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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION
R 589

DETERMINATION OF TOTAL MOISTURE IN HARD COAL

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BRIEF HISTORY

The ISO Recommendation R 589, *Determination of Total Moisture in Hard Coal*, 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 by the Technical Committee began in 1950 and led, in 1961, to the adoption of a Draft ISO Recommendation.

In October 1962, this Draft ISO Recommendation (No. 553) 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	Greece	Republic of South Africa
Austria	India	Romania
Belgium	Iran	Spain
Brazil	Italy	Sweden (for Method A)
Chile	Japan	Switzerland
Czechoslovakia	Netherlands	Turkey
Denmark	New Zealand	United Kingdom
France	Poland	U.S.S.R.
Germany	Portugal	Yugoslavia

Two Member Bodies opposed the approval of the Draft:

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

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in July 1967, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 589 July 1967

DETERMINATION OF TOTAL MOISTURE IN HARD COAL

1. SCOPE

This ISO Recommendation describes three methods of determining the total moisture content of hard coal. Two of the methods are applicable in all cases, but the third should be used only for coals which are not susceptible to oxidation.

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 recommended should be comparable within the limits of tolerance quoted.

2. PRINCIPLE

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

The coal is heated in a flask under reflux conditions with toluene or xylene (see Note 1, page 10). The moisture from the coal 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 the lower layer, whilst the excess toluene or xylene 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 Note 2, page 10)

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

3. SAMPLE

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

3.2 The sample mass will not be less than 300 g; for methods A and B the maximum particle size will 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 kilogrammes will 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 is used to calculate the total moisture content (see Note 3, page 10).

3.4 Before commencing a determination, either by method A or method B, or in accordance with Note 8, page 10, of method C, mix the sample thoroughly in a closed container for at least one minute, preferably by mechanical means.

4. METHOD A

4.1 Reagents

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

4.1.1 *Toluene* (see Note 5, page 10). Boiling point 110 °C.

4.1.2 *Xylene* (see Note 5, page 10). Boiling range 135 to 140 °C.

4.2 Apparatus

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

4.2.1 *Distillation flask*, of minimum capacity 500 ml.

4.2.2 *Condenser*, of 200 mm minimum length, fitted with an extended lip to direct the distillate into the receiver without touching the sides (see Note 4, page 10).

4.2.3 *Receiver*, for the condensed water, graduated in tenths of a millilitre (see Note 4, page 10). An overflow tube connected to the receiver or to the lower portion of the condenser permits the return of condensed toluene or xylene to the distillation flask. The condenser may be fitted to condense either an upward flowing or downward flowing vapour stream.

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

4.2.4 *Glass tubing*. Pieces of glass tubing 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 or xylene 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 *Test*. Weigh to the nearest 0.1 g about 100 g of the sample (see Note 6, page 10) and transfer to the dry distillation flask. Add 200 ml of the toluene (4.1.1) or of the xylene (4.1.2) 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 (4.1.1) or the xylene (4.1.2) 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 reagent employed, using the spray tube. 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. 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, e.g. 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 Calculation and reporting of result

If m = mass of coal taken, expressed in grammes,

v_c = corrected volume of water, read from graph, expressed in millilitres,

M = moisture in the coal analysed, per cent,

then

$$M = \frac{v_c \times 100}{m}$$ (assuming that 1 ml of water has the mass of 1 g).

The result obtained represents:

either (1) the percentage of total moisture in the sample, if the latter has not previously been air-dried;

or (2) the percentage of residual moisture if the sample has previously been air-dried (see Note 3, page 10).

The final result should be expressed to the nearest 0.1%, stating that the determination has been carried out by method A and whether toluene or xylene has been used.

5. METHOD B

5.1 Reagents

5.1.1 *Nitrogen.* Dry and containing less than 30 parts per million of oxygen (see Annex A, page 11).

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 should be sensitive to 1 mg.

5.2.1 *Nitrogen oven.* An 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 Figure, page 11.

5.2.2 *Weighing vessels.* Shallow vessels, 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 should be such that the weight 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 Note 7, page 10). Replace the cover, cool rapidly on a metal plate for 10 minutes, transfer to a desiccator and weigh after a further 10 minutes.

5.4 Calculation and reporting of result

If m_1 = mass of empty vessel plus cover, expressed in grammes,
m_2 = mass of vessel plus cover plus sample before heating, expressed in grammes,
m_3 = mass of vessel plus cover plus sample after heating, expressed in grammes,
M = moisture in the coal analysed, per cent,

then

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

The result obtained represents:

either (1) the percentage of total moisture in the sample, if the latter has not previously been air-dried;

or (2) the percentage of residual moisture if the sample has previously been air-dried (see Note 3, page 10).

The final result should be expressed to the nearest 0.1%, stating that the determination has been carried out by method B.

6. METHOD C

6.1 Apparatus

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

6.1.1 *Air oven.* An oven capable of maintaining a temperature within the range of 105 to 110 °C with a sufficiently rapid rate of atmosphere change, e.g. 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 clause 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 Note 8, page 10). 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 Note 7, page 10), weighing whilst hot to avoid absorption of moisture during cooling. The time required may be from three to six hours, or more, depending on the particle size of the coal.

6.3 Calculation and reporting of result

If m_1 = mass of container plus sample as received, expressed in grammes,
m_2 = mass of empty tray, expressed in grammes,
m_3 = mass of tray plus sample after heating, expressed in grammes,
m_4 = mass of dry empty container, expressed in grammes,
M = moisture in the coal analysed, per cent,

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

The result obtained represents:

either (1) the percentage of total moisture in the sample, if the latter has not previously been air-dried;

or (2) the percentage of residual moisture if the sample has previously been air-dried (see Note 3, page 10).

The final result should be expressed to the nearest 0.1 %, stating that the determination has been carried out by method C.

7. PRECISION OF DETERMINATION

Moisture content	Maximum acceptable differences between results obtained	
	in the same laboratory	in different laboratories
less than 10%	0.5% absolute	(see clause 7.2)
10% and over	one twentieth of the mean result	(see clause 7.2)

7.1 In the same laboratory

The results of duplicate determinations (see Note 9, page 10), carried out at different times in the same laboratory by the same operator with the same apparatus on representative portions taken from the same laboratory sample should not differ by more than the above values.

7.2 In different laboratories

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

NOTES

1. The results obtained using toluene and using xylene may not be identical for all coals but any differences should be within the tolerance of the method (see section 7).

2. 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 should be used.

3. The air-drying procedure for the preparation of the moisture sample is described in ISO Recommendation R *, dealing with sampling and sample preparation; the air-drying of visibly wet samples must be carried out in the same laboratory as the determination of residual moisture.

   If X = the air-drying loss expressed as a percentage of the original sample,

   and M = the percentage of residual moisture determined in the air-dried sample,

   then total moisture, per cent $= X + M\left(1 - \frac{X}{100}\right)$.

4. 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.

5. The solubility of water in either toluene or xylene 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.

6. 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.

7. 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 minutes.

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

9. Duplicate determinations by method C can be carried out on duplicate samples taken in accordance with the procedure described in ISO Recommendation R *.

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* At present at the stage of draft proposal.

ANNEX A

NITROGEN PURIFICATION TRAIN

It is essential that the nitrogen used for ventilating the minimum free space oven should be comparatively pure, since even the small residual amount of oxygen which is found in commercial nitrogen may cause oxidation and hence produce a low result in the moisture figure determined by the loss in mass on drying. A good purification train for nitrogen is therefore required; a suitable one is described here which is capable of dealing with at least 600 ml per minute at a pressure of a few millimetres mercury gauge. A convenient source of the gas is a cylinder of compressed nitrogen. The purification train consists of a quartz tube 500 mm long and 37 mm internal diameter, containing 1.2 kg of reduced copper in wire form. The purification tube is heated by a suitable furnace, which encloses the tube to a length of 380 mm and allows part of the copper wire filling to protrude approximately 80 mm at the exit end in order to produce a temperature drop in the gas stream. The reduced copper is heated to approximately 500 °C. The last traces of oxygen are removed by the very large surface area of the copper.

If it is necessary to reduce any copper oxide formed during the purification of the nitrogen, this can be done by passing hydrogen through the tube containing the copper, which is heated to 450 to 500 °C. During the reduction process the water formed is expelled to atmosphere. The tube should be purged with nitrogen before use, the reduced copper emptied from the tube and any agglomerated material broken down, and the tube refilled.

Alternatively, a commercial source of nitrogen may be employed in the absence of a purification train, provided the gas in the cylinder contains not more than 30 parts per million of oxygen.

In either case, the purified nitrogen is passed through a column of magnesium perchlorate to remove traces of moisture which may be present.

Dimensions in millimetres

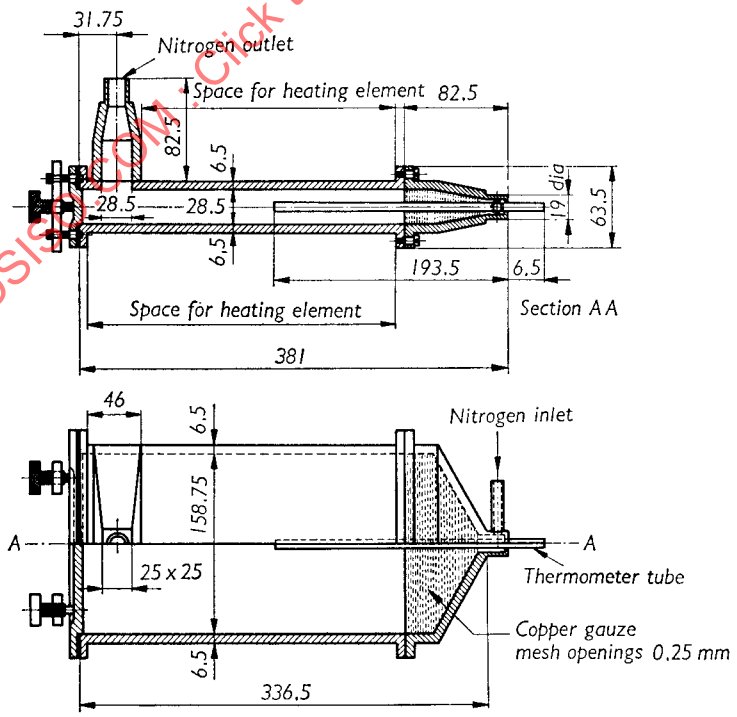

FIGURE. — **Suitable nitrogen oven**