
4 copper gauze, having apertures of nominal size 0,25

Figure 1 — Typical nitrogen oven

6.2 Drying cabinet, capable of being controlled at a temperature of 30 °C to 40 °C and with provision for venting.

6.3 Tray, non-corrodible, of such dimensions that the loading of the sample layer does not exceed 1 g/cm^2.

6.4 Weighing dish, a shallow dish of silica or glass, with ground edges and fitted with a ground-glass cover, or of a non-corrodible, heat-resistant material with well-fitting lid.

The diameter of the dish shall be such that the mass of coal layer does not exceed 0,25 g/cm^2 for 10 g of the sample.

6.5 Balances, sensitive to 0,01 g and to 0,001 g.

6.6 Flow meter, capable of measuring the rate of flow nitrogen through the oven.

6.7 Drying tower, 500 ml capacity, packed with magnesium perchlorate or silica gel for drying the nitrogen.

6.8 Cooling vessel, for example a desiccator without desiccant, containing a metal plate, preferably of aluminium or copper.

The vessel may be provided with a means to pass nitrogen through it during the cooling period.

7 Sample

7.1 Single-stage method

Using closed crusher, crush the special moisture sample or the moisture sample taken from the common sample in accordance with ISO 5069-2 to pass a 3,15 mm sieve and divide to 500 g.

7.2 Two-stage method

Crush the special moisture sample or the moisture sample from the common sample in accordance with ISO 5069-2 to pass a 20 mm sieve and divide to 2 kg.

The sample mass, in kilograms, should not be less than 0,1 of the maximum grain size, in millimetres, for grain sizes between 3 mm and 20 mm and, in any case, not less than 500 g.

8 Procedure

8.1 Single-stage method

Raise the temperature of the oven to between 105 °C and 110 °C while passing nitrogen at a rate to provide 15 oven-volume changes per hour.

Weigh a clean, dry, empty weighing dish with its lid to the nearest 0,001 g. Spread into it an even layer of approximately 10 g of the sample (7.1). Cover and reweigh to the nearest 0,001 g.

Place the uncovered dish and its lid in the oven for 3 h or until constant mass is obtained.

NOTE The time required to achieve constant mass can be assessed by individual laboratories for each coal rank.

Remove the dish and dried sample from the oven, replace the lid, and allow to cool on a metal plate, for not longer than 10 min. Transfer the dish and sample to cooling vessel and allow it to cool to room temperature (approximately 20 min), then weigh the dish and sample to the nearest 0,001 g.

8.2 Two stage method

8.2.1 Air dry loss moisture

Weigh a non-corrodible tray to the nearest 0,01 g, spread into it an even layer of not less than 500 g of the coarsely crushed sample (7.2); reweigh to the nearest 0,01 g.

Dry the crushed sample for 2 h in the non-corrodible tray in air at ambient temperature. This procedure can be accelerated by placing the sample on a tray in the drying cabinet at a temperature not exceeding 40 °C and not less than 30 °C. In the latter case, allow the dried sample to attain equilibrium with the atmosphere before reweighing.

Redry the sample until the change in mass of the sample is less than 0,3 % of the original mass over a 2 h period. Take the lowest mass of the successive weighings of the non-corrodible tray and air-dried sample for calculation purposes.

8.2.2 Residual moisture

Crush the air-dried sample (8.2.1) to pass a 3,15 mm aperture sieve.

Weigh a clean, dry, empty weighing dish with its lid to the nearest 0,001 g. Spread into it an even layer of approximately 10 g of the above sample. Cover and reweigh to the nearest 0,001 g.

Place the uncovered dish and its lid in the oven, previously heated to 105 °C to 110 °C and pass nitrogen through it at a rate of approximately 15 oven-volume changes per hour.

Remove the dish with the dried sample from the oven and replace the cover (if possible while the dish is still in the nitrogen-flushed oven, otherwise immediately after removal from the oven). Allow the dish to cool on a thick, metal plate for not longer than 10 min. At the end of the 10 min cooling period, transfer the dish to a cooling vessel and allow it to cool to room temperature. As soon as room temperature is reached, re-weigh to the nearest 0,001 g.

9 Expression of results

9.1 Single-stage method

9.1.1 Total moisture content

The total moisture content, M_t, expressed as mass percent, shall be calculated using Equation (1):

$$M_t = \frac{m_2 - m_3}{m_2 - m_1} \times 100 \quad (1)$$

where

m_1 is the mass of weighing dish and its lid, expressed in grams;

m_2 is the mass of weighing dish, lid and sample before heating, expressed in grams;

m_3 is the mass of weighing dish, lid and sample after heating, expressed in grams.

9.2 Two-stage method

9.2.1 Air dry loss moisture

The air-dry moisture loss, M_f, expressed as mass percent, shall be calculated using Equation (2):

$$M_f = \frac{m_2 - m_3}{m_2 - m_1} \times 100 \quad (2)$$

where

m_1 is the mass of non-corrodible tray, expressed in grams;

m_2 is the mass of non-corrodible tray and sample before drying, expressed in grams;

m_3 is the mass of non-corrodible tray and sample after drying, expressed in grams.

9.2.2 Residual moisture content

The residual moisture content, M_{inh}, expressed as mass percent, is calculated using Equation (3):

$$M_{inh} = \frac{m_2 - m_3}{m_2 - m_1} \times 100 \quad (3)$$

where

m_1 is the mass of weighing dish and its lid, expressed in grams;

m_2 is the mass of weighing dish, its lid and air-dried sample before heating, expressed in grams;

m_3 is the mass of weighing dish, its lid and air-dried sample after heating, expressed in grams.

9.2.3 Total moisture content

The total moisture content, M_t, expressed as mass percent, is calculated using Equation (4):

$$M_t = M_f + \left(1 - \frac{M_f}{100}\right) \times M_{inh} \quad (4)$$

9.3 Take as the final result of the test the arithmetic mean of the results of duplicate determinations. Report the result to the nearest 0,1 %.

10 Precision of method

10.1 Repeatability

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

10.2 Reproducibility critical difference

The mean 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 values given in Table 1.

Table 1 — Precision of the method

Test	Repeatability limit %	Reproducibility critical difference %
Total moisture	0,5	1,2
Residual moisture	0,4	—

11 Test report

The test report shall contain the following information:

a) identification of the product tested;

b) reference of the method used;

c) result and the method of expression used.

Annex A
(informative)

Nitrogen-purification train

It is essential that the nitrogen used for ventilating the minimum-free-space oven should be comparatively pure, since even the small residual amount of oxygen that is found in commercial nitrogen can cause oxidation and hence produce a low result in the moisture figure determined by the loss in mass on drying. A good purification train for nitrogen is, therefore, required. A suitable one is described here, which is capable of dealing with at least 600 ml per minute at a pressure of a few millimetres mercury gauge. A convenient source of the gas is a cylinder of compressed nitrogen. The purification train consists of a quartz tube 500 mm long and with an internal diameter of 37 mm, containing 1,2 kg of reduced copper in wire form. The purification tube is heated by a suitable furnace, which encloses the tube to a length of 380 mm and allows the part of the tube containing the copper wire filing to protrude approximately 80 mm at exit end in order to produce a temperature drop in the gas stream. The reduced copper is heated to approximately 500 °C. The last traces of oxygen are removed by the very large surface area of copper.

If it is necessary to reduce any copper oxide formed during the purification of the nitrogen, this can be done by passing hydrogen through the tube containing the copper, which is heated to 450 °C to 500 °C. During the reduction process, the water formed is expelled to atmosphere. The tube should be purged with nitrogen before use, the reduced copper emptied from the tube and any agglomerated material broken down, and the tube refilled.

Alternatively, a commercial source of nitrogen may be employed in the absence of a purification train, provided that the gas in the cylinder contains not more than 30 µl of oxygen per litre of nitrogen.

In either case, the purified nitrogen shall be passed through a column of magnesium perchlorate (or silica gel) to remove traces of moisture that can be present.

ICS 73.040

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