INTERNATIONAL STANDARD 589

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Hard coal — Determination of total moisture

Houille — Détermination de l'humidité totale

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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 589 and found it technically suitable for transformation. International Standard ISO 589 therefore replaces ISO Recommendation R 589-1967 to which it is technically identical.

ISO Recommendation R 589 was approved by the Member Bodies of the following countries :

Australia	India	South Africa, Rep. of
Austria	Iran	Spain
Belgium	Italy	Sweden (for Method A)
Brazil	Japan	Switzerland
Chile	Netherlands	Turkey
Czechoslovakia	New Zealand	United Kingdom
Denmark	Philippines	U.S.S.R.
France	Poland	Yugoslavia
Germany	Portugal	
Greece	Romania	

The Member Bodies of the following countries expressed disapproval of the Recommendation on technical grounds :

Sweden (for Methods B and C)*
U.S.A.*

No Member Body disapproved the transformation of ISO/R 589 into an International Standard.

* Subsequently, these Member bodies approved the Recommendation.

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Hard coal — Determination of total moisture

0 INTRODUCTION

The moisture content of coal is not an absolute value and conditions for its determination have to be standardized. Results given by the different methods specified here should be comparable within the limits of tolerance quoted.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies three methods of determining the total moisture content of hard coal. Two of the methods are applicable to all coals, but the third shall be used only for coals which are not susceptible to oxidation.

2 PRINCIPLE

2.1 **Method A** (for all hard coals)

The sample is heated in a flask under reflux conditions with boiling toluene. The moisture from the coal is entrained by the toluene vapour and carried to a condenser fitted with a graduated receiver. The water then separates in the receiver, to form the lower layer, whilst the excess toluene is returned to the distillation flask by means of an overflow. The moisture in the coal is calculated from the mass of the sample and the volume of water collected.

2.2 **Method B** (for all hard coals)

The sample is dried in an oven at a temperature of 105 to 110 °C in a current of nitrogen and the moisture calculated from the loss in mass.

2.3 **Method C** (only for hard coals not susceptible to oxidation, see 9.1)

The sample is dried at a temperature of 105 to 110 °C in air and the moisture calculated from the loss in mass.

3 PREPARATION OF SAMPLE

3.1 Samples for the determination of moisture shall be received in sealed air-tight containers.

3.2 The sample mass shall not be less than 300 g; for methods A and B the maximum particle size shall not exceed 3 mm; for method C, which is normally applicable to samples with a maximum particle size of about 20 mm, the sample mass, in kilograms, shall not be less than 0,06 times the maximum particle size in millimetres.

3.3 During the course of its preparation the sample may have been air-dried, in which case a formula shall be used to calculate the total moisture content (see clause 7).

3.4 Before commencing a determination, either by method A or method B, or in accordance with 9.6, mix the sample thoroughly in the closed container for at least 1 min, preferably by mechanical means.

4 METHOD A

4.1 Reagent

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

Toluene (see 9.3), boiling point 110 °C.

4.2 Apparatus

All graduated apparatus shall be of the best analytical quality available, and the balance used shall be sensitive to 100 mg.

4.2.1 Distillation flask, minimum capacity 500 ml.

4.2.2 Condenser, minimum length 200 mm, fitted with an extended lip to direct the distillate into the receiver along its axis without touching the sides (see 9.3).

4.2.3 Receiver, for the condensed water, graduated in 0,1 ml (see 9.2).

An overflow tube shall be connected to the receiver or to the lower portion of the condenser to permit the return of condensed toluene to the distillation flask. The condenser may be fitted to condense either an upward flowing or downward flowing vapour stream.

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The condenser, receiver and flask shall be fitted together by means of ground joints.

4.2.4 Glass tubing, pieces 5 mm in diameter and 5 mm long, with sharp edges (or other suitable means of preventing violent ebullition).

4.2.5 Spray tube : a glass tube through which toluene can be supplied to wash down the inner surface of the condenser (only required when an upward flow condenser is employed).

4.3 Procedure

4.3.1 *Determination*

Weigh, to the nearest 0,1 g, about 100 g of the sample (see 9.4) and transfer to the dry distillation flask. Add 200 ml of the toluene (4.1) in such a way that any coal adhering to the neck or sides of the distillation flask is washed down by the reagent. Place two or three pieces of glass tubing (4.2.4) in the distillation flask to prevent violent ebullition, fill the receiver with the toluene (4.1) and assemble the apparatus. Heat the distillation flask and keep the contents boiling briskly.

Continue the distillation until no further water collects in the graduated receiver. If an upward condenser is used, wash down any drops of water adhering to the inner surface of the condenser or to the upper walls of the receiver with the toluene, using the spray tube. Continue the distillation for a sufficient time to ensure than any water washed back into the distillation flask has been carried over into the receiver. Read the volume of the distillate after any cloudiness has dispersed.

4.3.2 *Calibration*

Standardize the apparatus by distilling a series of known volumes of water, accurately measured, for example by a microburette, covering the range of moisture contents in the fuels likely to be encountered. Plot a graph, showing the millilitres of water added against the scale reading of the water in the receiver, and use it to correct the volume of water obtained in each test.

4.4 Expression of results

The moisture (M) in the coal as analysed, in per cent, is given by the formula

$$M = \frac{m_2 \times 100}{m_1}$$

where

m_1 is the mass, in grams, of coal taken;

m_2 is the mass, in grams, of water collected (numerically equal to the corrected volume, in millilitres, of water collected).

The result shall be reported to the nearest 0,1 %, stating that the determination has been carried out by method A.

5 METHOD B

5.1 Reagents

5.1.1 Nitrogen, dry and containing less than 30 ppm of oxygen (see the annex).

5.1.2 Desiccant : either fresh or freshly regenerated silica gel or other desiccant, for use in the desiccator.

5.2 Apparatus

The balance used shall be sensitive to 1 mg.

5.2.1 Nitrogen oven, capable of being maintained at a temperature within the range 105 to 110 °C and with provision for passing a current of dry oxygen-free nitrogen through it at a rate sufficient to change the atmosphere 15 times per hour. A suitable oven is illustrated in the figure.

5.2.2 Weighing vessels, shallow, of silica or glass, with ground edges and fitted with ground-on covers, or of non-corrodible and heat resistant material with well-fitting lids. The diameter of each vessel shall be such that the mass of the coal layer does not exceed 0,3 g/cm² for a 10 g sample.

5.3 Procedure

Weigh, to the nearest 0,01 g, a clean, dry empty vessel and its cover and spread uniformly into it not less than 10 g of sample. Weigh the covered vessel and its contents to determine the mass of coal taken.

Place the cover in a desiccator and heat the uncovered vessel in the oven at a temperature of 105 to 110 °C until constant in mass (see 9.5). Replace the cover, cool rapidly on a metal plate for 10 min, transfer to a desiccator and weigh after a further 10 min.

5.4 Expression of results

The moisture (M) in the coal as analysed, in per cent, is given by the formula

$$M = \frac{m_2 - m_3}{m_2 - m_1} \times 100$$

where

m_1 is the mass, in grams, of the empty vessel plus cover;

m_2 is the mass, in grams, of the vessel plus cover plus sample before heating;

m_3 is the mass, in grams, of the vessel plus cover plus sample after heating.

The result shall be reported to the nearest 0,1 %, stating that the determination has been carried out by method B.

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Dimensions in millimetres

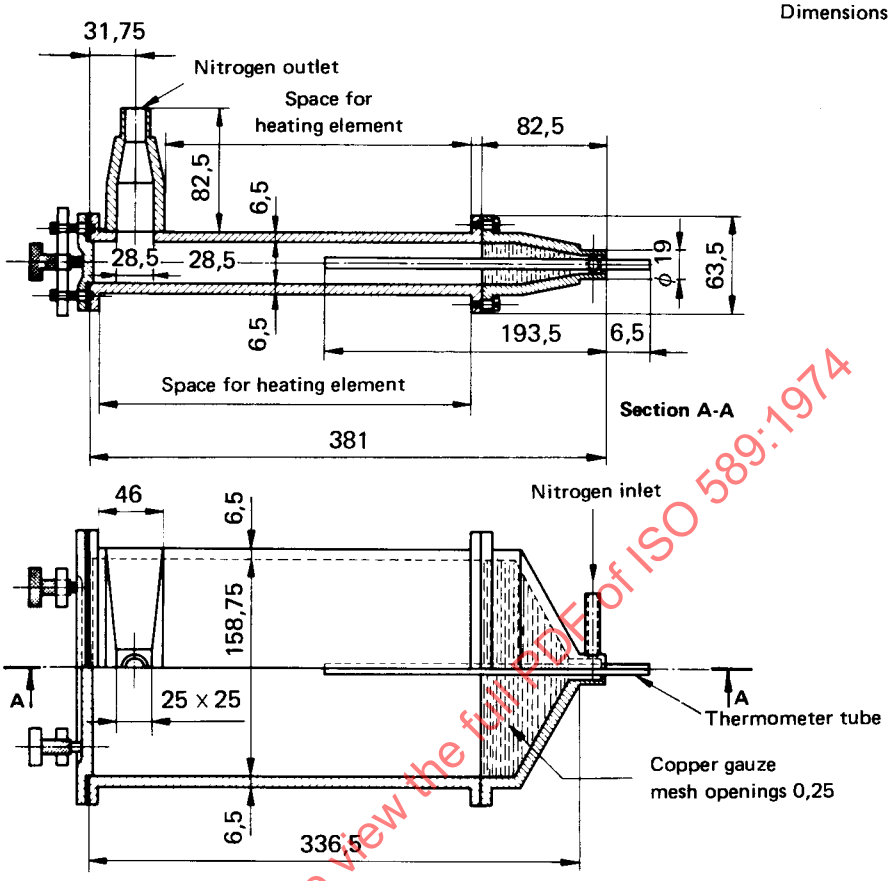

FIGURE – **Suitable nitrogen oven**

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6 METHOD C

6.1 Apparatus

The balance used shall be sufficiently sensitive to enable the sample and container, as received, to be weighed to the nearest 0,1 %.

6.1.1 Air oven, capable of maintaining a temperature within the range of 105 to 110 °C with a sufficiently rapid rate of atmosphere change, for example 3 to 5 times per hour.

6.1.2 Trays of non-corrodible and heat resistant material, and of such dimensions that they will hold the total sample (see 3.2) in the proportion of approximately 1 g of sample per cm^2 of surface area.

6.2 Procedure

Weigh the sample and container, as received, to the nearest 0,1 % (if the sample is 3 mm top size, see 9.6). Weigh a dry empty tray, transfer the sample as completely as possible to the tray and spread evenly, allowing about 1 cm^2 of surface to 1 g of sample. Dry the wet container with any sample adhering to it by warming, then transfer the remaining sample to the tray and weigh the dry empty container.

Place the charged tray in the oven at a temperature of 105 to 110 °C. Heat the tray and its contents until constant in mass (see 9.5), weighing while hot to avoid absorption of moisture during cooling. The time required may be from 3 to 6 h, or more, depending on the particle size of the coal.

6.3 Expression of results

The moisture (M) in the coal as analysed, in per cent, is given by the formula

$$M = \frac{(m_1 - m_4) - (m_3 - m_2)}{(m_1 - m_4)} \times 100$$

where

m_1 is the mass, in grams, of container plus sample as received;

m_2 is the mass, in grams, of empty tray;

m_3 is the mass, in grams, of tray plus sample after heating;

m_4 is the mass, in grams, of dry empty container.

The result shall be reported to the nearest 0,1 %, stating that the determination has been carried out by method C.

7 CALCULATION AND REPORTING OF TOTAL MOISTURE

7.1 Sample not air dried

Where air drying was not carried out during the preparation of the sample, the moisture in the coal as analysed, *M* (see 4.4, 5.4 or 6.3), shall be reported as the total moisture.

7.2 Sample air dried

Where air drying was carried out during the preparation of the sample, in accordance with the procedures specified in ISO 1988[1], the total moisture, in per cent, is given by the formula

$$X + M\,(1 - X/100)$$

where

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

M is the residual moisture, in per cent, determined in the air-dried sample.

7.3 Reporting

The result for the determination of total moisture shall be reported to the nearest 0,1 % and the method of determination shall be given.

8 PRECISION OF THE METHOD

Moisture content	Maximum acceptable differences between results	
	Repeatability	Reproducibility
less than 10 %	0,5 % absolute	(see 8.2)
10 % and over	one twentieth of the mean result	(see 8.2)

8.1 Repeatability

The results of duplicate determinations (see 9.7), carried out at different times in the same laboratory by the same operator with the same apparatus on representative portions taken from the same gross sample, shall not differ by more than the above values.

8.2 Reproducibility

No value for reproducibility can be quoted for determinations carried out in different laboratories, since insufficient evidence is available for this to be done.

9 NOTES ON PROCEDURE

9.1 In general, coals may be regarded as not susceptible to oxidation if they belong to classes 0 to 5 inclusive of the International Classification of Hard Coals by Type, adopted by the United Nations Economic Commission for Europe; in case of doubt, method A or B shall be used.

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

9.3 The solubility of water in toluene is small and only a very slight error in the determination can arise from variation in the condition of saturation of the entraining reagent. In order to reduce this error to insignificance, it is recommended that the reagent should be used in the same condition during the determination as during the calibration of the apparatus.

9.4 Alternatively, 50 g of the sample may be taken if the moisture content is such that the capacity of the receiver is likely to be exceeded if 100 g of sample is taken.

9.5 Constancy in mass is defined as a change not exceeding 0,2 % of the total loss in mass during a further period of heating of not less than 30 min.

9.6 If the sample received for method C has been prepared by crushing to 3 mm in size, not less than 10 g shall be taken and method B shall be used, except that the air oven (6.1.1) replaces the nitrogen oven (5.2.1).

9.7 Duplicate determinations by method C can be carried out on duplicate samples taken in accordance with the procedure specified in ISO 1988.

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.

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1) ISO 1988, *Hard coal — Sampling.* (At present at the stage of draft.)