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Methods for

The analysis and testing of coal and coke

**Part 4. Moisture, volatile matter
and ash in the analysis sample
of coke**

Gr 6

British Standards Institution



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The following BSI references relate to the work on this standard:

Committee reference SFE/45 and SFE/45/8. Draft for comment 72/40881

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- | | |
|--|---|
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- | | |
|---|---|
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Foreword

This publication is Part 4 of a series of sixteen Parts issued under the same number, BS 1016, dealing with methods for the analysis and testing of coal and coke. The methods given in BS 1016 are specified for the analysis of coal or of coke or, in some instances, for the analysis of both coal and coke. Experience has shown that, in general, the methods may also be used for the analysis of manufactured smokeless fuels other than coke made by a conventional process. However, as no systematic survey has been made of their applicability to all such fuels, it is not possible to state whether the methods for coal or those for coke will be more appropriate for a particular fuel; usually the former are likely to be satisfactory for fuels prepared at temperatures below 800 °C.

This revision has been undertaken to introduce metric units throughout and to adopt a uniform presentation. No mandatory technical changes have been made from the 1965 edition. This revision deals with the determination of moisture, volatile matter and ash in the analysis sample of coke. The methods are such that it is sometimes convenient to analyse a number of different samples simultaneously. Where multiple determinations are possible, any additional apparatus required has been specified. However, for simplicity, all procedures have been written for a single sample. When a system of multiple determinations is adopted, duplicate tests on the same sample are carried out at separate times or in separate apparatus.

Two methods are specified for the determination of moisture, namely, the direct gravimetric method and drying in nitrogen in a minimum-free-space oven. The latter method is essentially the same as that given in ISO/R 687, 'Determination of moisture in the analysis sample of coke', differing only in that the sample is dried in nitrogen at 200 °C in this standard, and in air at 200 °C or in nitrogen at 320 °C in ISO/R 687.

The methods for the determinations of volatile matter and of ash are technically equivalent to those specified respectively in ISO/R 562, 'Determination of the volatile matter of hard coal and of coke', and ISO/R 1171, 'Determination of ash of solid mineral fuels'.

Notes on procedure and apparatus are given in Clause 9.

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British Standard Methods for

The Analysis and testing of coal and coke

Part 4 Moisture, volatile matter and ash in the analysis sample of coke

1. Scope

This British Standard describes the determination of moisture, volatile matter and ash in the analysis sample of coke.

2. Definitions

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

- (1) *Coke*. The solid residue of the distillation of coal at high temperature (above 800 °C).
- (2) *Moisture in the analysis sample*. The moisture in the coke sample as analysed.
- (3) *Volatile matter*. The loss in mass, less that due to moisture, that occurs when the coke is heated out of contact with air under standardized conditions.
- (4) *Ash*. The inorganic matter remaining after the coke has been incinerated to constant mass under standardized conditions.
- (5) *Repeatability**. The maximum acceptable difference between duplicate determinations carried out at different times in the same laboratory on the same analysis sample by the same operator using the same apparatus.
- (6) *Reproducibility**. The maximum acceptable difference between the mean of duplicate determinations carried out in one laboratory and the mean of duplicate determinations carried out in any other laboratory on representative samples taken from the same bulk sample after the last stage of the reduction process.

3. Coke sample

The coke used for the analysis is the analysis sample of 50 g of dry coke having a top size not greater than 0.2 mm (ground to pass a 212 µm test sieve to BS 410), taken and prepared according to BS 1017 : Part 2.

The sample shall be thoroughly mixed, preferably by mechanical means† immediately before analysis. Portions for duplicate determinations shall be weighed out consecutively, although the determinations shall not be made in the same apparatus at the same time.

4. Moisture in the analysis sample

4.1 General. The moisture in the analysis sample, while having little significance in itself, is required for calculating other results to any desired basis. Moisture determinations shall therefore be made at the same time as those for volatile matter and ash.

Either of two methods may be used for the determination:

- (1) Direct gravimetric determination (see 4.2).
- (2) Drying in nitrogen (see 4.3).

Although the two methods have been found to give substantially the same results, the indirect method (see 4.3) may occasionally give slightly lower values.

* See BS 1016 : Part 16

† A mixer of a type similar to that described in BS 1017 : Part 2 is suitable.

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NOTE. Although in the past, it has been assumed that all moisture was released from coke by heating for 1½ hours at 105 °C to 110 °C in air, recent work (H. C. Wilkinson, *Fuel* (London) 1965, 44, 191) has shown that complete removal of moisture from the analysis sample of coke is not achieved within a reasonable time unless much higher temperatures are used. Otherwise, prolonged periods of heating at moderate temperatures are necessary. It is therefore considered that the determination of moisture in coke by heating at 105 °C to 110 °C will give erroneous results; the determination of moisture in this temperature range is therefore omitted from the standard and is permissible only for routine control purposes.

4.2 Direct gravimetric determination

4.2.1 Principle. A known mass of the sample is heated in a current of nitrogen at 320 °C and the water driven off is collected in an absorption tube. The moisture in the coke is calculated from the increase in mass of the absorption tube.

4.2.2 Special reagents. The following reagents are required:

- (1) *Nitrogen*, containing less than 10 p.p.m. of oxygen.
- (2) *Magnesium perchlorate**, 0.7 mm to 1.2 mm.

4.2.3 Special apparatus. The apparatus required is shown diagrammatically in Fig. 1. It consists of the following components:

(1) *Oven*. An oven, preferably of the block type, capable of accommodating a glass retort tube and of maintaining a temperature within the range 300 °C to 340 °C in the tube. The oven may be designed to accommodate a number of these tubes.

(2) *Retort tube*. A glass retort tube, 275 mm long and 15 mm internal diameter, with a depression 170 mm from the sealed end, fitted with a flexible, heat resistant stopper through which pass two glass tubes. One of these, the inlet tube, is connected to the flowmeter and terminates just inside the stopper. The other, the outlet tube, is connected to the absorption tube and passes down the retort tube almost to the sealed end (see Fig. 2). The connection between the flowmeter and the inlet tube is made with polyvinyl chloride tubing. The connection between the outlet tube and the absorption tube is made with a short length of rubber tubing.

(3) *Drying tower*. A drying tower, 250 ml capacity, packed with magnesium perchlorate for drying the nitrogen.

(4) *Flowmeter*. A flowmeter capable of measuring a flow rate of 100 ml per minute. If the flowmeter contains a liquid this shall be a non-volatile oil.

(5) *Absorption tube*. An absorption tube which shall be either a U-tube, fitted with glass stoppers and filled with magnesium perchlorate retained by plugs of glass wool, or a Riley tube, the centre column of which is filled with magnesium perchlorate retained by plugs of glass wool.

If multiple determinations are made, the following will also be required:

- (6) *Manifold*. A manifold made so that the branches are in line with the corresponding flowmeters.
- (7) *Control valves*. A needle valve or screw clip, placed between each flowmeter and the manifold, for controlling the nitrogen flow rate.

4.2.4 Procedure. Adjust the rate of flow of the nitrogen passing through the flowmeter to 100 ml/min. Connect an empty retort tube to a closed absorption tube and to the nitrogen stream from the flowmeter. Check for leaks in the system. Open the tap(s) of the absorption tube and readjust the flow rate to 100 ml/min.

Insert the retort tube into the oven heated to 320 °C. After 15 min (see Note 1) close the tap(s) of the absorption tube, disconnect, wipe with a clean dry cloth free from loose fibres and allow the absorption tube to stand for 20 min in the balance room. Open the tap(s) of the absorption tube momentarily to equalize the pressure and then weigh to the nearest 0.1 mg. While the absorption tube is standing prior to weighing, remove the retort tube from the oven and allow to cool with a stream of dry nitrogen passing through it. When the retort tube is cool remove it, with the stopper and tubes still in it, to the balance room.

Weigh about 1 g of the coke sample to the nearest 0.1 mg on a scoop and transfer the coke to the retort tube, removing the stopper and tubes just prior to the introduction of the sample. Spread the coke in an even layer between the sealed end and the depression in the retort tube. Replace the stopper and tubes, reassemble the apparatus and check for leaks as before (see Note 2). Resume the passage of nitrogen at 100 ml/min.

Insert the charged retort tube into the oven. After 60 min close the tap(s) of the absorption tube, disconnect, condition and weigh the absorption tube as described above.

Carry out a blank determination using the same procedure as for the moisture determination but omitting the coke sample (see Note 3). The increase in mass of the absorption tube should not be more than 1 mg.

*This is the quality of reagent referred to as 'magnesium perchlorate (dried)', sometimes known as 'Anhydron'.