International Standard 589

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Hard coal — Determination of total moisture

Houille — Détermination de l'humidité totale

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Foreword

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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.

International Standard ISO 589 was developed by Technical Committee ISO/TC 27, *Solid mineral fuels*.

This second edition was submitted directly to the ISO Council, in accordance with clause 5.10.1 of part 1 of the Directives for the technical work of ISO. It cancels and replaces the first edition (i.e. ISO 589-1974), which had been approved by the member bodies of the following countries :

Australia
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No member body had expressed disapproval of the document.

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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 the tolerances 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 References

ISO 1988, *Hard coal — Sampling.*

ISO 5272, *Toluene for industrial use — Specifications.*

3 Principle

3.1 **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.

3.2 **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.

3.3 **Method C** (only for hard coals not susceptible to oxidation, see 10.1)

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

4 Preparation of sample

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

4.2 The sample mass shall be not 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 be not less than 0,06 times the maximum particle size in millimetres.

4.3 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 8).

4.4 Before commencing a determination, either by method A or method B, or in accordance with 10.6, mix the sample thoroughly in the closed container for at least 1 min, preferably by mechanical means.

5 Method A

5.1 Reagent

During the analysis, use only distilled water or water of equivalent purity.

5.1.1 Toluene (see 10.3), complying with the requirements of ISO 5272.

5.2 Apparatus

Ordinary laboratory apparatus : the graduated apparatus shall be in conformity with the International Standards drawn up by ISO/TC 48, *Laboratory glassware and related apparatus.*

5.2.1 Distillation flask, of minimum capacity 500 ml.

5.2.2 Condenser, of minimum length 200 mm, fitted with an extended lip to direct the distillate into the receiver along its axis without touching the sides (see 10.3).

5.2.3 Receiver, for the condensed water, graduated in 0,1 ml (see 10.2).

An overflow tube connected to the receiver or to the lower portion of the condenser permits 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.

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

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

5.2.5 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).

5.3 Procedure

5.3.1 Determination

Weigh, to the nearest 0,1 g, about 100 g of the sample (see 10.4) and transfer to the dry distillation flask (5.2.1). Add 200 ml of the toluene (5.1.1) 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 (5.2.4) in the distillation flask to prevent violent ebullition, fill the receiver (5.2.3) with the toluene (5.1.1) 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 (5.2.3) and then for a further 10 min. To remove any drops of water adhering to the surface of the apparatus, a few minutes before the end of the distillation, turn off the water to the condenser to allow the toluene vapour to wash down any drops of water; other appropriate procedures may also be used.

Read the volume V of water to the nearest 0,5 ml. If the upper layer of solvent in the receiver is not clear, place the receiver in a water bath heated to 60 °C for 20 min, allow to cool to toom temperature and read the volume of water.

5.3.2 Calibration

Calibrate the apparatus by distilling a series of known volumes of water, accurately measured, for example by means of 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 millilitres of water recovered, and use it to correct the volume of water obtained in each test.

5.4 Expression of results

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

$$\frac{V_0}{m} \times 100$$

where

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

V_0 is the corrected volume of water, in millilitres, read from the graph.

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

6 Method B

6.1 Reagents

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

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

6.2 Apparatus

Ordinary laboratory apparatus and

6.2.1 Nitrogen oven, capable of being controlled 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.

6.2.2 Weighing bottles, 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 bottle shall be such that the surface (mass) density of the coal layer does not exceed 0,3 g/cm^2 for a 10 g sample.

6.2.3 Balance, accurate to 1 mg.

6.3 Procedure

Weigh, to the nearest 0,01 g, a clean, dry empty bottle (6.2.2) and its cover and spread uniformly into it not less than 10 g of the sample. Weigh the covered bottle and its contents to determine the mass of coal taken.

Place the cover in a desiccator and heat the uncovered bottle in the oven (6.2.1), controlled at 105 to 110 °C, until constant in mass (see 10.5). Replace the cover, cool rapidly on a metal plate for 10 min, transfer to a desiccator and weigh after a further 10 min.

6.4 Expression of results

The moisture, M, in the coal as analysed, expressed as a percentage by mass, is given by the formula

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

where

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

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

m_3 is the mass, in grams, of the bottle 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.

Dimensions in millimetres

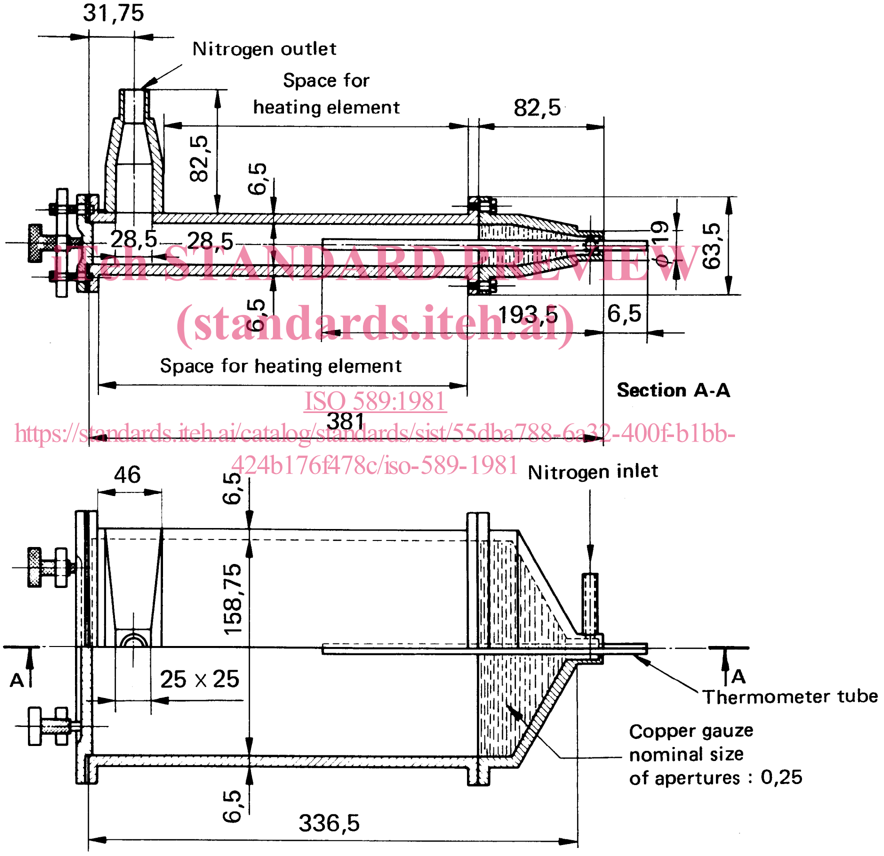

Figure — Suitable nitrogen oven

7 Method C

7.1 Apparatus

Ordinary laboratory apparatus and

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

7.1.2 Trays, of non-corrodible and heat-resistant material, and of such dimensions that they will hold the total sample (see 4.2) in the proportion of approximately 1 g of sample per square centimetre of surface area.

7.1.3 Balance, sufficiently accurate to enable the sample and container, as received, to be weighed to the nearest 0,1 %.

7.2 Procedure

Weigh the sample and container, as received to the nearest 0,1 % (if the sample is 3 mm top size, see 10.6). Weigh a dry empty tray (7.1.2), transfer the sample as completely as possible to the tray and spread evenly, allowing about 1 cm^2 of surface area 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 (7.1.1), controlled at 105 to 110 °C. Heat the tray and its contents until constant in mass (see 10.5), 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.

7.3 Expression of results

The moisture, M, in the coal as analysed, expressed as a percentage by mass, is given by the formula

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

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.

8 Calculation and reporting of total moisture

8.1 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 5.4, 6.4 or 7.3), shall be reported as the total moisture.

8.2 Sample air dried

Where air drying was carried out during the preparation of the sample, in accordance with the procedures specified in ISO 1988, the total moisture, expressed as a percentage by mass, is given by the formula

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

where

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

M is the residual moisture, expressed as a percentage by mass, determined in the air-dried sample.

8.3 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.

9 Precision of the methods

Moisture content	Maximum acceptable differences between results	
	Same laboratories (Repeatability)	Different laboratories (Reproducibility)
less than 10 %	0,5 % absolute	(see 9.2)
10 % and over	one twentieth of the mean result	(see 9.2)

9.1 Repeatability

The results of duplicate determinations (see 10.7), 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.

9.2 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.

10 Notes on procedure

10.1 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.

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

10.3 In view of the low miscibility of water with toluene, it can be shown that only a very small possibility of error in the determination may arise from variation in the condition of saturation of the entraining reagent. In order to reduce this error to insignificance, however, it is recommended as a precaution that the reagent should be used in the same condition during the determination as during the calibration of the apparatus.

10.4 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 when 100 g of sample is taken.

10.5 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.

10.6 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 (7.1.1) replaces the nitrogen oven (6.2.1).

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

11 Test report

The test report shall include the following particulars :

a) an identification of the product tested;

b) the reference of the method used;

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 regarded as optional.

Annex

Nitrogen purification train

(Forms part of the Standard.)

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 the part of the tube containing 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 ppm of oxygen.

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