BS 1016-1:
1973

CONFIRMED
SEPTEMBER 1989

Methods for

The analysis and testing of coal and coke —

Part 1: Total moisture of coal

UDC [662.66 + 662.74]:543 + 662.66:543.712.2

Co-operating organizations

The Solid Fuel Industry Standards Committee, under whose supervision this British Standard was prepared, consists of representatives from the following Government departments and scientific and industrial organizations:

- Association of Consulting Engineers
- British Cast Iron Research Association
- British Coke Research Association*
- British Ironfounders' Association
- British Mechanical Engineering Confederation
- British Steel Industry*
- Chamber of Coal Traders
- Chemical Industries Association*
- Coal Utilisation Council
- Coke Oven Managers' Association*
- Combustion Engineering Association*
- Council of Ironfoundry Associations
- Department of the Environment
- Department of Trade and Industry
- Domestic Solid Fuel Appliances Approval Scheme
- Electricity Council, the Central Electricity Generating Board and Area Boards in England and Wales*
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- Institute of British Foundrymen*
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- Women's Advisory Council on Solid Fuel

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- British Coal Exporters' Federation
- British Gas Corporation
- Cement Makers' Federation
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- Institution of Electrical Engineers
- London Coal Exporters' Association
- Scientific Instrument Manufacturers' Association of Great Britain
- Society of Chemical Industry
- Solid Smokeless Fuels Federation
- South Wales Institute of Engineers
- Independent experts

This British Standard, having been approved by the Solid Fuel Industry Standards Committee, was published under the authority of the Executive Board on 15 October 1973

First published, March 1942
First revision, February 1957
Second revision, October 1973

The following BSI references relate to the work on this standard:
Committee reference SFE/45 and SFE/45/8
Draft for comment 72/40657

ISBN 0 580 076873

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, 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.

Amendments issued since publication

Amd. No.	Date of issue	Comments

Contents

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1 Scope

This British Standard describes alternative methods of determining the total moisture on samples of coal taken and prepared by the procedures given in BS 1017-1.

NOTE The titles of the British Standards referred to in this standard are listed on the inside back cover.

2 Definitions

For purposes of this British Standard, the following definitions apply:

2.1
total moisture

the moisture in the coal as sampled

2.2
free moisture

the moisture that is lost by the coal sample in attaining approximate equilibrium with the air to which it is exposed

2.3
moisture in air-dried coal

the moisture in the coal sample after it has attained approximate equilibrium with the air to which it is exposed

2.4
high rank coal

a coal containing (arbitrarily) less than 4 % of moisture (in air-dried coal) and more than 84 % of carbon (dry, ash free basis)

2.5
low rank coal

any coal other than a high rank coal. If the rank of the coal is not known it is treated as a low rank coal

2.6
repeatability

the maximum acceptable difference between single determinations carried out in one laboratory on two separate moisture samples taken simultaneously in accordance with the principles laid down in BS 1017[1)]

2.7
reproducibility

the maximum acceptable difference between single determinations carried out in different laboratories on two separate moisture samples taken simultaneously in accordance with the principles laid down in BS 1017[1)]

3 Coal sample

The sample for the determination of total moisture shall have been taken and prepared according to BS 1017-1. It will be received in the laboratory in a sealed, air-tight container and will consist either of 1 kg of coal having a maximum particle size of 13 mm or of not less than 300 g of air-dried coal having a maximum particle size of 3 mm.

All samples, other than a 1 kg sample prepared from a coal having an initial top size of less than 13 mm or from a visibly dry high rank coal of any top size, will have been air-dried before receipt and details of the loss in mass on air-drying, i.e. the free moisture, will be recorded on the label of the sample container (but see final paragraph of Foreword).

4 Moisture in the 13 mm coal sample

4.1 Distillation with toluene

4.1.1 *Principle.* This method is suitable for all coals. The whole of the 1 kg sample of coal is heated with toluene under reflux conditions. The toluene distils into a graduated receiver, carrying with it the water from the coal. The water then separates to form the lower layer. The excess toluene overflows from the receiver and returns to the flask. The moisture in the coal is calculated from the masses of the sample taken and of the water collected.

4.1.2 *Special reagent.* The following reagent is required:

Toluene. Boiling point 110 °C, dry[2)].

4.1.3 *Special apparatus.* The following apparatus is required:

Dean and Stark apparatus complying with BS 756 and comprising:

1) a copper flask, capacity two litres.
2) a receiver, capacity 100 ml.
3) a condenser and spray tube, as specified in BS 756.
4) heating apparatus: the flask may be heated directly by gas or electricity, or by immersion in a heated oil bath.

The receiver and condenser shall be cleaned before each determination by treating them with a suspension of potassium dichromate in sulphuric acid, or other suitable cleansing agent.

[1)] It should be noted that this definition differs from those in Parts 3 to 16 of BS 1016.

[2)] The residual toluene in the flask after a determination may be filtered and dried for re-use.

4.1.4 *Procedure.* Weigh the sample and container (see Note 1) as received to the nearest gram. Transfer the coal as completely as possible to the flask, carefully clean all traces of the sample from the socket in the neck of the flask and connect to the glass portion of the apparatus. Add 400 ml of the toluene through the top of the reflux condenser. Heat the flask so that brisk distillation occurs and continue to distil until no more water collects in the graduated receiver (see Note 2). This normally takes 6 to 8 h. During distillation wash down any drops of water adhering to the inner surface of the condenser and upper walls of the receiver with toluene, using the spray tube. Record the volume of water collected.

Weigh the wet container with any coal left adhering to it and dry completely at 105 °C to 110 °C. Cool and reweigh. Empty out the residual coal and reweigh the dry empty container (see Note 3).

4.1.5 *Calculation of result.* Calculate the result as follows:

m_1 = Mass of container plus sample as received (g),

m_2 = Mass of water collected (g) (numerically equal to the volume of water collected [ml)],

m_3 = Mass of wet container plus adhering wet coal (g),

m_4 = Mass of dry container plus dry coal (g),

m_5 = Mass of dry empty container (g),

M = Percentage of moisture in the sample analysed,

$$\text{then } M = \frac{m_2 + (m_3 - m_4)}{m_1 - m_5} \times 100$$

4.2 Drying at 105 °C to 110 °C in air

4.2.1 *Principle.* This method shall be used only for high rank coals. The whole of the 1 kg sample of coal is heated to constant mass in an air oven at 105 °C to 110 °C and its moisture calculated from the loss in mass of the sample.

4.2.2 *Special apparatus.* The following apparatus is required:

1) *Air oven.* An oven capable of maintaining a temperature within the range 105 °C to 110 °C and in which the atmosphere changes three to five times per hour. The number of atmosphere changes in the oven shall be determined by one of the methods described in BS 2648.

2) *Tray.* Non-corrodible metal tray approximately 0.1 m^2 in area by 25 mm deep.

4.2.3 *Procedure.* Weigh the sample and container (see Note 1) as received to the nearest gram. Weigh the dry empty tray, transfer the coal as completely as possible to the tray and spread evenly. Place the charged tray in the oven at a temperature of 105 °C to 110 °C. Dry the wet container with any coal adhering to it by warming, transfer the remaining coal to the tray and weigh the dry empty container (see Note 4). Heat the tray and its contents until constant in mass. The time required is normally 5 to 6 h. The loss in mass of the hot sample shall be checked at the end of the fourth hour and at hourly intervals thereafter. Weigh hot to avoid absorption of moisture during cooling.

4.2.4 *Calculation of result.* Calculate the result as follows:

If m_1 = Mass of container plus sample as received (g),

m_2 = Mass of empty tray (g),

m_3 = Mass of tray plus sample after heating (g),

m_4 = Mass of dry empty container (g),

M = Percentage of moisture in the sample analysed,

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

5 Moisture in the 3 mm coal sample

5.1 Distillation with toluene

5.1.1 *Principle.* This method is suitable for all coals. A known mass of coal is heated in a distillation flask with toluene under reflux conditions. The toluene distils into a graduated receiver, carrying with it the water from the coal. The water then separates to form the lower layer. The excess toluene overflows from the receiver and returns to the flask. The moisture in the coal is calculated from the masses of the sample taken and of the water collected.

5.1.2 *Special reagent.* The following reagent is required:

Toluene. Boiling point 110 °C, dry[3].

5.1.3 *Special apparatus.* The following apparatus is required:

Dean and Stark apparatus complying with BS 756 and comprising:

1) A glass flask, capacity 500 ml.

2) A receiver, capacity 10 ml (25 ml for samples expected to contain more than 10 % of moisture).

3) A condenser and spray tube, as in BS 756.

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[3] The residual toluene in the flask after a determination may be filtered and dried for re-use.

4) Heating apparatus: the flask may be heated directly by gas or electricity, or by immersion in a heated oil bath.

The receiver and condenser shall be cleaned before each determination by treating them with a suspension of potassium dichromate in sulphuric acid, or other suitable cleansing agent.

5.1.4 *Procedure.* Before opening the container, thoroughly mix the sample, preferably by mechanical means, for at least 1 min. Weigh to the nearest 0.1 g about 100 g of the sample and transfer to the dry distillation flask. Add 200 ml of the toluene in such a way that any coal adhering to the neck or sides of the distillation flask is washed down by the reagent. Fill the receiver with the toluene 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. During distillation wash down any drops of water adhering to the inner surface of the condenser, or to the upper walls of the receiver, with toluene, 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. Record the volume of water collected.

Calibrate 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 mass of water added (assuming 1 ml has a mass of 1 g) against the scale reading of the water in the receiver and use it to correct the mass of water obtained in each test.

5.1.5 *Calculation of result.* Calculate the result as follows:

If m_1 = mass of coal taken (g),
m_2 = mass of water collected (g),
M = percentage of moisture in the sample analysed,

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

5.2 Drying in nitrogen

5.2.1 *Principle.* This method is suitable for all coals. A known mass of coal is heated to constant mass in a current of nitrogen in an oven maintained at 105 °C to 110 °C and its percentage moisture calculated from the loss in mass of the coal.

5.2.2 *Special reagents.* The following reagents are required:

1) *Nitrogen.* Dry and containing less than 10 p.p.m. of oxygen; this is commercially available in cylinders.

2) *Desiccant.* Either fresh or freshly regenerated, self-indicating activated alumina, silica gel or anhydrous calcium sulphate.

5.2.3 *Special apparatus.* The following apparatus is required:

1) *Oven.* A minimum-free-space oven capable of maintaining a temperature within the range 105 °C to 110 °C and with provision for the nitrogen to pass through it at about 15 oven volumes per hour. A suitable oven is illustrated in Figure 1. Arrangements should be made for measuring the flow of nitrogen through the oven.

2) *Weighing vessels.* Shallow vessels, of glass with ground-on covers, or of corrosion-resistant metal with well-fitting covers, of such a size that the loading of the coal layer does not exceed 0.3 g/cm^2. Circular dishes 65 mm in diameter are suitable for 10 g of coal sample.

5.2.4 *Procedure.* Before opening the container, thoroughly mix the sample, preferably by mechanical means, for at least 1 min. Weigh a clean dry weighing vessel with its cover to the nearest milligram, spread into it about 10 g of the sample in an even layer and replace the cover. Reweigh to determine the mass of sample taken. Place the cover on a metal plate in a desiccator and heat the uncovered vessel in the oven at a temperature of 105 °C to 110 °C in a stream of nitrogen (see Note 5) until constant in mass (1½ to 3 h is normally sufficient). Remove the vessel and dry sample from the oven, replace the cover and set to cool on a metal plate for 10 min. Transfer to the metal plate in the desiccator and weigh after a further 10 min.

5.2.5 *Calculation of result.* Calculate the result as follows:

If m_1 = mass of empty vessel plus cover (g),
m_2 = mass of vessel plus cover plus sample before heating (g),
m_3 = mass of vessel plus cover plus sample after heating (g),
M = percentage of moisture in the sample analysed,

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

6 Calculation and reporting

6.1 Where air-drying was carried out during the preparation of the sample (for exceptions, see Clause **3**), the free moisture will be recorded on the label of the sample container. In such cases:

If M^f = percentage of free moisture (determined as in BS 1017-1),

M = percentage of moisture in the sample as analysed by the procedures in Clauses **4** or **5**,

M_{as} = percentage of total moisture in the coal as sampled,

then

$$M_{as} = M^f + M\left(1 - \frac{M^f}{100}\right)$$

Where air-drying has not been carried out, then $M_{as} = M$.

6.2 The result for the determination of total moisture should be reported to the nearest 0.1 %. Where two determinations are carried out, the mean of the results should be reported.

7 Precision of the determination

In most parts of BS 1016 the definitions and numerical values for precision are based on acceptable differences between duplicate determinations carried out on the analysis sample and therefore relate only to errors involved in the laboratory determination. However, the nature of the determination of total moisture is such that carrying out duplicate determinations would in most cases involve additional errors due to sampling and sample preparation. This arises because only one value for free moisture can be obtained from one total moisture sample. Thus the definitions of repeatability and reproducibility given in Clause **2** are confined to Parts 1 and 2 (see Note 6).

For many commercial purposes, including boiler efficiency testing, it is common practice to take moisture samples in duplicate, either as two separate moisture samples from the consignment, or as two sub-samples taken from the gross sample (see BS 1017-1). Examination of a great deal of experimental data has shown that there is no significant difference between the precision of these two sampling procedures, and the values quoted below cover both methods.

	Repeatability	**Reproducibility**
Total moisture	0.8 % absolute	1.5 % absolute

8 Notes

NOTE 1 Throughout this clause, the description “container” includes the lid or other closure.

NOTE 2 If the capacity of the receiver is insufficient to accommodate all the water distilling from the sample, the condensed water should be withdrawn from the receiver as necessary and measured in a graduated cylinder.

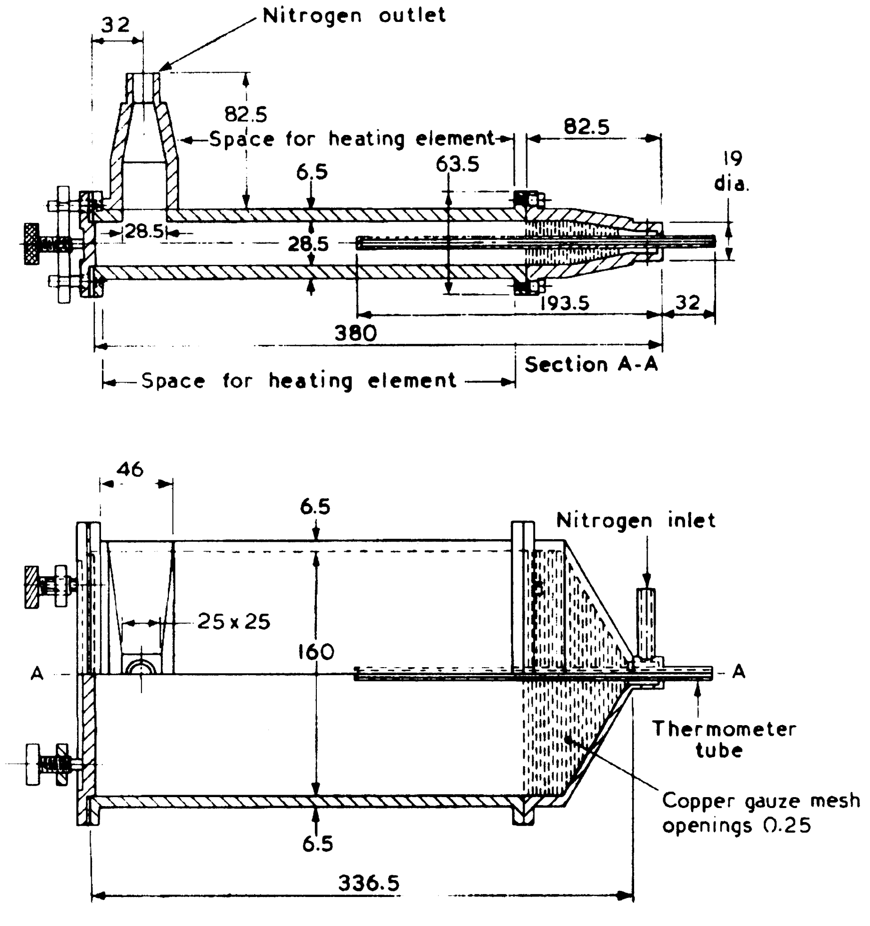
NOTE 3 If the sample can be removed completely leaving a dry, empty container, this drying is omitted and

$$M = \frac{m_2}{m_1 - m_5} \times 100$$

NOTE 4 If the sample can be removed completely leaving a dry, empty container, this drying is omitted.

NOTE 5 For high rank coals only, it is permissible to dry in air in an oven meeting the specification given in **4.2**.

NOTE 6 It is, however, possible to carry out duplicate determinations of moisture on the air-dried sample of 3 mm coal (see Clause **5**). The difference between the results of such duplicates should not exceed 0.2 %.



Dimensions in millimetres.

Figure 1 — Suitable nitrogen oven

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Publications referred to

This standard makes reference to the following British Standards

BS 756, *Dean and Stark apparatus.*

BS 1017, *The sampling of coal and coke.*

BS 1017-1, *Sampling of coal.*

BS 2648, *Performance requirements for electrically-heated laboratory drying ovens.*

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