INTERNATIONAL STANDARD 1015

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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

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 27 has reviewed ISO Recommendation R 1015 and found it technically suitable for transformation. International Standard ISO 1015 therefore replaces ISO Recommendation R 1015-1969 to which it is technically identical.

ISO Recommendation R 1015 was approved by the Member Bodies of the following countries :

Australia	India	Romania
Austria	Iran	South Africa, Rep. of
Canada	Italy	Spain
Czechoslovakia	Japan	Switzerland
Denmark	Korea, Rep. of	Turkey
Egypt, Arab Rep. of	Netherlands	United Kingdom
France	New Zealand	U.S.S.R.
Germany	Portugal	Yugoslavia

No Member Body expressed disapproval of the Recommendation.

No Member Body disapproved the transformation of ISO/R 1015 into an International Standard.

Printed in Switzerland

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

1 SCOPE AND FIELD OF APPLICATION

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 REFERENCE

ISO . . ., *Brown coals and lignites — Sampling and sample preparation.*[1)]

3 PRINCIPLE

The brown coal or lignite is heated in a flask under reflux conditions with 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 — The results obtained using toluene and using xylene may not be identical for all brown coals and lignites but any differences should be within the tolerance of the method (see clause 9).

4 REAGENTS

The reagent used shall be of analytical reagent quality and distilled water should be used throughout.

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

or

4.2 Xylene (see note), boiling range 135 to 140 °C.

NOTE — 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. In order to reduce this error to insignificance, however, 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 obtainable.

5.1 Distillation flask, capacity 500 ml minimum.

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.

The condenser, receiver and flask are fitted together by means of ground glass joints (see note). 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.

NOTE — 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.

5.4 Glass tubing, pieces 5 mm in diameter and 5 mm long, with sharp edges.

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.

1) In preparation.

5.6 Burette, graduated in 0,05 ml divisions.

5.7 Balance, accurate to 10 mg.

6 PREPARATION OF SAMPLE

6.1 The sample for the determination of total moisture shall be crushed to pass a sieve of 3 mm square aperture. If special mills which prevent loss of moisture are available, the sample may be crushed directly, otherwise the sample shall be brought into approximate moisture equilibrium with the atmosphere before crushing, in which case a formula is used to calculate the total moisture content (see note below). The sample, which will be received in a sealed airtight container, shall weigh not less than 150 g.

NOTE – If an air-drying process has been carried out, the total moisture, M_T, as a percentage by mass, is calculated from the formula

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

where

X is the air-drying loss, as a percentage by mass, of the original sample;

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

6.2 For the determination of moisture in the analysis sample, the sample is crushed to pass a sieve of 0,2 mm aperture and air dried.

7 PROCEDURE

7.1 Calibration of apparatus

Calibrate each apparatus by distilling a series of accurately known volumes of water, measured from 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. 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 %, weigh 25 g), and transfer to the dry distillation flask. 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 – 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.

7.3 Determination

Fill the receiver with the same reagent. Place two or three pieces of the glass tubing in the distillation flask to prevent violent ebullition and assemble the apparatus. Start the flow of water through the condenser 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 stream, 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, 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 sample as analysed, expressed as a percentage, is given by the formula

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

where

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

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

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 an air-drying procedure has been included in the preparation of the sample (see note, 6.1), or

c) the percentage of moisture in the analysis sample.

The final result shall be reported to the nearest 0,1 %.

9 PRECISION OF THE METHOD

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

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 the principles laid down in ISO . . ., shall not exceed the value given above.

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 the principles laid down in ISO . . ., shall not exceed the value given above.

10 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard, or regarded as optional;

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