

Methods for

Analysis and testing of coal and coke —

**Part 100: General introduction and
methods for reporting results**

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Committees responsible for this British Standard

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Foreword

This Part of BS 1016 has been prepared under the direction of the Solid Mineral Fuels Standards Policy Committee. It provides an introduction to the new series of Parts of BS 1016 (from Part 101 onwards) and it is also a revision of BS 1016-16:1981, which is withdrawn. The section on reporting of results is technically equivalent to BS 1016-16:1981.

Under a scheme for rationalizing and restructuring BS 1016, the Parts numbered from 1 to 21 are gradually being withdrawn and replaced by Parts in the new series, as given in Table 1 (see clause 4). Those Parts in the earlier series which are still current are as follows, with the new Part numbers (which will be given to revisions when they are published) in parentheses.

- *Part 1: Total moisture of coal (Part 101);*
- *Part 2: Total moisture of coke (Part 102);*
- *Part 6: Ultimate analysis of coal (Part 106);*
- *Part 7: Ultimate analysis of coke (Part 106);*
- *Part 8: Chlorine in coal and coke (Part 106);*
- *Part 9: Phosphorus in coal and coke (Part 106);*
- *Part 10: Arsenic in coal and coke (Part 106);*
- *Part 11: Forms of sulphur in coal (Part 106);*
- *Part 14: Analysis of coal ash and coke ash (Part 114);*
- *Part 15: Fusibility of coal ash and coke ash (Part 113);*
- *Part 17: Size analysis of coal (Part 109);*
- *Part 18: Size analysis of coke (Part 110);*
- *Part 20: Determination of Hardgrove grindability index of hard coal (Part 112);*
- *Part 21: Determination of moisture-holding capacity of hard coal (Part 103).*

This Part of BS 1016 is related to ISO 1170:1977, published by the International Organization for Standardization (ISO).

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 12, 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.

1 Scope

This Part of BS 1016 gives a general introduction to BS 1016. It also provides formulae for the conversion of analytical data for coal and coke to the various different bases in common use, procedures for the interpretation of analytical results and other background information.

A list of the symbols used in this Part of BS 1016 is given in clause 3.

NOTE The symbol used for a particular quantity in this Part of BS 1016 is not necessarily the same as that used for the same quantity in another Part.

2 References

2.1 Normative references

This Part of BS 1016 incorporates, by reference, provisions from specific editions of other publications. These normative references are cited at the appropriate points in the text and the publications are listed on the inside back cover. Subsequent amendments to, or revisions of, any of these publications apply to this Part of BS 1016 only when incorporated in it by updating or revision.

2.2 Informative references

This Part of BS 1016 refers to other publications that provide information or guidance. Editions of these publications current at the time of issue of this standard are listed on the inside back cover, but reference should be made to the latest editions.

3 Definitions and symbols

3.1 Definitions

For the purposes of this Part of BS 1016, the definitions given in BS 3323:1992 apply.

3.2 Symbols

The following symbols are used to denote the specified quantities.

Symbol	Quantity
A	ash, expressed as a percentage by mass
A_{sh}	shale ash, expressed as a percentage by mass
C	total carbon content, expressed as a percentage by mass
C_{o}	organic carbon content, expressed as a percentage by mass
$[Cl]$	chlorine content, expressed as a percentage by mass
$[Cl]_{\text{o}}$	organic chlorine content, expressed as a percentage by mass
$[CO_2]$	carbon dioxide (from carbonates) content, expressed as a percentage by mass

Symbol	Quantity
$[Fe]$	iron content, expressed as a percentage by mass
H	total hydrogen (excluding hydrogen from moisture) content, expressed as a percentage by mass
$\{H_2\}$	relative molecular mass of hydrogen
$\{H_2O\}$	relative molecular mass of water
M	moisture content, expressed as a percentage by mass
MM	mineral matter content, expressed as a percentage by mass
n	number of test results
N	nitrogen content, expressed as a percentage by mass
O	total oxygen (excluding oxygen from moisture) content, expressed as a percentage by mass
O_{min}	oxygen content of mineral matter
O_{o}	organic oxygen content, expressed as a percentage by mass
$Q_{\text{gr,p}}$	gross calorific value at constant pressure
$Q_{\text{gr,v}}$	gross calorific value at constant volume
$Q_{\text{net,p}}$	net calorific value at constant pressure
$Q_{\text{net,v}}$	net calorific value at constant volume
r	repeatability limit
s	repeatability standard deviation
s'	reproducibility standard deviation
S	total sulfur content, expressed as a percentage by mass
S_{a}	sulfur content of ash, expressed as a percentage by mass
S_{o}	organic sulfur content, expressed as a percentage by mass
S_{p}	pyritic sulfur content, expressed as a percentage by mass
S_{s}	sulfate sulfur content, expressed as a percentage by mass
$[SO_3]$	sulfur trioxide content of ash, expressed as a percentage by mass
t	value of Student's t at 95 % confidence level
V	volatile matter content, expressed as a percentage by mass
W	total moisture (including water of constitution combined with the mineral matter) content, expressed as a percentage by mass
Y	correction factor for the heat liberated by pyrites burning to iron (III) oxide during the determination of calorific value

Table 1 — Structure of BS 1016

Part/ Section/ Subsection	Title	Replaces Part	Related ISO standards
100	General introduction and methods for reporting results	16	1170
101 ^a	Determination of total moisture content of coal	1	589
102 ^a	Determination of total moisture content of coke	2	579
103 ^a	Determination of moisture-holding capacity of hard coal	21	1018
104	Proximate analysis of coal and coke	3,4	—
104.1	Determination of moisture content of the general analysis sample of coal	—	331
104.2	Determination of moisture content of the general analysis sample of coke	—	687
104.3	Determination of volatile matter content	—	562
104.4	Determination of ash	—	1171
105	Determination of gross calorific value	5	1928
106	Ultimate analysis of coal and coke	—	—
106.1	Determination of carbon and hydrogen content	6,7	—
106.1.1 ^a	High temperature combustion method	—	609
106.1.2 ^a	Liebig method	—	625
106.2 ^a	Determination of nitrogen content	6,7	333
106.3 ^a	Determination of oxygen content	—	1994
106.4	Determination of total sulfur content	6,7	—
106.4.1	Eschka method	—	334
106.4.2 ^a	High temperature combustion method	—	351
106.5 ^a	Determination of forms of sulfur	11	157
106.6	Determination of chlorine content	8	—
106.6.1 ^a	Eschka method	—	587
106.6.2 ^a	High temperature combustion method	—	352
106.7 ^a	Determination of carbon dioxide content of coal	6	925
106.8 ^a	Determination of phosphorus content	9	622
106.9 ^a	Determination of arsenic content	10	601
107	Caking and swelling properties of coal	12	—
107.1	Determination of crucible swelling number	—	501
107.2	Assessment of caking power by Gray-King coke test	—	502
107.3	Determination of swelling properties using a dilatometer	—	8264
108	Tests special to coke	13	—
108.1	Determination of shatter indices	—	616
108.2	Determination of Micum and Irsid indices	—	556
108.3	Determination of bulk density (small container)	—	567
108.4	Determination of bulk density (large container)	—	1013
108.5	Determination of density and porosity	—	1014
108.6	Determination of critical air blast value	—	—
109 ^a	Size analysis of coal	17	1953
110 ^a	Size analysis of coke	18	728, 2325
111	Determination of abrasion index of coal	19	—
112 ^a	Determination of Hardgrove grindability index of hard coal	20	5074
113 ^a	Determination of fusibility of coal ash and coke ash	15	540
114 ^a	Analysis of coal ash and coke ash	14	—

^a In preparation.

The following subscripts, or partial subscripts, are used to denote the basis to which results are reported, as indicated.

Subscript	meaning
ad	air-dried basis
as	as sampled basis
d	dry basis
daf	dry, ash-free basis
dmmf	dry, mineral-matter-free basis

4 Structure of BS 1016

The structure of BS 1016 is given in Table 1, which shows the new Parts and the corresponding Parts in the old series which have been, or will be, replaced by them.

5 Basis of reporting results

5.1 General

The analyses described in BS 1016-6 to BS 1016-11 (BS 1016-106 in the new series), BS 1016-21 (BS 1016-103 in the new series), BS 1016-104, BS 1016-105 and BS 1016-106.4.1 are carried out on samples of coal or coke that have been ground to pass a 212 μm test sieve conforming to BS 410:1986 and air-dried to approximate equilibrium with the laboratory atmosphere.

For certain purposes it is necessary to report results on some other basis e.g. as follows:

- as received
- dry
- dry, ash-free
- dry, mineral-matter-free

The methods of converting results to these other bases are described in 5.2 to 5.5.

The analysis of the pure coal substance, which is of particular importance for the classification of coals, is that expressed on the dry, mineral-matter-free basis and so the estimation of mineral matter is discussed in 5.5.

The calorific value of a fuel determined in the laboratory is the gross calorific value at constant volume (see Part 105). Methods for calculating the gross calorific value at constant pressure, as well as the net calorific values at constant volume and at constant pressure are described in 5.6.

The reporting of results for the methods described in BS 1016-14, BS 1016-15, BS 1016-17, BS 1016-18 and BS 1016-20 (BS 1016-114, BS 1016-113, BS 1016-109, BS 1016-110 and BS 1016-112 respectively in the new series) and BS 1016-107, BS 1016-108 and BS 1016-111 is covered comprehensively in the respective Parts.

5.2 Calculation to other bases

Almost all analytical values can be converted from any given basis to another basis by multiplying them by the appropriate formula shown in Table 2 after insertion of the numerical values for the symbols.

The formulae in Table 2 are exact and simple to use, but for some conversions to the dry, ash-free and the dry, mineral-matter-free bases, the corrections indicated in 5.4 and 5.5 may be necessary.

NOTE Carbon dioxide from carbonates, total sulfur content, pyritic sulfur content, sulfate sulfur content, phosphorus content and arsenic content are not reported on the dry, ash-free or the dry, mineral-matter-free bases as the terms are not applicable to these determinations.

5.3 Dry basis

When coal analyses are converted to this basis, the effect of a factor that is partly dependent on the laboratory atmosphere is removed.

For coke analyses, the analysis sample is usually prepared from the dried sample of coke passing a 13.2 mm test sieve after the determination of total moisture content in accordance with BS 1016-2. Finely ground coke is extremely hygroscopic and can absorb up to 2 % (m/m) of moisture. Therefore, if a significant period of time elapses between preparation of the sample and its analysis, its moisture content should be determined and the results of the analysis corrected to the dry basis using the appropriate formula. However, if the analysis is carried out within a short time of the sample being prepared, only a small amount of moisture will be picked up, and the moisture content on the air-dried basis (M_{ad}) will be small and the conversion factor will be close to unity.

5.4 Dry, ash-free basis

The dry, ash-free basis is a hypothetical condition in which the coal or coke is assumed to be free of both moisture and ash.

Carbon content, hydrogen content and calorific value can be converted to the dry, ash-free basis by the formula given in Table 2. The formula may also be used for the volatile matter content of most coals. For anthracites and coals of low volatile matter content that also have a high carbonate content [greater than 2 % (*m/m*)], it is necessary to make allowance for the carbon dioxide evolved during the determination of the volatile matter content and the following equation is used.

$$V_{\text{daf}} = (V_{\text{ad}} - [\text{CO}_2]_{\text{ad}}) \times \frac{100}{100 - (M_{\text{ad}} + A_{\text{ad}})} \quad (1)$$

When reporting organic carbon content on this basis, it is necessary that the carbon present as carbonate is deducted from the total carbon content and the calculation for this is given by the following equation.

$$C_{\text{o,daf}} = (C_{\text{ad}} - 0.273[\text{CO}_2]_{\text{ad}}) \times \frac{100}{100 - (M_{\text{ad}} + A_{\text{ad}})} \quad (2)$$

5.5 Dry, mineral-matter-free basis

5.5.1 General

The dry, mineral-matter-free basis is a hypothetical condition, corresponding to the concept of pure coal or coke substance.

Since the dry, ash-free basis for coal ignores the changes in mineral matter when coal is burnt, the dry, mineral-matter-free basis is preferred whenever the mineral matter content can be calculated (see 5.5.2), even if some analytical values have to be assumed in the absence of results determined on the sample.

In the case of coal in the dry, mineral-matter-free state, it is considered to be free of both moisture and the various minerals associated with the coal when sampled. Coke on the same basis is considered to be free of both moisture and substances formed during the coking process from the minerals present in the coal charge.

The formulae for converting to this basis are shown in Table 2 and are applied by inserting the appropriate value for mineral matter content, either determined or calculated. Additionally, most analytical results require correction for changes in mineral matter during analysis and these are considered individually in 5.5.4.

It will rarely be necessary to calculate an analytical result expressed on the dry, mineral-matter-free basis to any other basis, but should the need arise, it is necessary that any correction added (or deducted) during the conversion is deducted (or added) at the appropriate stage of the reversion.

5.5.2 Mineral matter content of coal

Analytical methods are available for the direct determination of the mineral matter content of coal [1] [2].

In the absence of a determined value, the mineral matter content of a sample of coal can be estimated to a very close approximation from other analytical results by equations (3) to (5). These equations relate the mineral matter content of the ash and take account of the chemical changes that take place in the mineral matter when the coal is burned during the determination of ash. The principal changes are as follows:

- liberation of carbon dioxide from carbonates;
- liberation of water-of-constitution from silicates;
- conversion of iron pyrites to iron (III) oxide;
- liberation of chlorine from chlorides;
- fixation of sulfur gases by basic oxides.

Depending upon the information available, either of the following two equations may be used for calculating the mineral matter content of coal.

1) The King, Maries, and Crossley (K.M.C.) formula [3] modified by Millott [4] is based on a study of the nature of mineral matter associated with British coals. Its use requires a detailed chemical analysis to have been made¹⁾. The equation is as follows.

$$MM_{\text{ad}} = 1.13A_{\text{ad}} + 0.8[\text{CO}_2]_{\text{ad}} + 0.5S_{\text{p,ad}} + 2.8S_{\text{s,ad}} - 2.8S_{\text{a,ad}} + 0.5[\text{Cl}]_{\text{ad}} \quad (3)$$

where $S_{\text{a,ad}}$ is derived from the sulfur trioxide content of ash, $[\text{SO}_3]$, by the following equation.

$$S_{\text{a,ad}} = 0.40[\text{SO}_3] \times \frac{A_{\text{ad}}}{100} \quad (4)$$

2) Millott's original formula was modified by Brown, Caldwell and Fereday [5] to give the British Coal Utilization Research Association (BCURA) formula, as follows.

$$MM_{\text{ad}} = 1.10A_{\text{ad}} + 0.53S_{\text{ad}} + 0.74[\text{CO}_2]_{\text{ad}} - 0.36 \quad (5)$$

¹⁾ See BS 1016-14:1963.

Table 2 — Formulae for calculation of results to different bases

NOTE After correction, see 5.4 to 5.6.					
Given basis	Desired basis				
	Air-dried (as analysed)	As sampled (as received) (as despatched)(as fired)	Dry	Dry, ash-free	Dry, mineral-matter-free
Air-dried (as analysed)		$\frac{100 - M_{as}}{100 - M_{ad}}$	$\frac{100}{100 - M_{ad}}$	$\frac{100}{100 - (M_{ad} + A_{ad})}$	$\frac{100}{100 - (M_{ad} + MM_{ad})}$
As sampled (as received) (as despatched) (as fired)	$\frac{100 - M_{ad}}{100 - M_{as}}$		$\frac{100}{100 - M_{as}}$	$\frac{100}{100 - (M_{as} + A_{as})}$	$\frac{100}{100 - (M_{as} + MM_{as})}$
Dry	$\frac{100 - M_{ad}}{100}$	$\frac{100 - M_{as}}{100}$		$\frac{100}{100 - A_d}$	$\frac{100}{100 - MM_d}$
Dry, ash-free	$\frac{100 - (M_{ad} + A_{ad})}{100}$	$\frac{100 - (M_{as} + A_{as})}{100}$	$\frac{100 - A_d}{100}$		$\frac{100 - A_d}{100 - MM_d}$
Dry, mineral-matter-free	$\frac{100 - (M_{ad} + MM_{ad})}{100}$	$\frac{100 - (M_{as} + MM_{as})}{100}$	$\frac{100 - MM_d}{100}$	$\frac{100 - MM_d}{100 - A_d}$	

5.5.3 Mineral matter content of coke

There is no method for the direct determination of the mineral matter content of coke.

Various formulae have been proposed for calculating the mineral matter content, none of which has been found entirely satisfactory. A modification by the former National Coal Board [6] of an equation suggested by the British Coke Research Association [7] which has met with most approval is as follows.

$$MM_{ad} = A_{ad} + 0.68S_{ad} - 0.14[Fe]_{ad} + 0.14 \quad (6)$$

If the iron content of the coke, $[Fe]_{ad}$ is not known, an alternative equation is as follows.

$$MM_{ad} = A_{ad} + 0.51S_{ad} + 0.21 \quad (7)$$

5.5.4 Conversions

The following conversions should be used as appropriate.

a) *Carbon.*

$$C_{o,dmmf} = (C_{ad} - 0.273[CO_2]_{ad}) \times \frac{100}{100 - (M_{ad} + MM_{ad})} \quad (8)$$

For coal, the carbon present as carbonate should be deducted from the total carbon content. For coke, the carbonate correction is not applicable.

b) *Hydrogen.*

$$H_{dmmf} = (H_{ad} - 0.014A_{ad} + 0.02S_{p,ad} + 0.02[CO_2]_{ad}) \times \frac{100}{100 - (M_{ad} + MM_{ad})} \quad (9)$$

The first expression in parentheses is the total hydrogen content (excluding hydrogen from moisture) corrected for the hydrogen in the water-of-constitution of silicates (i.e. shale in the mineral matter, hence the pyrite and carbonate factors). This water-of-constitution is not released at the temperature (105 °C to 110 °C) used in the determination of moisture content. If the pyritic sulfur content has not been determined, the factor $0.02S_{p,ad}$ may be replaced by $(0.016S_{ad} - 0.01)$. The derivation of these correction factors is given in Annex A.

The water-of-constitution in shale is driven off during carbonization and corrections are not needed for the hydrogen content of coke.

c) *Nitrogen.*

$$N_{dmmf} = N_{ad} \times \frac{100}{100 - (M_{ad} + MM_{ad})} \quad (10)$$

d) *Sulfur.*

$$S_{o,dmmf} = (S_{ad} - S_{p,ad} - S_{s,ad}) \times \frac{100}{100 - (M_{ad} + MM_{ad})} \quad (11)$$

The total sulfur content as determined in coal includes pyritic sulfur and sulfate sulfur. These mineral forms of sulfur should be deducted to permit the calculation of the organic sulfur content to the dry, mineral-matter-free basis.

When the total sulfur content only has been determined and the BCURA formula [8] is used for estimating the mineral matter content, the organic sulfur content may be calculated from the following equation.

$$S_{o,dmmf} = (0.16S_{ad} + 0.60 - 0.005A_{ad} - 0.05[CO_2]_{ad}) \times \frac{100}{100 - (M_{ad} + MM_{ad})} \quad (12)$$

When the total sulfur content is less than 0.7 % (*m/m*), the error involved in assuming that it is all organic sulfur is small.

Sulfur is present in coke in organic form combined with carbon, in mineral form as iron (II) sulfide (not always as stoichiometric FeS) and as sulfate in lesser amount.

There are no means available for calculating accurately, on the dry, mineral-matter-free basis, the amount of organic sulfur present in coke. It may be assumed to be the difference between the total sulfur content and the mineral sulfur content (taken as the sum of the sulfide and sulfate sulfur contents).

e) *Oxygen.* A number of methods have been published for the experimental determination of the oxygen content of coal. The determined oxygen content includes the oxygen in the coal substance, in the carbonate minerals (as carbon dioxide), and in the silicates (as water).

A deduction for the oxygen in mineral matter is necessary before converting the oxygen content of coal to the dry, mineral-matter-free basis, as follows:

$$O_{o,dmmf} = (O_{ad} - O_{min,ad}) \times \frac{100}{100 - (M_{ad} + MM_{ad})} \quad (13)$$

In the absence of an agreed experimental method, the oxygen content of coal may be calculated by difference, all values being expressed on the dry, mineral-matter-free basis, as follows:

$$O_o = 100 - (C_o + H + N + S_o + 0.5[Cl]) \quad (14) \quad \text{or}$$

This value for the oxygen content of coal by difference is approximate, in that it contains the cumulative errors in the determinations of carbon content, hydrogen content, nitrogen content and chlorine content, as well as the errors involved in the calculation of the organic sulfur content and the mineral matter content; nor does it allow for any minor constituents that may be present in the coal substance. The oxygen content of coke is usually between 0.4 % (*m/m*) and 0.8 % (*m/m*). At this level, it cannot be estimated by the differences method with sufficient accuracy. An experimental method [9] is available for the determination of the oxygen content of coke which has a repeatability limit of 0.2.

f) *Chlorine*. The chlorine content determined on the analysis sample includes chlorine combined with the coal substance and chlorine from the mineral matter. The latter should be deducted before calculation. In the equation for oxygen by difference [see item e)] and in the following equation, the organic chlorine content is assumed [10] to be 50 % (*m/m*) of the total chlorine:

$$[Cl]_{o,dmmf} = 0.5[Cl]_{ad} \times \frac{100}{100 - (M_{ad} + MM_{ad})} \quad (15)$$

The form in which chlorine is present in coke is unknown. The amount rarely exceeds 0.2 % (*m/m*) and can be neglected in calculating coke analyses to the dry, mineral-matter-free basis.

5.5.5 Volatile matter

The as analysed value for the volatile matter content includes volatile matter evolved from minerals during heating; on the dry, mineral-matter-free basis the volatile matter from the coal substance only is required. One of the following three equations may be used according to the analytical data available [11].

$$V_{dmmf} = (V_{ad} - 0.13A_{ad} - 0.2S_{p,ad} - 0.7[CO_2]_{ad} - 0.7[Cl]_{ad} + 0.2) \times \frac{100}{100 - (M_{ad} + MM_{ad})} \quad (16)$$

$$V_{dmmf} = (V_{ad} - 0.13A_{ad} - 0.2S_{ad} - 0.7[CO_2]_{ad} - 0.7[Cl]_{ad} + 0.32) \times \frac{100}{100 - (M_{ad} + MM_{ad})} \quad (17)$$

or

$$V_{dmmf} = (V_{ad} - 0.13A_{ad} - 0.2S_{ad} - 0.7[CO_2]_{ad} + 0.12) \times \frac{100}{100 - (M_{ad} + MM_{ad})} \quad (18)$$

5.5.6 Gross calorific value at constant volume

The as analysed value for gross calorific value at constant volume is corrected for the heat of formation and dilution of sulfuric acid, assuming that all the sulfur in the coal or coke is converted into sulfuric acid. On the dry, mineral-matter-free basis, the aim is to represent the heat developed from the organic substance of the coal and, since the as analysed calorific value includes the heat liberated by pyrites burning to iron (III) oxide, a deduction for the latter should be made as follows.

$$Q_{gr,v,dmmf} = (Q_{gr,v,ad} - YS_{p,ad}) \times \frac{100}{100 - (M_{ad} + MM_{ad})} \quad (19)$$

where $y = 0.126$ MJ/kg.

If the pyritic sulfur in the coal has not been determined, the following assumption can be made:

$$S_p = 0.78S - 0.49 \quad (20)$$

and the correction ($YS_{p,ad}$) in the main equation becomes $0.098S_{ad} - 0.062$.

5.6 Calorific values, gross and net

BS 1016-105 describes methods for the determination of the gross calorific value at constant volume of the analysis sample. This may be calculated to the as sampled basis by using the formula given in Table 2, as follows:

$$Q_{\text{gr,v,as}} = Q_{\text{gr,v,ad}} \times \frac{100 - M_{\text{as}}}{100 - M_{\text{ad}}} \quad (21)$$

Three further calorific values may be calculated from the gross calorific value at constant volume. These are almost invariably required on an as sampled basis and the following equations take account of this requirement.

NOTE Details of the derivations of these equations are given in BS 7420:1991.

- $Q_{\text{gr,p}} = Q_{\text{gr,v,as}} + 0.006H_{\text{as}} - 0.0008O_{\text{as}}$;
- $Q_{\text{net,v}} = Q_{\text{gr,v,as}} - 0.206H_{\text{as}} - 0.023W$;
- $Q_{\text{net,p}} = Q_{\text{gr,v,as}} - 0.212H_{\text{as}} - 0.024W - 0.0008O_{\text{as}}$.

The values of H_{as} and O_{as} may be derived for practical purposes by multiplying H_{dmmf} and O_{dmmf} by

$$\frac{100 - (M_{\text{as}} + MM_{\text{as}})}{100}$$

and W may be taken as $M_{\text{as}} + 0.1A_{\text{as}}$.

6 Interpretation of results

6.1 Types of error

An error is measured by the amount a determined result differs from the true value. Three kinds of error are encountered in quantitative scientific work. In increasing order of importance for users of this standard they are as follows:

- systematic errors;
- random errors;
- experimental mistakes.

It should be noted that systematic and random errors arise from factors that are unknown or uncontrolled and the use of these terms should not be taken to imply carelessness or negligence.

6.2 Systematic error

Systematic error is present when results on average are persistently higher or lower than the true value. It can be detected and measured only by comparing results with a known true value or with results obtained by a method known to be accurate.

It is impossible to eliminate systematic errors entirely, but the methods described in BS 1016 have been chosen as being capable of yielding results with a negligible systematic error if the operator uses the equipment specified and adheres strictly to the procedures described.

6.3 Random error

Random error produces a scattering of results of repeated measurements about a central value, the arithmetic mean. It differs from systematic error in that, for a large number of results, the sum of the errors will tend to zero i.e. the mean result and true result will converge.

The distribution patterns of random error contain important information about sets of results. From the distribution of the results about the mean it is possible to infer the range within which the true value is likely to lie and to eliminate experimental mistakes (see 6.4). In this Part of BS 1016 the standard deviation, s , is used to describe the spread of results. The calculation of this characteristic is described in all textbooks on statistics.

The most widely used prediction for the spread of results for a single sample and the one adopted for this standard is that 95 % of all test results will lie within the range $\pm 1.960s$ of the mean.

Restating this and rounding off the constant, the assumption that the arithmetic mean of a large number of results will lie within $\pm 2s$ of a single result will be wrong only five times out of every 100 tests and will be correct 95 times.

It is the spread of random errors which determines the precision of results obtainable by the operator from the sample by the method prescribed.

Precision means the closeness of any single result to the mean of a large number of replicate results (see also 6.4.4 and 6.4.5). It is defined at the 95 % confidence level as twice the standard deviation, i.e.:

$$\text{precision} = 2s$$

The standard deviations of the tests described in BS 1016 have been derived from a large amount of experimental work over a long period in a large number of laboratories. From these standard deviations, the precision of single results at the 95 % confidence level has been calculated as $\pm 2s$. These values for precision, where appropriate, are given in the relevant test method. They show the random error that can be associated with a single test result, as in the following example.

- the standard deviation of the determination of gross calorific value, $Q_{\text{gr,v,ad}} = 0.045 \text{ MJ/kg}$;
- the precision of a single test result is $\pm 2 \times 0.045 \text{ MJ/kg} = \pm 0.09 \text{ MJ/kg}$;

— a test result of say 23.00 MJ/kg can be reported as (23.00 ± 0.09) MJ/kg, i.e. the mean of a large number of tests on the same sample obtained by the same operator using the same apparatus can be expected to lie between 23.09 MJ/kg and 22.91 MJ/kg.

The precision of reported results can be improved by accumulating further information from the results of additional (replicate) tests. The precision of the mean of a number of test results is numerically lower than that of a single result. It may be calculated from the following equation:

$$\text{precision of the mean} = 2s/\sqrt{n} \quad (22)$$

where n is the number of test results.

Thus, if duplicate tests are carried out, the precision of their mean will be $2s/\sqrt{2} = 1.414s$.

This is an improvement in precision of approximately 30 %.

NOTE The second determination may be more valuable to check that a gross mistake has not occurred in the first determination rather than to improve the precision.

Simple calculation shows that the improvement in precision falls off rapidly as more tests are carried out; thus four tests are needed to numerically halve the precision, and nine tests to reduce it to one third. The cost of replicate testing and the value of the consequently improved precision will be factors in assessing how many determinations should be carried out.

6.4 Experimental mistakes

6.4.1 General

Experimental mistakes are produced by faulty equipment or incorrect technique.

The reasons for a “wild” result can sometimes be obvious e.g. the operator may know that he/she did something wrong, or may have observed a functional failure in the apparatus. Such a test should be stopped or marked irrevocably for discard, even if the result should eventually come out close to expectation.

It is unwise to seek a reason for rejecting a result after the fact; prejudice and emotion tend to cloud judgement. Nevertheless, if a given circumstance is used as justification for rejecting a result, then results should be rejected at each recurrence of the same circumstance. Results should never be rejected merely because they flout prior expectations.

NOTE The given circumstance should not be so closely defined as to make recurrence highly unlikely. E.g. when a result is discarded because two events happened together, it should be remembered that if the chance of a given circumstance occurring is rare, say 1 in 1000, the chance of two such independent events occurring together is 1 in a million, i.e. highly unlikely.

The investigation of unusual results may sometimes prove worthwhile; important factors could be discovered leading to improved performances.

6.4.2 Duplicate testing

In the circumstances of a single determination, nothing can be known about the correctness or otherwise of the result. Accordingly, a second determination is carried out as a check. If the two results are in close agreement, both may be assumed to be correctly observed since the chance of the same error recurring is extremely slight; a wide discrepancy, however, may indicate that one of them is a mistaken value.

NOTE If an instrument is used, however, a check should be made to ensure that there is not an inherent error in the instrument.

6.4.3 Repeatability

It is important that repeat tests should be carried out under identical conditions, i.e. on the same analysis sample in the same laboratory by the same operator using the same apparatus but at a different time.

NOTE Tests for checking repeatability exclude the use of simultaneous duplicate determinations for checking repeatability.

The maximum acceptable difference between duplicate results obtained in this way is the repeatability limit of the method, which is given in the relevant test method in BS 1016 where appropriate. Duplicate results with a difference less than the value quoted for the method concerned can be accepted as having been carried out correctly in accordance with the procedure described. Results with a difference greater than the value quoted need checking by further testing (see 6.4.5).

The repeatability limit, r , given in each of the test methods has been calculated from the following equation:

$$r = ts\sqrt{2} \quad (23)$$

where

s is the standard deviation of a single set of results;

t is the appropriate value of Student's t at 95 % confidence level; this value varies with the number of independent results in a set and, in this particular context, is generally between 2.0 and 2.1.

6.4.4 Confirmatory tests

If the difference of duplicate results exceeds the appropriate repeatability limit, a mistake may have occurred, but it is not certain; further tests are needed to clarify this. Further testing, however, increases the chance of observing an outlying value and so the repeatability limit for duplicate results is inapplicable. The following procedures should be adopted.

- a) If the difference between duplicate determinations is greater than the appropriate repeatability limit, r , a further determination is carried out. If the spread of the three results is less than $1.2r$, all three may be accepted, otherwise a fourth determination is carried out.
- b) If the spread of the four results is less than $1.3r$, all may be accepted. If the spread is greater than $1.3r$ and three out of the four results lie within a range of $1.2r$, these three may be accepted and the fourth rejected. If neither of these conditions applies, a fifth determination may be carried out.
- c) If the spread of the five results is less than $1.4r$, all may be accepted. If not, and four out of five results lie within a range of $1.3r$, these four may be accepted and the fifth rejected.

A special investigation should be made of all circumstances affecting the analysis, sample homogeneity, apparatus, operation and other conditions if concordant results are not obtained from five tests, or if more than 5 % of duplicate results fail the repeatability test.

It is important to note two things in this procedure.

- a) The reported value is to be the mean of all the acceptable results.
- b) One outlying result may be rejected from four tests or from five tests. It is not possible to reject any result on the basis of only three tests, nor to reject two results from five tests.

By using this procedure 5 % of valid results will be discarded, but this is the necessary price to pay for confidence in the reported results.

An example is as follows.

The repeatability limit, r , of duplicate determinations of the gross calorific value, $Q_{gr,v,ad}$, is 0.125 MJ/kg. The maximum permissible spread for the number of results is then:

Number of results	Maximum permissible spread
3	$1.2r$ i.e. 0.150 MJ/kg
4	$1.3r$ i.e. 0.165 MJ/kg
5	$1.4r$ i.e. 0.175 MJ/kg

6.4.5 Reproducibility

The reproducibility limit, or the reproducibility critical difference, of each of the test methods described in BS 1016 is given in the relevant method, where appropriate. Because other variables are introduced when tests are not carried out on the same analysis sample, reproducibility limits generally are greater than the repeatability limits. They have been calculated by equation (23) given in 6.4.3 for repeatability limit, but using different values for the standard deviation, s' , appropriate to the spread of results between laboratories. These were derived from the experimental work described in 6.3.

Reported results (the mean of duplicate determinations) from two laboratories with a difference less than the reproducibility limit quoted for the method are equally valid providing both laboratories are working correctly in accordance with the procedure described. If the difference exceeds the reproducibility limit, the need for independent tests on the arbitration (reserve) sample and an investigation into methods of testing is indicated. If more than two laboratories are compared, the factors given in 6.4.5 may be applied to the reproducibility to indicate whether or not the spread of results is satisfactory.

6.5 Monitoring of laboratory/operator techniques

It is a practice in many laboratories to monitor the standard of work produced by a laboratory or operator by issuing, with the routine work, a special standardized sample, the test results of which have been well established by repeated testing by a number of operators. It is required that the reported result should be in reasonable agreement with the expected value, i.e. within $\pm 2s$. It is essential that standardized samples have long term stability.

An alternative procedure is to plot the spread of results from n replicate results from a routine sample tested by a particular laboratory or operator. Obviously such a control chart is valid only if the number n is kept constant and if all results are plotted, including those that would be discarded by the procedures described in 6.4.4. Trends towards improvement or deterioration in technique are revealed over a period of time. The advantage of this procedure over the use of a standardized sample is that the monitoring relates to routine work and an atmosphere of special testing is avoided. It is a well known fact that standards of performance are changed, usually for the better, when carried out under inspection.

One set of close results from replicate testing should not be accepted with greater approval than another set with a wider spread. Provided that both sets are within the prescribed repeatability limits, both sets are equally acceptable. It is only after examination of replicate test results over a period of time that judgements can be made comparing the quality of laboratories or operators.

NOTE Some large organizations are able to cross-check the work of all their laboratories, by distributing subsamples taken from the same bulk sample. In these circumstances more sophisticated statistical techniques can be employed.

Annex A (informative)
Derivations of the factors used for the calculation of hydrogen content to the dry, mineral-matter-free basis
 [see 5.5.4 b)]

The shale ash, A_{sh} , is given by Leighton and Tomlinson [11] as follows:

$$A_{sh} = A_{ad} - (1.25S_{p,ad} + 1.33[CO_2]_{ad} + 0.35[Cl]_{ad} + 0.5) \quad (B.1)$$

The average content of water of constitution of shales is 11.2 % (m/m) [4].

Therefore, 100 g of shale gives 11.2 g of water and 88.8 g of ash.

Therefore, the content of water of constitution of shale

$$= \frac{11.2}{88.8} \text{ of shale ash}$$

$$= 0.1261A_{sh}$$

Hence, the amount of hydrogen in the water of constitution of shale

$$= \frac{\{H_2\}}{\{H_2O\}} \times 0.1261A_{sh}$$

$$= \frac{2.016}{18.015} \times 0.1261A_{sh}$$

$$= 0.0141A_{sh}$$

Therefore, the correction to be applied is

$$- (0.014A_{ad} - 0.018S_{p,ad} - 0.19[CO_2]_{ad} - 0.005[Cl]_{ad} - 0.007)$$

which with suitable rounding becomes

$$- (0.014A_{ad} - 0.02S_{p,ad} - 0.02[CO_2]_{ad})$$

If the pyritic sulfur content has not been determined, the following relationship, from Leighton and Tomlinson [11], may be used in the above equations:

$$S_{p,ad} = 0.78S_{ad} - 0.49 \quad (B.2)$$

Then,

$$A_{sh} = A_{ad} - (0.975S_{ad} + 1.33[CO_2]_{ad} + 0.35[Cl]_{ad} + 0.11)$$

Therefore, the correction to be applied is

$$- (0.0141A_{ad} - 0.014S_{ad} - 0.019[CO_2]_{ad} - 0.005[Cl]_{ad} - 0.0016)$$

which when rounded becomes

$$- (0.014A_{ad} - 0.014S_{ad} - 0.02[CO_2]_{ad})$$

List of references (see clause 2)

Normative references

BSI publications

BRITISH STANDARDS INSTITUTION, London

BS 410:1986, *Specification for test sieves.*

Informative references

BSI publications

BRITISH STANDARDS INSTITUTION, London

BS 3323:1992, *Glossary of terms relating to sampling, testing and analysis of solid mineral fuels.*

BS 7420:1991, *Guide for determination of calorific values of solid, liquid and gaseous fuels.*

BS 1016, *Methods for analysis and testing of coal and coke.*

BS 1016-14:1963, *Analysis of coal ash and coke ash.*

BS 1016-15:1970, *Fusibility of coal ash and coke ash.*

BS 1016-17:1979, *Size analysis of coal.*

BS 1016-18:1981, *Size analysis of coke.*

BS 1016-20:1981, *Determination of Hardgrove grindability index of hard coal.*

BS 1016-21:1981, *Determination of moisture-holding capacity of hard coal.*

BS 1016-104, *Proximate analysis.*

BS 1016-105:1992, *Determination of gross calorific value.*

BS 1016-106, *Ultimate analysis of coal and coke.*

BS 1016-107, *Caking and swelling properties of coal.*

BS 1016-108, *Tests special to coke.*

BS 1016-111:1993, *Determination of abrasion index of coal.*

ISO publications

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION (ISO), Geneva. (All publications are available from BSI Sales.)

ISO 1170:1977, *Coal and coke — Calculation of analyses to different bases²⁾.*

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²⁾ Referred to in the foreword only.

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