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Brown coals and lignites — Determination of moisture content — Direct volumetric method

Charbons bruns et lignites — Détermination de l'humidité — Méthode volumétrique directe

Reference number
ISO 1015:1992(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 1015 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Sub-Committee SC 5, *Methods of analysis*.

This second edition cancels and replaces the first edition (ISO 1015:1975), of which it constitutes a minor revision.

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Brown coals and lignites — Determination of moisture content — Direct volumetric method

1 Scope

This International Standard specifies a direct volumetric method of determining the moisture content of brown coals and lignites. It may be used for the determination of either total moisture or the moisture in the analysis sample.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was 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 edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

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 Principle

A test portion is heated under reflux conditions in a flask containing boiling toluene or xylene. The moisture is entrained by the toluene or xylene vapour and carried to a condenser fitted with a graduated receiver. The water then separates in the receiver to form a lower layer while the excess reagent is returned to the distillation flask by means of an overflow. The moisture content is calculated from the mass of sample taken and the volume of water collected.

NOTE 1 The results obtained using toluene and xylene may not be identical for all brown coals and lignites. It is preferable to use toluene, as opposed to xylene, in cases where the latter may cause liberation of water from coal functional groups. Furthermore, to obtain moisture results comparable to alternative methods such as that described in ISO 5068:1983, *Brown coals and lignites — Determination of moisture content — Indirect gravimetric method*, it is advisable to employ toluene which boils in a range similar to the drying temperature specified in ISO 5068.

4 Reagents

During the analysis, use only reagents of recognized analytical grade, and distilled water or water of equivalent purity.

4.1 Toluene (see note 2), distillation point 110 °C.

or

4.2 Xylene (see note 2), distillation interval 135 °C to 140 °C.

WARNING — Xylene and toluene are flammable, and toxic by inhalation, ingestion or skin absorption.

NOTE 2 In view of the low solubility of water in either toluene or xylene, it can be shown that only a very small error in the determination could arise from variations in the condition of saturation of the entraining reagent. However, in order to reduce this error to insignificance, it is recommended that the reagent be used in the same condition for the determination as during calibration of the apparatus.

5 Apparatus

All graduated apparatus shall be of the best analytical quality available.

5.1 Distillation flask, of minimum capacity 500 ml.

5.2 Condenser, having a minimum length of water jacket of 200 mm, and fitted with an extended lip to direct the distillate into the receiver without touching the sides.

5.3 Receiver, for the condensed water, graduated in 0,1 ml.

It is important that the receiver and condenser be clean. To ensure this, they shall be treated with a cleansing reagent such as a strong solution of potassium dichromate in sulphuric acid.

The condenser, receiver and flask are fitted together by means of ground glass joints. An overflow tube connected to the receiver or to the lower portion of the condenser permits the return of condensed reagent to the distillation flask. The condenser may be fitted to condense either an upward-flowing or downward-flowing vapour stream.

5.4 Pieces of glass tubing, 5 mm in diameter and 5 mm long, with sharp edges, to be used as boiling aids.

5.5 Spray tube, of glass, through which the reagent can be supplied to wash down the inner surface of the condenser. This precaution is required only when an upward-flow condenser is employed.

5.6 Burette, graduated in 0,05 ml divisions.

5.7 Balance, accurate to 10 mg.

6 Preparation of sample

6.1 Prepare the sample for the determination of total moisture in accordance with ISO 5069-2:1983, clause 7.

NOTE 3 If the air-drying process has been carried out according to ISO 5069-2:1983, clause 9, the total moisture, M_T, expressed as a percentage by mass, is calculated from the formula

$$M_T = W_{ex} + M\left(1 - \frac{W_{ex}}{100}\right)$$

where

W_{ex} is the moisture loss due to air-drying, expressed as a percentage by mass;

M is the percentage of residual moisture in the air-dried sample.

6.2 Prepare the sample for the determination of moisture in the analysis sample in accordance with ISO 5069-2:1983, clause 8.

7 Procedure

7.1 Calibration of apparatus

Calibrate each apparatus by distilling a series of accurately known volumes of water, measured using the burette, covering the range of moisture contents likely to be encountered in the samples to be tested.

Plot a graph, showing the volume in millilitres of water added against the scale reading of the water recovered in the receiver (5.3). Use the graph to correct the volume of water obtained in each test.

The calibration shall be repeated when there is any change of reagents or of any part of the apparatus.

7.2 Test portion

Before commencing the determination of moisture in the analysis sample, mix the air-dried sample thoroughly for at least 1 min, preferably by mechanical means.

Weigh, to the nearest 0,01 g, about 50 g of the sample [when the moisture content is expected to be above 20 % (m/m), weigh 25 g], and transfer to the dry distillation flask (5.1). Add 200 ml of the toluene (4.1) or xylene (4.2) in such a way that any sample adhering to the neck or side of the flask is washed down by the reagent.

NOTE 4 The mass of the test portion and the size of the receiver are interrelated. In general, the condensed water should occupy at least one-third of the graduated volume of the receiver (5.3).

7.3 Determination

Fill the receiver with the same reagent. Place two or three pieces of the glass tubing (5.4) in the distillation flask to prevent violent ebullition and assemble the apparatus. Start the flow of water through the condenser (5.2) and heat the flask uniformly and gently so that its contents begin to boil after about 15 min. Subsequently adjust the rate of heating to ensure a distillation rate of 2 to 4 drops per second.

Continue the distillation until the toluene or xylene reflux is clear and no further water collects in the receiver. If a condenser is used for an upward-flowing vapour steam, wash down any drops of water adhering to the inner surface of the condenser or to the upper walls of the receiver with the reagent employed using the spray tube (5.5), and continue the distillation for a sufficient time to ensure that any water washed back into the distillation flask has been carried over into the receiver. Allow the cloudiness of the distillate to clear and read the volume of water collected in the receiver.

8 Expression of results

Assuming that the density of the water is 1 g/ml, the moisture content, M of the analysed sample, expressed as a percentage by mass, is given by the formula

$$M = \frac{V_c}{m} \times 100$$

where

V_c is the corrected volume, in millilitres, of water read from the graph (see 7.1);

m is the mass, in grams, of the test portion.

The result obtained represents

a) the percentage by mass of total moisture in the sample, if the latter has not been air-dried previously; or

b) the percentage of residual moisture, if any air-drying procedure has been included in the preparation of the sample (see 6.1, note 3); or

c) the percentage of moisture in the analysis sample.

The final result shall be reported to the nearest 0,1 % (m/m).

9 Precision of the method

9.1 Repeatability

The maximum acceptable difference between single determinations carried out in one laboratory on two separate moisture samples taken simultaneously, in accordance with ISO 5069-2, shall not exceed the values given in table 1.

9.2 Reproducibility

The maximum acceptable difference between single determinations carried out in different laboratories on two separate moisture samples taken simultaneously, in accordance with ISO 5069-2, shall not exceed the values given in table 1.

Table 1 — Precision data

Moisture content	Maximum acceptable differences between results	
	Repeatability	Reproducibility
Less than 20 % (m/m)	0,4 % (m/m)	0,8 % (m/m)
20 % (m/m) and over	2,0 % of result	4,0 % of result

10 Test report

The test report shall include the following particulars:

a) the reference of the method used;

b) the entraining reagent used and its degree of saturation (i.e "wet" or "dry");

c) the results and the method of expression used;

d) any unusual features noted during the determination;

e) any operation not included in this International Standard or in the International Standard to which reference is made, or any optional operations.

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