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ISO RECOMMENDATION

R 1015

DETERMINATION OF MOISTURE IN BROWN COALS AND LIGNITES BY THE DIRECT VOLUMETRIC METHOD

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BRIEF HISTORY

The ISO Recommendation R 1015, *Determination of moisture in brown coals and lignites by the direct volumetric method,* was drawn up by Technical Committee ISO/TC 27, *Solid mineral fuels,* the Secretariat of which is held by the British Standards Institution (BSI).

Work on this question led, in 1966, to the adoption of a Draft ISO Recommendation.

In August 1967, this Draft ISO Recommendation (No. 1282) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in March 1969, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 1015 March 1969

DETERMINATION OF MOISTURE IN BROWN COALS AND LIGNITES BY THE DIRECT VOLUMETRIC METHOD

1. SCOPE

This ISO Recommendation describes 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. 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 whilst 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 section 8).

3. REAGENTS

All reagents should be of analytical reagent quality and distilled water should be used throughout.

3.1 *Toluene,* boiling point 110 °C.

3.2 *Xylene,* 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 should be used in the same condition for the determination as during calibration of the apparatus.

4. APPARATUS

4.1 *Distillation flask,* capacity 500 ml minimum.

4.2 *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.

4.3 *Receiver,* for the condensed water, graduated in 0.1 ml divisions. 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.

NOTES

1. The condenser, receiver and flask are fitted together by means of ground glass joints. The condenser may be fitted to condense either an upward flowing or downward flowing vapour stream.

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

4.4 *Glass tubing.* Pieces of glass tubing, 5 mm in diameter and 5 mm long, with sharp edges.

4.5 *Spray tube.* A glass tube 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.

4.6 *Burette,* graduated in 0.05 ml divisions.

5. SAMPLE

5.1 The sample for the determination of total moisture should 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 should 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, should weigh not less than 150 g.

NOTE. – If an air-drying process has been carried out, the total moisture (*TM*), expressed as a percentage, by mass, is calculated from the following formula :

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

where

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

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

5.2 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.

6. PROCEDURE

6.1 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 fuels 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 should be repeated when there is any change of reagents or of any part of the apparatus.