INTERNATIONAL STANDARD

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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

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Contents

Page

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.

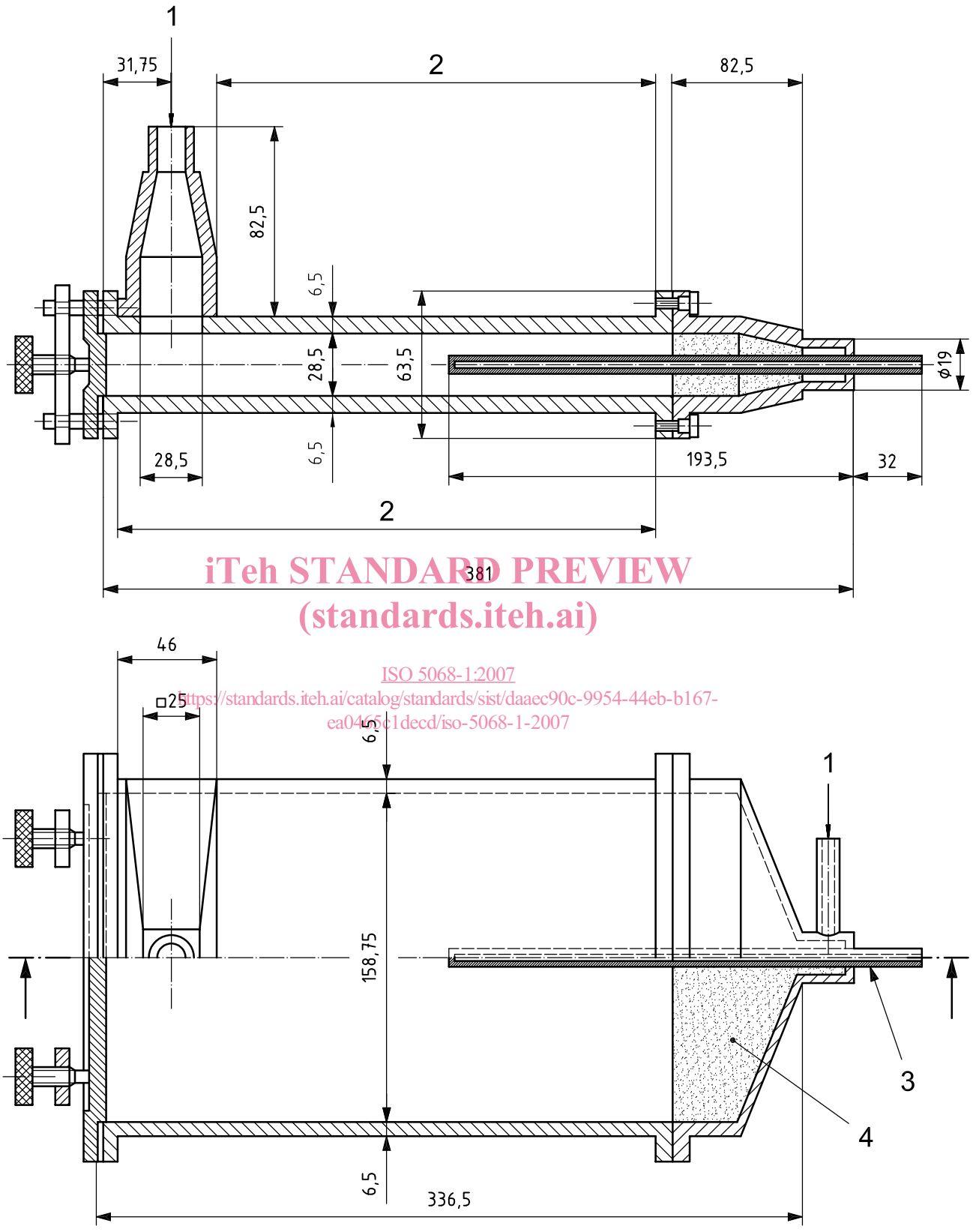
6 Apparatus

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.