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Methods for

Analysis and testing of coal and coke —

Part 21: Determination of moisture-holding capacity of hard coal

[ISO title: Hard coal — Determination of moisture-holding capacity]

UDC [662.66 + 662.74]:662.66:543.817

Cooperating organizations

The Solid Fuel Standards Committee, under whose direction this British Standard was prepared, consists of representatives from the following:

Association of Consulting Engineers
British Carbonization Research Association*
British Ironfounders Association
Chamber of Coal Traders
Chartered Institution of Building Services
Coke Oven Managers' Association
Combustion Engineering Association
Consumer Standards Advisory Committee of BSI
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Domestic Solid Fuel Appliances Approval Scheme
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Health and Safety Executive
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Institute of Energy
Low Temperature Coal Distillers Association of Great Britain Ltd.
National Coal Board*
Society of British Gas Industries
Solid Smokeless Fuels Federation*
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The organizations marked with an asterisk in the above list, together with the following, were directly represented on the Technical Committee entrusted with the preparation of this British Standard:

British Gas Corporation
British Steel Corporation
Cement Makers' Federation
Chemical Industries Association
Institute of British Foundrymen
Institute of Petroleum
Scientific Instrument Manufacturers' Association
Individual experts

This British Standard, having been prepared under the direction of the Solid Fuel Standards Committee, was published under the authority of the Executive Board and comes into effect on 31 August 1981

The following BSI references relate to the work on this standard:
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Amd. No.	Date of issue	Comments

Contents

National foreword

This British Standard, which has been prepared under the direction of the Solid Fuel Standards Committee, is identical with ISO 1018 *Hard coal — Determination of moisture-holding capacity*, published in 1975 by the International Organization for Standardization (ISO), and prepared by ISO/TC 27, *Solid mineral fuels*.

This British Standard is one of a series issued under the same number, BS 1016, dealing with methods for analysis and testing of coal and coke; the other Parts are as follows:

- — *Part 1: Total moisture of coal*[1]*;*
- — *Part 2: Total moisture of coke;*
- — *Part 3: Proximate analysis of coal;*
- — *Part 4: Moisture, volatile matter and ash in the analysis sample of coke;*
- — *Part 5: Gross calorific value of coal and coke;*
- — *Part 6: Ultimate analysis of coal;*
- — *Part 7: Ultimate analysis of coke;*
- — *Part 8: Chlorine in coal and coke;*
- — *Part 9: Phosphorus in coal and coke;*
- — *Part 10: Arsenic in coal and coke;*
- — *Part 11: Forms of sulphur in coal;*
- — *Part 12: Caking and swelling properties of coal;*
- — *Part 13: Tests special to coke;*
- — *Part 14; Analysis of coal ash and coke ash;*
- — *Part 15: Fusibility of coal ash and coke ash;*
- — *Part 16: Reporting of results*[1]*;*
- — *Part 17: Size analysis of coal;*
- — *Part 18: Size analysis of coke;*
- — *Part 19: Determination of the index of abrasion of coal;*
- — *Part 20: Determination of Hardgrove grindability index of hard coal.*

Terminology and conventions. The text of the International Standard has been approved as suitable for publication as a British Standard without deviation. Some terminology and certain conventions are not identical with those used in British Standards; attention is especially drawn to the following.

The comma has been used throughout as a decimal marker. In British Standards it is current practice to use a full point on the baseline as the decimal marker.

Where the words "International Standard" appear, referring to this standard, they should be read as "British Standard".

Cross-references. The Technical Committee has reviewed the provisions of ISO 589:1974, to which reference is made in the footnote on page 1, and ISO 1988:1975, to which reference is made in clauses **2** and **6**, and has decided that they are suitable for use in conjunction with this standard. The related British Standards for ISO 589 and ISO 1988 are BS 1016 "*Methods for the analysis and testing of coal and coke*" — Part 1:1973 "*Total moisture of coal*" and BS 1017 "*Methods for sampling of coal and coke*" — Part 1:1977 "*Sampling of coal*" respectively.

NOTE ISO 1988 and BS 1017-1 have some technical differences but both are under revision; it is expected that the BS will be identical with the ISO Standard when the revision is complete.

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[1] Under revision.

HAZARD WARNING. This British Standard method does not necessarily detail all precautions necessary to meet the requirements of the Health and Safety at Work etc. Act 1974. Attention should be paid to any appropriate safety precautions, particularly with regard to the use of vacuum desiccators, and the method should be operated only by trained personnel.

NOTE *Textual errors*. When adopting the text of the International Standard the errors given below were noticed. They have been marked in this British Standard and have been reported to ISO in a proposal to amend the text of the International Standard.

In the plan view of Figure 1, the top positioning stud on the top right hand dish has been omitted.

In **7.3.1** the lid of the dish should be stored in a desiccator whilst the dish is in the conditioning vessel.

In **A.3** the lid of the dish should be placed in the oven with the dish and not in a desiccator.

Summary of pages

This document comprises a front cover, an inside front cover, pages i to iv, pages 1 to 6, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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0 Introduction

The moisture-holding capacity indicates the rank of hard coals and is used in coal classification for correcting the calorific value of the sample to the moist mineral matter-free basis. The full moisture-holding capacity is that of the coal in equilibrium with an atmosphere saturated with water vapour. Since there are insuperable experimental difficulties in working with such an atmosphere, the determination is carried out at 96 % relative humidity.

1 Scope and field of application

This International Standard specifies a method of determining the moisture-holding capacity of hard coals.

2 Reference

ISO 1988, *Hard coal — Sampling.*

3 Principle

The coal is brought to equilibrium with an atmosphere of 96 % relative humidity at 30 °C and then dried to constant mass at 105 to 110 °C. The conditioning of the coal may be carried out either at atmospheric pressure or under reduced pressure. The moisture-holding capacity is reported as a percentage, by mass, of the conditioned moist coal.

4 Reagent

Potassium sulphate pulp. Add sufficient potassium sulphate to water to form a pulp.

5 Apparatus

5.1 Atmospheric pressure method

5.1.1 *Conditioning vessel* (see Figure 1). A double-walled vessel, manufactured of copper sheet, with a double-walled lid made in two pieces. The lid and vessel are covered by a foam rubber insulation jacket. Each half of the lid is secured by three equally spaced clamps, or by the addition of a 2 kg weight.

5.1.2 *Electric motor,* capable of driving a two-blade propeller at approximately 1 500 rev/min.

5.1.3 *Pump,* for circulating water through the conditioning vessel to maintain it at a temperature of 30 ± 0,1 °C.

5.1.4 *Dishes,* made of glass or corrosion-resistant metal, approximately 50 mm diameter by 10 mm deep, with well-fitting lids.

5.2 Reduced pressure method

5.2.1 *Conditioning vessel* (see Figure 2). A vacuum desiccator weighted to overcome its buoyancy when immersed in water. The desiccator is fitted with a mercury vacuum manometer, and a glass or corrosion-resistant metal stand is provided to carry dishes above the level of the pulp, so that the dishes are protected from spray due to frothing. The volume of free space in the desiccator is kept to a minimum by the choice of a suitable design, by increasing the volume of the pulp material or by adding inert material such as glass beads or washed sand to the pulp.

5.2.2 *Water bath* (see Figure 2), thermostatically controlled at a temperature of 30 ± 0,1 °C.

5.2.3 *Dishes* made of glass or corrosion-resistant metal, approximately 22 mm diameter by 15 mm deep. The lids for these dishes should form a sliding fit.

5.3 For both methods

5.3.1 *Vacuum pump*

5.3.2 *Filter crucible* or *funnel*

5.3.3 *Filter flask*

5.3.4 *Apparatus for determining moisture* by a method which precludes oxidation of the coal.[2)]

5.3.5 *Filter paper,* diameter about 200 mm.

6 Preparation of sample

It is essential that the coal be in a fresh, unchanged state. If the sample cannot be examined immediately, it shall be protected from oxidation by storing under water.

Crush the sample to pass a sieve of 0,2 mm aperture, using the procedures described in ISO 1988. The production of an excessive amount of fines shall be avoided.

7 Procedure

7.1 Preparation of apparatus

7.1.1 *Both methods*

Fill the conditioning vessel with the potassium sulphate pulp to the level indicated in Figure 1 or Figure 2.

7.1.2 *Atmospheric pressure method*

Connect the conditioning vessel to the pump and circulate the water at a rate sufficient to maintain a temperature of 30 ± 0,1 °C in the vessel.

[2)] The apparatus and procedure of Method B described in ISO 589, *Hard coal — Determination of total moisture*, are suitable for this purpose and the description is reproduced in Annex A.

7.1.3 ***Reduced pressure method***

Evacuate the desiccator several times until frothing ceases. To minimize subsequent frothing, the desiccator should be kept evacuated when not in use.

7.2 Preliminary treatment of the sample

Place about 20 g of the sample, crushed to pass a sieve of 0,2 mm aperture, into a conical flask, add about 100 ml of distilled water, shake several times and filter on a filter crucible or funnel. Wash the filtered coal with two or three separate portions of 25 ml of distilled water, taking care that the surface of the coal is not dried by excessive suction. Remove the coal from the filter and spread out between two layers of the filter paper, each layer consisting of eight sheets of the filter paper. The thickness of the coal layer should not be more than 4 mm. Place a weight of about 10 kg on the top layer of filter paper; a considerable part of the adherent surface water is removed by this pressure. After about 10 min, remove the weight and thoroughly mix the coal with a spatula.

7.3 Conditioning

7.3.1 ***Atmospheric pressure method***[3]

Weigh the empty dish and lid and spread uniformly into it approximately 2 g of the treated coal (see **7.2**). Place the dish in the conditioning vessel, controlled to a temperature of 30 ± 0,1 °C, so that it is below an air circulation exit. Replace the two halves of the lid and secure by means of clamps or weights. Start the propeller motor and pass air for a period of 3 to 6 h (see **7.3.2**, note 1). Switch off the motor, remove the dish and securely replace its lid. Transfer the dish on a metal tray to a balance and weigh quickly to within 0,2 mg.

7.3.2 ***Reduced pressure method***

Weigh the empty dish and lid and spread uniformly into it approximately 1 g of the treated coal (see **7.2**). Cover the loaded dish loosely with its lid and place in the conditioning vessel. Replace the lid of the conditioning vessel and evacuate it to a pressure of 2 to 2,5 kPa. Place the vessel into the water bath, maintained at a temperature of 30 ± 0,1 °C, and re-evacuate. The pressure should rise quickly to about 4 kPa, which is the vapour pressure of the saturated solution of potassium sulphate at 30 °C. If the pressure rises above 4 kPa, re-evacuate the vessel without otherwise disturbing it.

After a period of 24 ± 2 h, with the vessel still in the water bath, restore the pressure to atmospheric by slowly admitting dry air at 30 °C through the train, consisting of a capillary tube, a tower charged with dry magnesium perchlorate (see note 2) and a coiled copper tube immersed in the water bath and connected to the inlet of the vessel (see Figure 2). In order to avoid changes in the moisture content it is essential to ensure that there is no disturbance of the local atmosphere immediately adjacent to the conditioned coal. This may be achieved by adjusting the length and diameter of the capillary tube G (see Figure 2), so that the time taken to restore atmospheric pressure is approximately 15 min. Remove the vessel from the bath, remove the lid, transfer the covered dish immediately to a balance and weigh quickly to within 0,2 mg.

NOTE 1 The period required for equilibrium to be reached will depend on the rank of the coal. In the case of an "unknown" coal, several samples of the coal should be placed in the vessel and the moisture-holding capacity determined after various conditioning times.

NOTE 2 Regeneration of magnesium perchlorate should not be attempted, owing to the risk of explosion. When exhausted, the magnesium perchlorate should be washed down the sink with a stream of water.

7.4 Determination of moisture content

Determine the moisture content of the conditioned sample by the method described in Annex A, using the dish in which the coal was conditioned.

8 Expression of results

The moisture-holding capacity, MHC, of the sample as analysed, expressed as a percentage, is given by the formula

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

where

m_1 is the mass, in grams, of empty dish and lid;

m_2 is the mass, in grams, of coal, dish and lid after conditioning;

m_3 is the mass, in grams, of dried coal, dish and lid.

The result, preferably the mean of duplicate determinations (see clause **9**), shall be reported as follows:

Moisture-holding capacity	Report result to the nearest
Less than 5 %	0,1 %
5 % and over	0,5 %

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[3] See national foreword.

9 Precision of the method

Moisture-holding capacity	**Maximum acceptable differences between results**	
	Repeatability	**Reproducibility**
	0,5 % absolute	1,2 % absolute

9.1 Repeatability

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

9.2 Reproducibility

The means of the results of duplicate determinations, carried out in two different laboratories on representative portions taken from the same analysis sample, shall not differ by more than the above value.

10 Test report

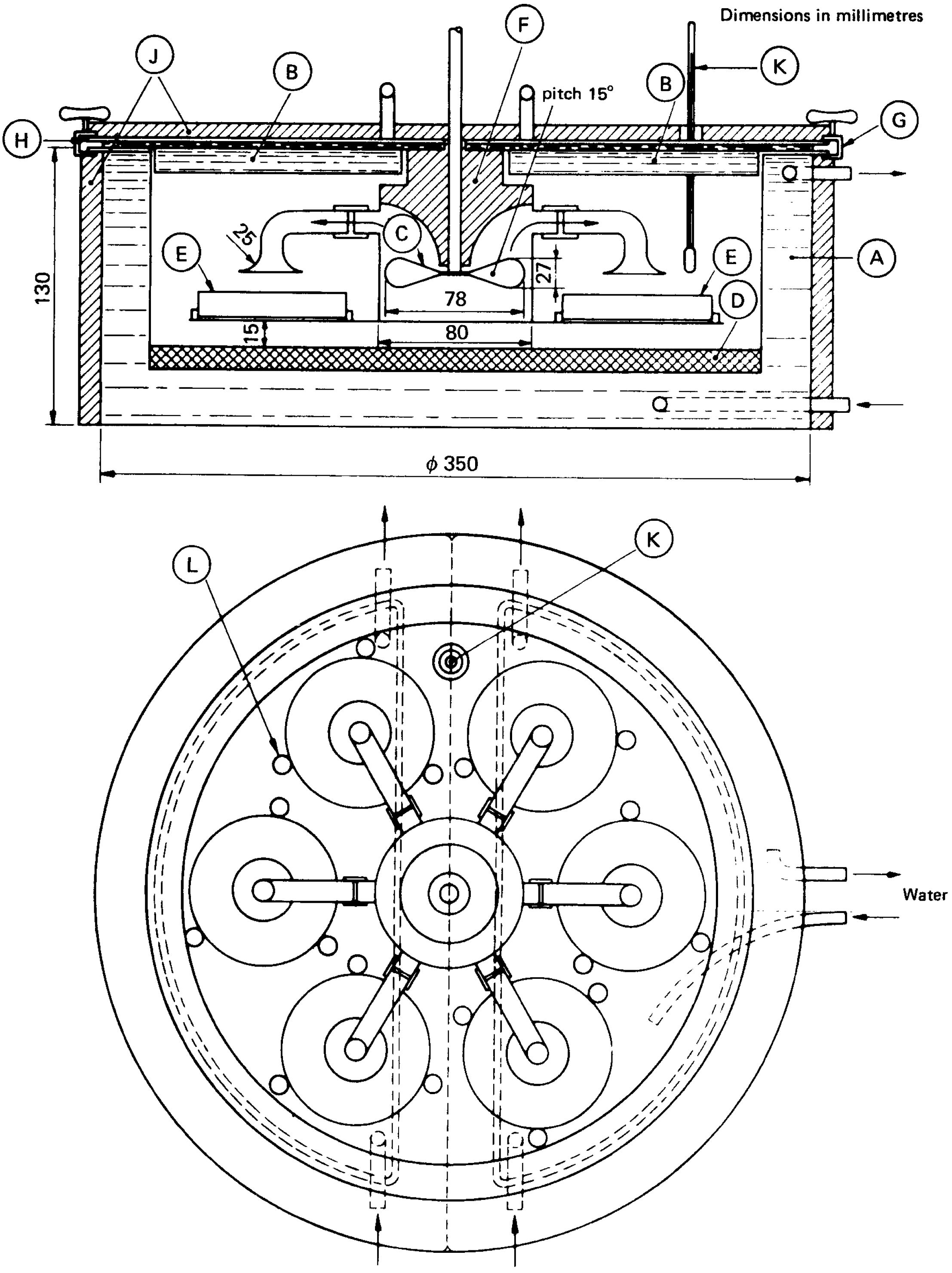
The test report shall include the following particulars:

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

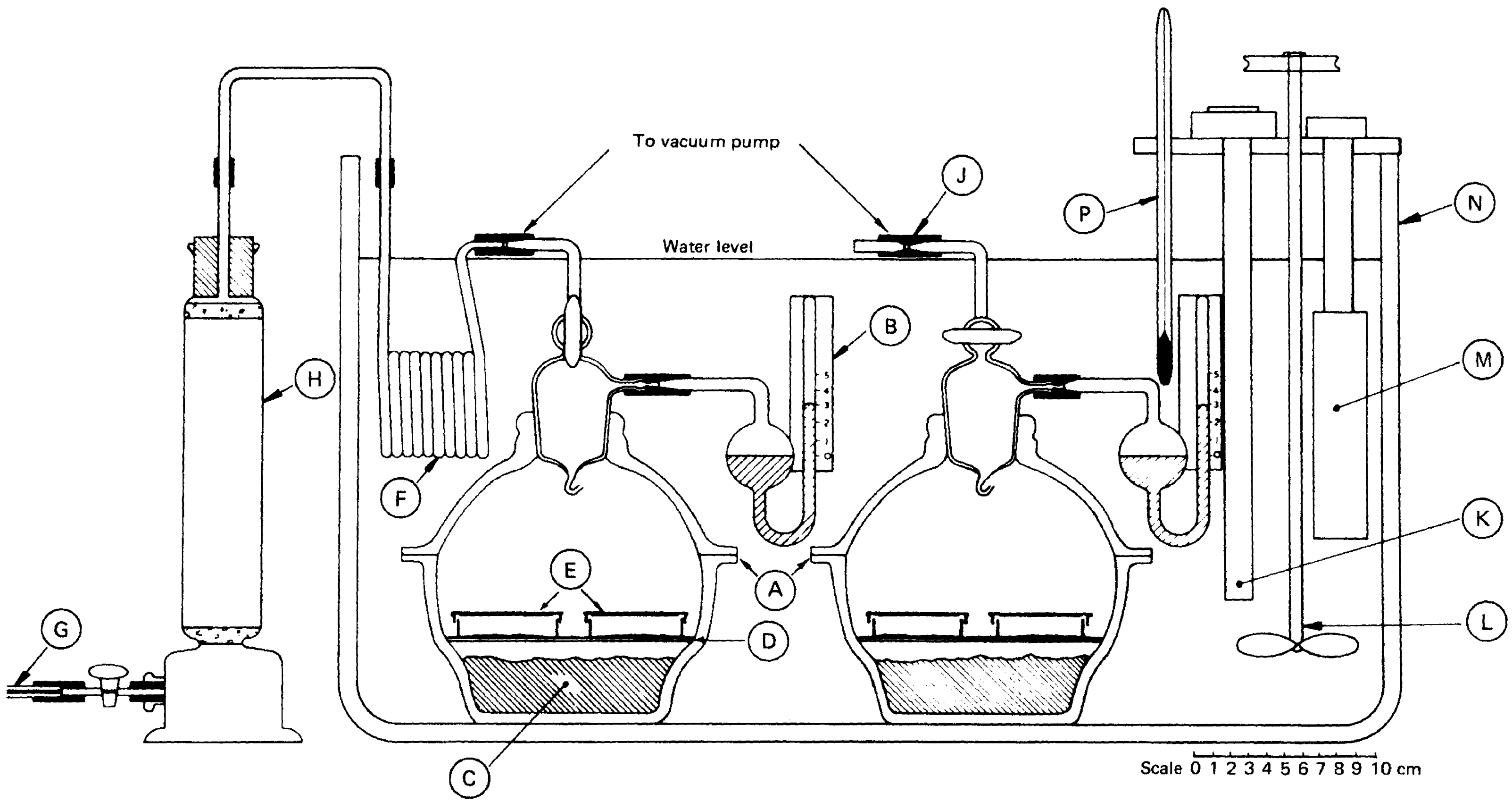
d) any operation not included in this International Standard, or regarded as optional.



Ⓐ Double-walled vessel
Ⓑ Double-walled lid in two pieces
Ⓒ Propeller – pitch 15°
Ⓓ Potassium sulphate pulp
Ⓔ Dishes
Ⓕ Solid brass plug
Ⓖ Screw clamp
Ⓗ Rubber gasket
Ⓙ Foam rubber
Ⓚ Thermometer
Ⓛ Positioning studs

Figure 1 — Apparatus for determination of moisture-holding capacity of coal (atmospheric pressure method)[a]

[a] See national foreword.



Ⓐ Reduced-pressure vessels, suitably weighted inside or outside to overcome buoyancy

Ⓑ Mercury manometer

Ⓒ Pulp of potassium sulphate and water

Ⓓ Glass or corrosion-resistant metal stand to support the samples, also serving as a screen against froth from the potassium sulphate pulp. A second parallel plate over the one actually covering the pulp prevents fouling of the support for the dishes.

Ⓔ Samples of coal contained in suitable glass or corrosion-resistant metal dishes

Ⓕ Spiral of copper tubing

Ⓖ Capillary tubing of suitable length and bore

Ⓗ Tower packed with fresh magnesium perchlorate

Ⓙ Rubber seal

Ⓚ Thermoregulator

Ⓛ Stirrer

Ⓜ Heater

Ⓝ Tank, preferably with glass sides

Ⓟ Thermometer

Figure 2 — Apparatus for determination of moisture-holding capacity of coal (reduced pressure method)

Annex A Determination of moisture in the conditioned coal

A.1 Reagents

A.1.1 *Nitrogen,* dry, and containing less than 30 ppm of oxygen (see Annex B).

A.1.2 *Desiccant:* either fresh or freshly regenerated silica gel or other desiccant, for use in the desiccator.

A.2 Apparatus

A.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 fifteen times per hour.

A.2.2 *Weighing vessels*: see **5.1.4** or **5.2.3**.

A.3 Procedure[4)]

Remove the lid from the dish and place it in the desiccator. Heat the uncovered dish in the oven at a temperature of 105 to 110 °C until constant in mass. Replace the lid, cool the dish rapidly on a cold plate, transfer to a desiccator for not more than 10 min and weigh to within 0,2 mg.

NOTE Heating for 1 h is normally sufficient. Constancy in mass is defined as a change not exceeding 1 mg in a further period of heating of 30 min.

Annex B Nitrogen purification train

B.1 It is essential that the nitrogen used for ventilating the oven should be comparatively pure, since even the small residual amount of oxygen found in commercial nitrogen causes oxidation of heated low-rank coal and hence produces 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, capable of dealing with at least 600 ml per minute at a pressure of a few millibars, consists of a quartz tube 500 mm long and 37 mm internal diameter, containing 1,2 kg of reduced copper in wire form. A convenient source of the gas is a cylinder of compressed nitrogen. 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.

B.2 If it is necessary to reduce any copper oxide formed in the nitrogen purification train, 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. This avoids channelling.

B.3 Finally pass the purified nitrogen through a column of magnesium perchlorate to remove traces of moisture which may be present.

B.4 In the absence of a purification train, a commercial source of nitrogen may be employed, provided that the gas contains less than 30 ppm of oxygen.

[4)] See national foreword.

Publications referred to

See national foreword.

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BSI
389 Chiswick High Road
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W4 4AL