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Methods for

The analysis and testing of coal and coke —

Part 14: Analysis of coal ash and coke
ash

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British Ceramic Research Association	London Coal Exporters' Association
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Foreword

This standard makes reference to the following British Standards:

BS 410, *Test sieves*.

BS 1016, *Methods for the analysis and testing of coal and coke — Part 3: Proximate analysis of coal — Part 4: Proximate analysis of coke*.

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

BS 1041, *Code for temperature measurement*.

In 1942, when BS 1016, "*Methods for the analysis and testing of coal and coke*", was published it superseded a number of other standards, each dealing with special methods, bringing them up to date and embodying them in one standard. BS 1016:1942 also dealt with the receipt of samples and their preparation in the laboratory for analysis and was linked directly with a companion standard on methods of collection of samples, BS 1017, "*The sampling of coal and coke*".

Continued work by the Committee concerned and international discussion under the aegis of the International Organization for Standardization have led to the revision of these collected methods and a new edition of BS 1016 has become necessary. A similar revision of BS 1017 has been undertaken and the link between the two standards is maintained. The division between them, however, has been changed and this standard deals only with the analysis and testing of coal and coke; all the preparation of samples for analysis is included in the revised BS 1017.

The new standard is published in separate parts, each covering a particular group of analyses, e.g. proximate analysis, ultimate analysis. This method of presentation permits the revision of any part with the minimum of delay without the necessity of issuing a new comprehensive standard. Where the methods for a particular group of analyses for coke differ widely from those for coal they are published as a separate part but where the modifications for coke are slight they are given as separate sections of the same part.

In the interests of international discussion it is important that there should be uniformity in definition and in method of reporting of results. Each part of the new standard begins, therefore, with a list of definitions relevant to the methods described in it and ends with a section dealing with reporting of the results.

Part 14 — Analysis of coal ash and coke ash — deals with the determination of the major constituents common to all ashes. In March, 1960, BSI Document PD 3714 gave a brief review of the considerable progress which has been made in the development of more rapid techniques. The committee responsible for this revision of BS 1016 has now recommended the adoption of the B.C.U.R.A. rapid method¹⁾ which, as well as being accurate, enables several ashes to be analysed in the time taken for one by the classical method. The saving of time has been made by eliminating lengthy chemical separations and substituting selective complexometric and physico-chemical methods which utilize the relatively inexpensive spectrophotometers and flame photometers now available commercially.

A growing need for even more rapid methods of ash analysis has led to the development of a spectrochemical method, based on the use of the Hilger large quartz spectrograph, for the determination of some of the major constituents. Its precision is lower than that of the B.C.U.R.A. rapid method and so far, because of the high initial cost of the equipment, its use has been limited to a few laboratories. However, the possibility of future developments requiring large numbers of ash analyses warrants a description of the method in an Appendix.

¹⁾ K. Archer, D. Flint and J. Jordan, *Fuel*, Lond., 1958, **37**, 421.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i to iv, pages 1 to 18, 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.

14.1 Definitions

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

14.1.1

ash (coal²⁾ and coke³⁾)

the inorganic matter remaining after the coal or coke has been incinerated to constant weight under standard conditions

14.1.2

within-laboratory tolerance

the maximum acceptable difference between determinations made in the same laboratory on the same preparation of ash by the same operator using the same apparatus

14.1.3

between-laboratory tolerance

the maximum acceptable difference between the means of two determinations carried out by two different laboratories on representative samples taken from the same preparation of ash

14.1.4

temperature measurement

where the temperature is specified as a definite figure, it is to be understood that this figure should be attained as closely as possible and that it should be subject only to the errors inherent in accurate temperature measurement, as defined in BS 1041⁴⁾

where a range of temperature is given, the temperature may be anywhere in the range without detriment to the result. However, the mean of the range should be aimed at in order that errors of measurement do not cause a temperature outside the specified range to be used inadvertently

Table 1 — Characteristic composition of British coal and coke ashes

Constituent	Expressed as	Range per cent
Silica	SiO ₂	15–55
Alumina	Al ₂ O ₃	10–40
Ferric oxide	Fe ₂ O ₃	1–40
Calcium oxide	CaO	1–25
Magnesium oxide	MgO	0.5–5
Sodium oxide	Na ₂ O	0–8
Potassium oxide	K ₂ O	0–5
Titanium oxide	TiO ₂	0–3
Manganese oxide	Mn ₃ O ₄	0–1
Sulphate	SO ₃	0–12
Phosphate	P ₂ O ₅	0–3

²⁾ BS 1016-3, "Proximate analysis of coal".

³⁾ BS 1016-4, "Proximate analysis of coke".

⁴⁾ BS 1041, "Code for temperature measurement".

⁵⁾ BS 1016-3, "Proximate analysis of coal".

⁶⁾ BS 1016-4, "Proximate analysis of coke".

14.2 Introduction

One of the important factors governing the industrial usage of coal and coke is the behaviour of the mineral matter associated with it. The plant may be designed for this to be removed as particulate ash, fly ash, clinker or liquid slag and operational difficulties may be encountered if any other than the intended form of residue is produced. The analysis of ash prepared under standard condition⁵⁾ ⁶⁾ is often used as a guide to the selection of suitable solid, fuel.

Ash consists mainly of the oxides of silicon, aluminium, iron, calcium, magnesium, titanium, manganese and the alkali metals, partly combined as silicates, sulphates and phosphates; its composition reflects the constitution of the original mineral matter. These constituents normally account for at least 99 per cent of the ash. The usual limits within which they are encountered are shown in Table 1, although the compositions of some ashes can be considerably outside these limits.

On exposure to air, some ashes rapidly absorb water and carbon dioxide. It is, therefore, necessary either to re-ignite each portion of ash immediately before weighing or to determine the loss on ignition using a separate portion. The results are reported on the "Ignited at 800 °C" basis.

Although the procedure is written for a single analysis of one ash, full advantage of the saving of time afforded by the method can best be taken by conducting several analyses simultaneously. The use of calibration graphs for these colorimetric methods leads to results which are less reproducible than those obtained by the specified procedure and is therefore not recommended.

14.3 Principle

The scheme of analysis is shown diagrammatically in Figure 1. Two solutions are prepared from the ash. Solution A is prepared by fusing the ash with sodium hydroxide, leaching the melt with water and acidifying with hydrochloric acid. It is used for the colorimetric determination of silica and alumina.

Solution B is prepared by decomposition of the ash with hydrofluoric, nitric and sulphuric acids. After removal of the silica by evaporation, the residue is dissolved in water. This solution is used for the flame photometric determination of sodium and potassium, the colorimetric determination of iron, titanium, manganese and phosphorus, and the determination of calcium and magnesium by titration with EDTA.

Separate portions of the sample are used for the gravimetric determination of sulphate and the determination of loss on ignition at 800 °C.

The spectrophotometer or flame photometer is calibrated for every determination using standard solutions prepared from salts of adequate purity or, in the case of silica and alumina, from either N.B.S. standard feldspar No. 99 or B.C.S. firebrick No. 269.

Unless otherwise stated, the coloured solutions prepared for optical density measurements are stable for several hours.

Should an abnormally low total be obtained, the oxides of other elements, e.g. arsenic, boron, barium, germanium and tin, may be present and, if desired, may be identified and determined by appropriate chemical or spectrographic methods. Substantial percentages of such oxides may influence the results of other analytical determinations.

14.4 Special apparatus

Muffle furnace. A muffle furnace⁷⁾ capable of giving a zone within the range 490–510 °C in 30 minutes from cold, of being raised to 790–810 °C in a further 60 to 90 minutes and of maintaining this latter temperature at the end of the run-up period. The ventilation shall be such as to give at least four air changes per minute at 800 °C⁸⁾.

Silica trays, 15 cm × 8 cm × 1 cm.

Silica plate, a silica plate 6 mm thick, which is an easy sliding fit into the muffle furnace.

Platinum crucibles, each of capacity 30 ml, with lid.

Cooling block, of aluminium.

Nickel crucibles, each of capacity 40 ml, with lid.

Spectrophotometer, of adequate sensitivity and selectivity⁹⁾.

Flame photometer, of adequate sensitivity and selectivity¹⁰⁾.

14.5 Coal or coke sample

The coal or coke used for the preparation of the sample of ash shall be prepared in a similar manner to the ½ lb analysis sample of coal or the 2 oz analysis sample of coke, taken according to BS 1017¹¹⁾, ground to pass a 72 mesh BS test sieve¹²⁾ and thoroughly mixed, preferably by mechanical means. The weight of coal or coke necessary will vary with its ash content, but sufficient is required to provide at least 5 g of ash.

14.6 Preparation of ash

Using the silica trays, prepare 5 g of ash in the manner described for the determination of ash in coal (Part 3¹³⁾) or coke (Part 4¹⁴⁾). The loading of the trays shall not exceed 0.14 g/cm² for coal or 0.07 g/cm² for coke.

Grind the ash in an agate mortar so that it all passes a 240 mesh BS test sieve. Re-ignite the ash at 800 °C for one hour in the muffle furnace. Allow to cool, store in an airtight glass bottle of 1 oz capacity and mix thoroughly.

14.7 Loss on ignition

PRINCIPLE

One of two procedures may be adopted to minimize errors due to the absorption of water and carbon dioxide by the ash. Either the sample may be ignited at 800 °C to constant weight and cooled rapidly immediately before the portions for the preparation of the solutions are weighed out or the loss on ignition may be determined by the following procedure, using a separate portion of the sample weighed out at the same time as those for the rest of the analyses.

⁷⁾ The high temperature muffle furnace described by C. W. G. Ockelford (Fuel, 1945, 24, 151) is suitable.

⁸⁾ The air changes per minute can be obtained by measurement of the air flow in the flue by means of a pitot-static tube and sensitive manometer [E. Owen, Measurement of Air-flow, 3rd Ed. Revised, p. 88 (Chapman & Hall, Lond.)].

⁹⁾ A Unicam S.P.600 or similar instrument is suitable.

¹⁰⁾ An "Eel" flame photometer or similar instrument is suitable.

¹¹⁾ BS 1017, "The sampling of coal and coke".

¹²⁾ BS 410, "Test sieves".

¹³⁾ BS 1016-3, "Proximate analysis of coal".

¹⁴⁾ BS 1016-4, "Proximate analysis of coke".