

Australian Standard™

Coal and coke—Analysis and testing

**Part 16: Assessment and reporting of
results**

This Australian Standard was prepared by Committee MN-001, Coal and Coke. It was approved on behalf of the Council of Standards Australia on 29 April 2005. This Standard was published on 14 June 2005.

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Australian Building Codes Board
Australian Coal Association
Australian Coal Preparation Society
Australian Institute of Energy
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STANDARDS AUSTRALIA

RECONFIRMATION

OF

AS 1038.16—2005

Coal and coke—Analysis and testing

Part 16: Assessment and reporting of results

RECONFIRMATION NOTICE

Technical Committee MN-001 has reviewed the content of this publication and in accordance with Standards Australia procedures for reconfirmation, it has been determined that the publication is still valid and does not require change.

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NOTES

Australian Standard™

Coal and coke—Analysis and testing

Part 16: Assessment and reporting of results

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PREFACE

This Standard was prepared by the Standards Australia Subcommittee on Coal Evaluation, under the supervision of the Committee MN-001, Coal and Coke, as a revision of AS 1038.16—1996, *Coal and coke—Analysis and testing, Part 16: Assessment and reporting of results*.

This Standard will be continuously revised because of ongoing development and updating of all other Standards in the AS 1038 series and associated Standards. For this reason, information presented in each edition of AS 1038.16 may at any given time be ahead of or behind the actual state of development or updating of other Standards. It is therefore recommended that this Standard be used only in conjunction with other appropriate Standards.

This edition contains information regarding quality control (QC) sample testing and measurement uncertainty.

The objective of this Standard is to provide those involved with the testing of coal and coke with a means for assessing their test results to ensure that the values reported are consistent with the requirements of the industry.

The term ‘informative’ has been used in this Standard to define the application of the appendix to which it applies. An ‘informative’ appendix is for information and guidance only.

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STANDARDS AUSTRALIA

Australian Standard

Coal and coke—Analysis and testing

Part 16: Assessment and reporting of results

1 SCOPE

This Standard provides procedures and information for the assessment and reporting of results obtained by the use of methods detailed in the AS 1038 series and associated Standards. Technical aspects relevant to the derivation of formulas required for calculation and to the acceptance of analytical results are also provided.

2 REFERENCED DOCUMENTS

The following documents are referred to in this Standard:

AS

1038	Coal and coke—Analysis and testing
1038.1	Part 1: Higher rank coal—Total moisture
1038.2	Part 2: Coke—Total moisture
1038.3	Part 3: Proximate analysis of higher rank coal
1038.4	Part 4: Coke—Proximate analysis
1038.5	Part 5: Gross calorific value
1038.6.1	Part 6.1: Higher rank coal and coke—Ultimate analysis—Carbon and hydrogen
1038.6.2	Part 6.2: Higher rank coal and coke—Ultimate analysis—Nitrogen
1038.6.3.1	Part 6.3.1: Higher rank coal and coke—Ultimate analysis—Total sulfur—Eschka method
1038.6.3.2	Part 6.3.2: Higher rank coal and coke—Ultimate analysis of higher rank coal—Total sulfur—High-temperature combustion method
1038.6.3.3	Part 6.3.3: Higher rank coal and coke—Ultimate analysis—Total sulfur—Infrared method
1038.8.1	Part 8.1: Coal and coke—Chlorine—Eschka method
1038.8.2	Part 8.2: Coal and coke—Chlorine—High-temperature combustion method
1038.9.1	Part 9.1: Higher rank coal and coke—Phosphorus—Ash digestion/molybdenum blue method
1038.9.2	Part 9.2: Higher rank coal and coke—Phosphorus—Coal extraction/phosphomolybdovanadate method
1038.9.3	Part 9.3: Higher rank coal and coke—Phosphorus—Ash digestion/phosphomolybdovanadate method
1038.10.1	Part 10.1: Determination of trace elements—Coal, coke and fly-ash—Determination of eleven trace elements—Flame atomic absorption spectrometric method
1038.10.2	Part 10.2: Determination of trace elements—Coal and coke—Determination of arsenic, antimony and selenium—Hydride generation method
1038.10.3	Part 10.3: Determination of trace elements—Coal and coke—Determination of boron content—ICP-AES method
1038.10.4	Part 10.4: Determination of trace elements—Coal, coke and fly-ash—Determination of fluorine content—Pyrohydrolysis method

- 1038.10.5.1 Part 10.5.1: Determination of trace elements—Coal, coke and fly-ash—Trace elements—Determination of mercury content—Tube combustion method
- 1038.11 Part 11: Coal—Forms of sulfur
- 1038.12.1 Part 12.1: Higher rank coal—Caking and coking properties—Crucible swelling number
- 1038.12.2 Part 12.2: Higher rank coal—Caking and coking properties—Determination of Gray-King coke type
- 1038.12.3 Part 12.3: Higher rank coal—Caking and coking properties—Dilatation
- 1038.12.4.1 Part 12.4.1 Higher rank coal—Caking and coking properties—Plasticity—Continuous-torque Gieseler method
- 1038.12.4.2 Part 12.4.2 Higher rank coal—Caking and coking properties—Plasticity—Discontinuous-torque Gieseler method
- 1038.13 Part 13: Tests specific to coke
- 1038.14.1 Part 14.1: Higher rank coal ash and coke ash—Major and minor elements—Borate-fusion/flame atomic absorption spectrometric method
- 1038.14.2 Part 14.2: Higher rank coal ash and coke ash—Major and minor elements—Acid digestion/flame atomic absorption spectrometric method
- 1038.14.3 Part 14.3: Higher rank coal ash and coke ash—Major and minor elements—Wavelength dispersive X-ray fluorescence spectrometric method
- 1038.15 Part 15: Higher rank coal ash and coke ash—Ash fusibility
- 1038.17 Part 17: Higher rank coal—Moisture-holding capacity (equilibrium moisture)
- 1038.18 Part 18: Coke—Size analysis
- 1038.19 Part 19: Higher rank coal—Abrasion index
- 1038.20 Part 20: Higher rank coal—Hardgrove grindability index
- 1038.21.1.1 Part 21.1.1 Higher rank coal and coke—Relative density—Analysis sample/density bottle method
- 1038.21.1.2 Part 21.1.2 Higher rank coal and coke—Relative density—Analysis sample/volumetric method
- 1038.21.2 Part 21.2: Higher rank coal and coke—Relative density—Lump sample
- 1038.22 Part 22: Higher rank coal—Mineral matter and water of constitution
- 1038.23 Part 23: Higher rank coal—Carbonate carbon
- 1038.25 Part 25: Coal—Durham cone handleability
- 1152 Specification for test sieves
- 2418 Coal and coke—Glossary of terms
- 2434 Methods for the analysis and testing of lower rank coal and its chars
- 2434.1 Part 1: Determination of the total moisture content of lower rank coal
- 2434.2 Part 2: Lower rank coal—Determination of the volatile matter
- 2434.3 Part 3: Lower rank coal—Determination of the moisture holding capacity
- 2434.4 Part 4: Dried lower rank coal and its chars—Determination of apparent density—Mercury displacement method
- 2434.5 Part 5: Lower rank coal and its chars—Determination of moisture in bulk samples of lower rank coal and in analysis samples of char
- 2434.6 Part 6: Lower rank coal—Ultimate analysis—Classical methods
- 2434.7 Part 7: Lower rank coal—Determination of moisture in the analysis sample
- 2434.8 Part 8: Lower rank coal—Determination of ash
- 2434.9 Part 9: Determination of four acid-extractable ions in lower rank coal
- 2617 Sampling from coal seams
- 2706 Numerical values—Rounding and interpretation of limiting values

2850	Chemical analysis—Interlaboratory test programs—For determining precision of analytical method(s)—Guide to the planning and conduct
AS	
4156	Coal preparation
4156.1	Part 1: Higher rank coal—Float and sink testing
4264	Coal and coke—Sampling
4264.1	Part 1: Higher rank coal—Sampling procedures
4264.2	Part 2: Coke—Sampling procedures
4264.3	Part 3: Lower rank coal—Sampling procedures
ISO	
602	Coal—Determination of mineral matter
1994	Hard coal—Determination of oxygen content
ISO/IEC	
17025	General requirements for the competence of testing and calibration laboratories
BS	
1016	Methods for the analysis and testing of coal and coke
1016-100	Part 100: General introduction and methods for reporting results

3 TERMS, DEFINITIONS, NOTATION AND ABBREVIATIONS

For the purpose of this Standard, the definitions given in AS 2418 apply.

Symbols are used to represent quantities reported, primarily as a percentage by mass in the coal or coke. Major inorganic constituents of coal and coke are normally expressed as percentages by mass of the respective oxides in ash. However, they may be reported as the percentage by mass of the element in coal or coke. Trace elements, having concentrations less than approximately 0.1% by mass, may be reported more conveniently as milligrams per kilogram in the coal or coke. Calorific value is reported as megajoules per kilogram, whereas relative density is dimensionless.

<i>A</i>	ash
<i>AI</i>	abrasion index
<i>c</i>	maximum contraction
<i>C</i>	carbon (total, including carbon in minerals)
<i>C_m</i>	carbonate carbon from minerals
<i>C_o</i>	organic carbon
<i>CRI</i>	coke reactivity index
<i>CSR</i>	coke strength after reaction
<i>CSN</i>	crucible swelling number
<i>CV</i>	See <i>q</i>
<i>FC</i>	fixed carbon ($100 - A - M - VM$)
<i>F_s</i>	specific flow time
<i>H</i>	hydrogen (total, excluding moisture content of the air-dry sample, but including hydrogen from water of hydration in minerals)
<i>d</i>	maximum dilatation (may be prefixed by a positive or negative sign)

H_o	organic hydrogen
HGI	Hardgrove grindability index
I_{40}, I_{20}, I_{10}	IRSID indices
m	mass (may be numerically subscripted)
M	moisture
M_f	free moisture
M_r	residual moisture
M_{40}, M_{10}	Micum indices
MHC	moisture-holding capacity
MI	minerals and inorganics (lower rank coals)
MM	mineral matter
n	subgroup size; the number of sample observations per subgroup
O	oxygen (total, excluding oxygen from moisture)
O_o	organic oxygen
$q_{gr,v}$	calorific value (determined gross at constant volume)
r	repeatability
R	reproducibility
RD	relative density
s	standard deviation (total) $s = \sqrt{\frac{\sum (X_i - \bar{X})^2}{n-1}}$
s_w	within laboratory/operator standard deviation
s_b	between laboratory/operator standard deviation
S	sulfur (total)
S_o	organic sulfur
S_p	pyritic sulfur
S_s	sulfate sulfur
t	Student t statistic
T	temperature (may be numerically subscripted)
VM	volatile matter (excluding moisture but including water of hydration, carbon dioxide from carbonate minerals, and some pyritic sulfur)
VM_o	organic volatile matter
W	water of hydration from minerals
X	Value of measured quality characteristic (individual values are expressed as (X_1, X_2, X_3, \dots)).
\bar{X}	(X bar) Subgroup average value: $\bar{X} = \frac{\sum X_i}{n}$

Subscripted lower case symbols are used to distinguish between the different bases upon which results may be reported:

ad	Air-dry: the condition in which the analytical sample is spread out and conditioned through exposure for sufficient time so that its moisture content comes into equilibrium with the humidity of the laboratory atmosphere. This is normally the condition of the sample as analysed.
afm	Ash-free, moist: a hypothetical condition in which the sample is assumed to be free of ash, with a moisture determined by its moisture-holding capacity as determined by AS 1038.17.
ar	See 'as'.
as	As sampled (as despatched, as fired, as received): the result is not subject to any qualification. The analyses are reported for the sample having total moisture as sampled.
d	Dry: the condition in which the sample is assumed to be completely free of moisture, thereby removing the dependence of an analysis result on the humidity of a laboratory atmosphere.
daf	Dry, ash-free: a hypothetical condition for which the sample is assumed to be completely free of moisture and ash.
dmif	Dry, minerals and inorganics-free: a hypothetical condition applicable to lower rank coals only, in which the sample is assumed to be completely free of moisture, mineral matter and inorganic ions; used to compare the organic component concentrations of samples.
dmmf	Dry, mineral matter-free: a hypothetical condition in which the sample is assumed to be completely free of moisture and mineral matter; used primarily to compare organic component concentrations in samples.
domf	Dry, organic matter-free: a hypothetical condition in which the sample is assumed to be completely free of moisture and organic matter; used to compare mineral component concentrations in samples.
est	Estimated by calculation from a formula

Calorific value (q) can be calculated to alternative conditions:

$q_{gr,p}$	Gross calorific value at constant pressure
$q_{n,p}$	Net calorific value at constant pressure
$q_{n,v}$	Net calorific value at constant volume

Net calorific value figures values are usually required on an as-sampled basis.

Some examples of the usage of these symbols include:

A_d	Dry basis ash
M_{as}	Moisture as sampled (total moisture)
$S_{o,dmmf}$	Dry, mineral matter-free organic sulfur
VM_{daf}	Dry, ash-free volatile matter
P_{domf}	Dry, organic matter-free phosphorus

NOTE: The derivation of equations used for method calculations is given in Appendix A. A suggested hierarchy of parameters for a test report is given in Appendix B.

4 INTEGRITY OF RESULTS

4.1 General

The quality of results obtained by use of methods referred to in this Standard will be maximized when attention is given to each phase of the analytical procedure, from initial selection of sample to final reporting. The general requirements of quality assurance systems relevant to laboratory testing operations are given in ISO/IEC 17025. Statistical quality control techniques should also be applied as detailed below.

4.2 Sample collection

Incorrect methods of sample collection will negate all future care that is taken with subsequent operations of sample preparation and analysis. Standard procedures are given in AS 2617, AS 4264.1, AS 4264.2 and AS 4264.3 for the sampling of coal and coke. Special treatment of the sample within the laboratory for a particular method is detailed within the relevant Standard in the AS 1038 or AS 2434 series or associated Standards.

5 ASSESSING THE ACCURACY OF TEST RESULTS

5.1 General

A single test result, or a number of test results obtained under repeatability conditions, provides no information as to the accuracy of the result(s). To assess the accuracy, it is necessary to obtain a result, hereafter referred to as the reference result, concurrently on a suitable reference material and then to compare that result with the certified or reference value.

5.2 Reference material

Standards Australia has prepared several reference materials, e.g. coal and coal ash, that have certified or verified characteristics. In the absence of suitable certified or verified materials, a sample that has well accepted results because of repeated testing over a period of time by several different laboratories may serve for validation. It is important for any such reference material to maintain long-term stability. To minimize matrix effects, the chemical and physical composition of the certified reference material or other validated reference material should match, as closely as possible, that of the sample.

5.3 Accuracy of the reference result

If the 'reference value' for the reference material is denoted by m , and the laboratory's test result from n replicate tests is determined to be y , the difference should be tested as follows:

$$D|y - m| \leq \frac{1}{\sqrt{2}} \sqrt{R^2 - r^2 \frac{n-1}{n}} \quad \dots .5(1)$$

Two particular cases may be considered as follows:

- (a) When $n = 1$, the critical difference between the single result and the reference value is $\frac{R}{\sqrt{2}}$, i.e. $0.7R$.
- (b) When $n = 2$, the critical difference between the mean of the results and the reference value is $0.7 \sqrt{R^2 - \frac{r^2}{2}}$.

For further information, see AS 2850.

5.4 Accuracy of the test result

A test result can be assessed as meeting the accuracy requirements of the method only if the reference result determined concurrently with the test result is shown to be in agreement with the reference value (see Clause 5.3) and the reference material is appropriate for the test sample (see Clause 5.2).

5.5 Comparison of results with calculated values

In the assessment of results for reporting, it can sometimes be useful to compare results for VM_o and q with calculated values. These may be obtained by application of the equations based upon Seyler's relationships between carbon and hydrogen and volatile matter and calorific value in bright coals.* Useful indicative values may be so obtained and, for normal bright coals, differences between actual and calculated values will generally be within reproducibility tolerances. These equations are:

$$VM_{o,d,est} = 10.6 H_{o,d} - 1.24 C_{o,d} + 84.1 \quad \dots 5(2)$$

$$q_{d,est} = 1.62 H_{o,d} + 0.519 C_{o,d} - 17.9 \quad \dots 5(3)$$

Similarly, carbon and hydrogen may be estimated by the following equations:

$$C_{o,d,est} = 1.409 q_d - 0.216 VM_{o,d} + 43.4 \quad \dots 5(4)$$

$$H_{o,d,est} = 0.165 q_d + 0.069 VM_{o,d} - 2.86 \quad \dots 5(5)$$

6 ASSESSING THE PRECISION OF TEST RESULTS

6.1 Replicate sample testing

6.1.1 Procedure

Increased confidence in a test result may be achieved by carrying out the complete test method more than once. Where the method has been carried out twice, the difference between the test results should not exceed the value of r . If this is not the case, the usual recourse is to carry out the test method again. The recommended procedure is as follows:

- (a) If the difference between duplicate test results is greater than r , carry out the procedure a third time. All three results may be accepted if the range of test results is less than $1.2r$; otherwise the method should be carried out a fourth time.
- (b) If the range of four test results is less than $1.3r$, all may be accepted. If the range is greater than $1.3r$ but three of the four are within $1.2r$, these three may be accepted and the fourth rejected. Should neither condition apply, the method should be carried out a fifth time.
- (c) If the range of five test results is less than $1.4r$, all may be accepted. If the range is greater than $1.4r$ but four of the five are within $1.3r$, these four may be accepted and the fifth rejected.
- (d) If one outlier is obtained from four or five test results, it may be rejected, but it is invalid to reject any of only three test results or to reject two from five test results.
- (e) If five test results fail to achieve acceptability according to the above scheme, a detailed investigation should be made into all aspects of the test method procedure, including apparatus performance, operator technique, sample homogeneity, and sample preparation.

* SEYLER, C.A. 'Petrography and the classification of coal I and II.' *Proc. S. Wales Inst. Engrs*, 1938, 53, 254-327.

6.1.2 Application of procedure

Replicate application of the complete test method procedure may be carried out in several ways.

Within a laboratory, the method may be replicated concurrently or under repeatability conditions. In the former instance, it is expected that the difference between duplicate test results will be less than the value of r for the test method concerned. In the case of repeatability conditions, the relevant factor by which r should be multiplied for two or more replicate results is described in Clause 6.1.

For further information, see AS 2850.

NOTE: The precision of test methods in the AS 1038 and AS 2434 series is given in Appendix C.

6.1.3 Reproducibility

For integration and comparison of replicate results obtained in two or more laboratories, the principles in Clause 6.1 are also relevant for the factorization of R .

6.2 QC Sample testing

6.2.1 General

Additional information on test result precision may be determined through regular testing of a quality control sample. A reference material, such as that described in Clause 5.2 can be used for this purpose. If no such material is available, a stable and homogenous material should be obtained. A QC sample should be tested with each batch of samples tested.

When sufficient data for the QC sample has been collected, usually about 15 measurements, the average and standard deviation of the results should be determined.

6.2.2 Control charting

A control chart should be constructed for the QC sample data. A control chart (shown in Figure 1) is a plot of the result obtained for an individual determination of the control sample over time. The control chart Central line (CL) is the average result (as calculated above).

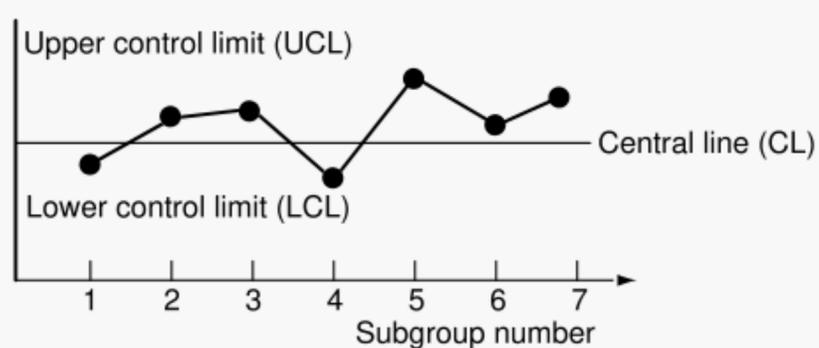


FIGURE 1 OUTLINE OF A CONTROL CHART

The upper and lower control limits are obtained using the equations:

$$\text{UCL} = \bar{X} + 3s \quad \dots 6(1)$$

$$\text{LCL} = \bar{X} - 3s \quad \dots 6(2)$$

When a result for a QC sample is outside the control limits, testing should be stopped and the cause for the outlier investigated. Testing should only be resumed when action has been implemented to address the cause of the outlier. Any samples tested with the outlying QC sample should be re-tested.

It may also be convenient to include warning limits on the control chart using the equation:

$$\text{UWL} = \bar{X} + 2s \quad \dots 6(3)$$

$$\text{LWL} = \bar{X} - 2s \quad \dots 6(4)$$

When a result for a QC sample is outside the warning limit (but still inside the control limit, testing may continue, but action should be taken to investigate the cause for the outlying result before results go outside the control limits.

Further information may be obtained using additional tests on the control chart. A set of eight supplementary tests used for interpreting patterns in control charts is schematically presented in Figure 2. Although this can be taken as a basic set of tests, analysts should be alert to any unique patterns of points that might indicate the influence of special causes in their process. These tests should therefore be viewed as simply practical rules for action whenever the presence of assignable causes is indicated. An indication of any of the conditions stipulated in these tests is an indication of the presence of assignable causes of variation that must be diagnosed and corrected. The upper and lower control limits are set at a distance of $3s$ above and below the central line. For the purpose of applying the tests, the control chart is equally divided into six zones, each zone being $1s$ wide. These are labelled A, B, C, C, B, A with zones C placed symmetrically about the central line.

6.2.3 Intermediate precision

The QC sample testing described in Clause 6.2.1 also provides the user of a test method with an intermediate measure of the precision of the test.

The intermediate precision can be calculated from the QC sample standard deviation using the equation:

$$R_{\text{INT}} = 2.83 s \quad \dots 6(5)$$

The value obtained for R_{INT} should lie between the repeatability and reproducibility limits, i.e. $r < R_{\text{INT}} < R$. A value greater than r should be expected as results are obtained over a greater time and may be performed by different operators and using different equipment. A value of less than R is expected as all results are produced in the same laboratory.

6.3 Uncertainty of measurement

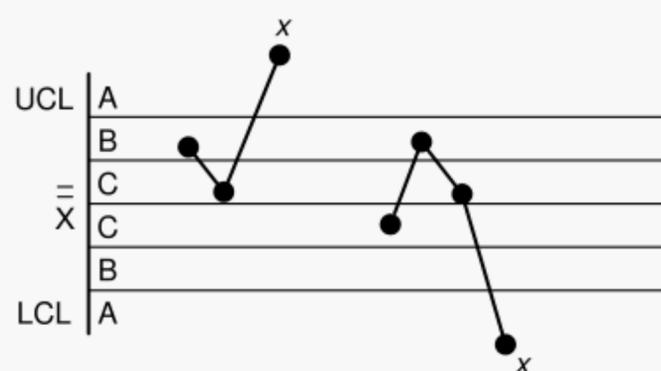
Users of the AS 1038 and AS 2434 series of coal and coke test methods and those who use the results obtained by these methods should be aware of the variability of the results which may be obtained, which is commonly referred to as the uncertainty of measurement. When a laboratory and its client agree on the test method to be used for a particular analysis, then it is assumed that all parties are aware of this variability.

NOTE: The best estimate of the variability of these test methods is the repeatability and reproducibility values quoted within each test method and duplicated in Appendix C of this Standard.

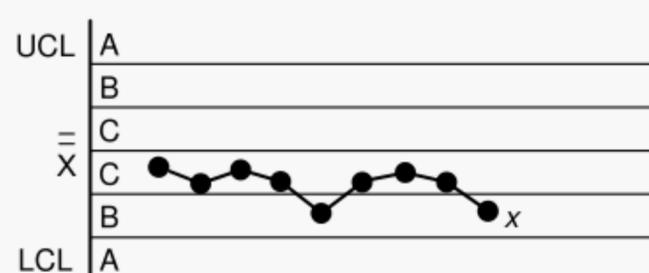
A laboratory using any of the test methods from the AS 1038 and AS 2434 series of coal and coke test methods should demonstrate its ability to meet the variability of the Standard methods by following the process described in Clauses 6.1 and 6.2 of this Standard.

A client of a laboratory using any of the test methods from the AS 1038 and AS 2434 series of coal and coke test methods should ensure that the laboratory is following the process described in Clauses 6.1 and 6.2 of this Standard.

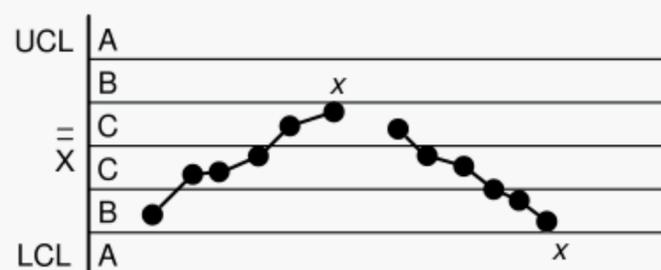
In some cases, additional sources of measurement variability may occur. These may need to be assessed and quantified, for more information on this process refer to the Eurachem/CITAC Guide 'Quantifying Uncertainty in Analytical Measurement'.



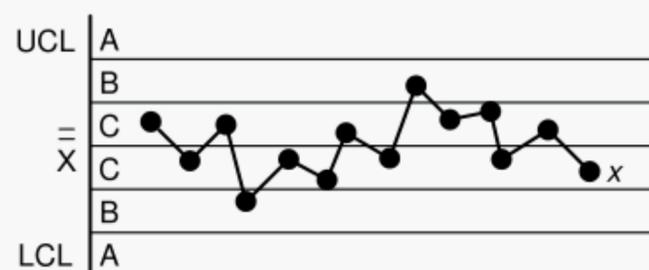
Test 1 One point beyond zone A



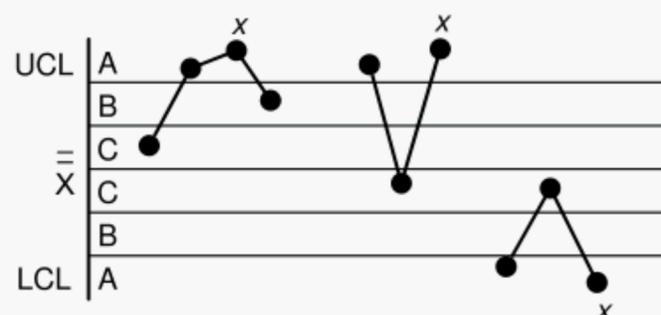
Test 2 Nine points in a row in zone C or beyond one side of central line



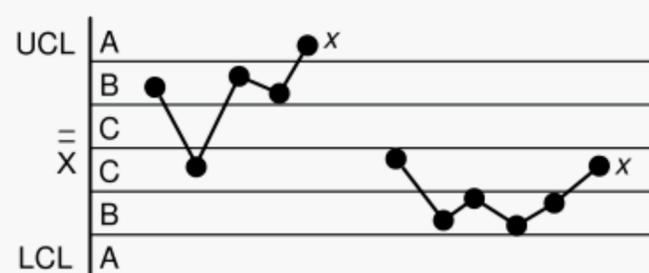
Test 3 Six points in a row steadily increasing or decreasing



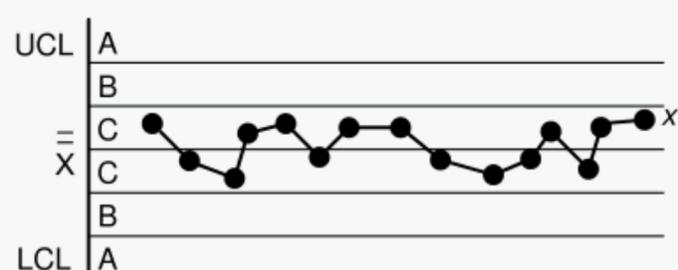
Test 4 Fourteen points in a row alternating up and down



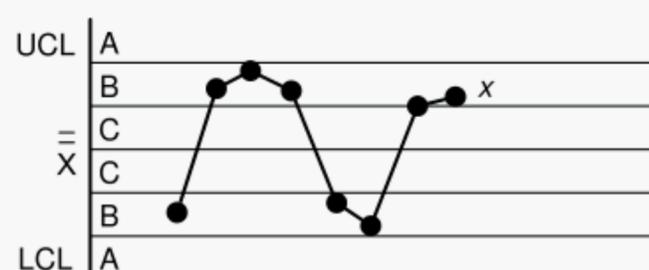
Test 5 Two out of three points in a row in zone A or beyond



Test 6 Four out of five points in a row in zone B or beyond



Test 7 Fifteen points in a row in zone C above and below central line



Test 8 Eight points in a row on both sides of central line with none in zone C

FIGURE 2 TESTS FOR ASSIGNABLE CAUSES

7 REPORTING ANALYSES TO DIFFERENT BASES

7.1 General

Chemical analysis methods covered by this Standard are carried out on coal or coke samples that have been pulverized to pass a 212 μm test sieve that complies with AS 1152. Samples are air-dried for minimal time to achieve effective equilibrium with the temperature and humidity of the laboratory atmosphere. Results so obtained may require conversion to another of the bases defined in Clause 3. Factors for conversion between bases are summarized in Table 1.

Discretion is required in the practical use of these factors. For example, reporting results on a dmmf basis is appropriate for components such as organic carbon and nitrogen, but not for mineral components such as carbonate carbon, pyritic sulfur, or phosphorus; similarly, the reverse condition applies for a domf basis, which is appropriate for comparing mineral but not organic components. Certain corrections have to be applied to the results of some analysis in order that valid comparisons can be made; these corrections are detailed in Clause 7.2.

The variety of bases for reporting, the corrections that may or may not be made, and the individual requirements that may occur in a particular reporting environment prevent the establishment of any preferred basis and uniformity in reporting results. This being so, it is critical that any corrections to an air-dry basis result be strictly in accordance with Clause 7.2, and that the use and accuracy of subscripts be adhered to.

7.2 Corrections applicable to analytical results

7.2.1 Volatile matter (AS 1038.3, AS 1038.4 and AS 2434.2)

Volatile matter as determined in coal includes some components of the mineral matter that may be volatilized during the pyrolysis procedure. Volatile matter (air-dry) is corrected for moisture by the calculations for VM_{ad} presented in the relevant parts, i.e.

$$VM_{o,ad} = VM_{ad} - 3.664C_{mm} - 0.5S_{p,ad} - W_{h,ad} \quad \dots 7(1)$$

Due to the empirical nature of the volatile matter procedure, caution is advised because these corrections to VM_{ad} may not necessarily be quantitatively valid for all samples.

With coke, volatile mineral components are assumed to be driven off from the feed coal during coke formation, and the determined VM_{ad} may be considered to be wholly organic.

7.2.2 Calorific value (AS 1038.5)

The energy released per unit quantity of fuel under constant volume conditions, where the products of combustion are at 25°C and the product water is in the liquid phase, is called the gross calorific value ($q_{gr,v}$). If the product water is in the vapour phase (at 0.1 MPa), the energy released per unit quantity of fuel is then called the net calorific value ($q_{net,v}$).

The gross or net calorific value, where determined under constant volume conditions (the usual case for solid and liquid fuels), approximates the internal energy of combustion of the fuel ($-\Delta U_o$), at the reference temperature of 25°C. The gross or net calorific value, where determined under constant pressure conditions (the usual case for gaseous fuels), approximates the enthalpy of combustion of the fuel (ΔH_o), at the reference temperature of 25°C. Contrary to the definition of ΔU_o and ΔH_o , it is usual to quote calorific value figures as positive quantities. The calorific value differs from the reference quantities referred to above in the temperatures of products and reactants and also heat transfer conditions.

The energy of vaporization (constant volume) for water at 25°C is 41.53 kJ/mol. This corresponds to 206.0 J/g for 1% of hydrogen in the fuel sample or 23.05 J/g for 1% moisture, respectively. The net calorific value at constant volume is derived from the corresponding gross calorific value (expressed in megajoules per kilogram) as follows:

$$q_{net,v,ad} = q_{gr,v,ad} - 0.206 H_{ad} - 0.0230 (M_{ad} + W_{ad}) \quad \dots 7(2)$$

The enthalpy of vaporization (constant pressure) for water at 25°C is 44.01 kJ/mol. This corresponds to 218.3 J/mol for 1% of hydrogen in the fuel sample or 24.43 J/g for 1% of moisture, respectively. The normal combustion of coal in a pulverized coal-fired boiler approximates 'net, constant pressure' conditions. The net calorific value at constant pressure for the air-dry sample may be derived from the gross calorific value at constant volume (expressed in megajoules per kilogram) as follows:

$$q_{net,p,ad} = q_{gr,v,ad} - 0.212 H_{ad} - 0.0008 (O_{ad} + N_{ad}) - 0.0244 (M_{ad} + W_{ad}) \quad \dots 7(3)$$

The relationship between gross calorific value at constant pressure and that at constant volume (expressed in megajoules per kilogram) is given by the following equation:

$$q_{\text{gr,p,ad}} = q_{\text{gr,v,ad}} - 0.006 H_{\text{ad}} - 0.001 (O_{\text{ad}} + N_{\text{ad}}) - 0.001 (M_{\text{ad}} + W_{\text{ad}}) \quad \dots 7(4)$$

For practical purposes, W_{ad} may be estimated as $0.1A_{\text{ad}}$, and H_{ad} and O_{ad} may be estimated by multiplying H_{dmmf} and O_{dmmf} by —

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

Where a value for MM_{ad} is not available, substitute H_{daf} , O_{daf} and $1.1A_{\text{ad}}$ for H_{dmmf} , O_{dmmf} and MM_{ad} , respectively.

It is assumed that the products of combustion of the fuel in the calorimeter consist of gaseous oxygen, nitrogen, carbon dioxide and sulfur dioxide, and water in either the liquid or gaseous phase. In calculating gross calorific value to a dry, mineral matter-free basis, a correction is necessary for the heat of combustion of pyritic to iron(III) oxide:

$$q_{\text{gr,v,dmmf}} = \frac{q_{\text{gr,v,ad}} - 0.1265S_{\text{p,ad}}}{100 - (M_{\text{ad}} + MM_{\text{ad}})} \quad \dots 7(5)$$

NOTE: The unit of calorific value is megajoules per kilogram (MJ/kg), which is equivalent to gigajoules per tonne (GJ/t). For conversion to —

- (a) kcal/kg, multiply MJ/kg by 238.85; and
- (b) Btu/lb, multiply MJ/kg by 429.92 or kcal/kg by 1.80.

7.2.3 Carbon (AS 1038.6.1 and AS 2434.6)

Total carbon, as determined in coal, comprises organic carbon and carbonate carbon from the mineral matter. The organic carbon component is therefore calculated from—

$$C_{\text{o,ad}} = C_{\text{ad}} - C_{\text{m,ad}} \quad \dots 7(6)$$

Carbonate carbon is not found in lower rank coal having pH less than 5.

Coke is manufactured at temperatures that will decompose carbonate minerals in the feed coal. Total carbon as determined in coke is considered to be wholly organic.

7.2.4 Hydrogen (AS 1038.6.1 and AS 2434.6)

Total hydrogen, as determined in coal or coke, comprises organic hydrogen, hydrogen in the water of constitution of the mineral matter, and hydrogen in the moisture. Hydrogen is explicitly corrected for the moisture component of the air-dry sample in the calculation as presented in AS 1038.6.1 and AS 2434.6. Organic hydrogen is calculated from—

$$H_{\text{o,ad}} = H_{\text{ad}} - \frac{W_{\text{h,ad}}}{9} \quad \dots 7(7)$$

With coke, the water of constitution is driven off during formation, and the determined total hydrogen is considered to be wholly organic.

7.2.5 Sulfur (AS 1038.6.3.1, AS 1038.6.3.2, AS 1038.6.3.3 and AS 2434.6)

Total sulfur, as determined in coal, includes pyritic and sulfate sulfur forms in the mineral matter. Organic sulfur is calculated from—

$$S_{\text{o,ad}} = S_{\text{ad}} - S_{\text{p,ad}} - S_{\text{s,ad}} \quad \dots 7(8)$$

During the coking process, pyrite is partially decomposed to non-stoichiometric iron(II) sulfide, but there is no available standard method that will determine all forms of sulfur in coke.

7.2.6 Chlorine (AS 1038.6.3.2 and AS 1038.8.1)

No information is available concerning the partitioning of chlorine between organic and mineral matter in Australian coals, though a conventional assumption is that it is largely associated with the mineral matter in higher rank coal. In lower rank coals, significant quantities of sodium chloride may be present in the water phase. Caution is needed, however, as some laboratory samples subjected to float-and-sink testing according to AS 4156.1 may be severely contaminated by halogens from organic liquid media used. An accompanying, though less significant, contamination of organic carbon and volatile matter would then also be present.

7.2.7 Mineral matter and water of constitution (AS 1038.22)

The equations presented in AS 1038.22 are comprehensive and do not require additional treatment within the scope of this Standard. The following two methods are described in AS 1038.22 for mineral matter determination:

- (a) Radio-frequency oxygen plasma oxidation.
- (b) Air oxidation (370°C).

The radio-frequency method yields a residue that contains essentially the original unchanged mineral matter of the coal. This residue is suitable for semi-quantitative analysis of the mineral matter composition. When applied to higher rank coals, irreversible loss of water of constitution from hydrated calcium sulfate minerals, partial oxidation of pyrite and other transformations of minerals may occur to some extent. In general, errors associated with these transformations are insignificant.

The air oxidation method may be used to estimate the quantity of mineral matter in the coal. However, the oxidation residue is unsuitable for semi-quantitative analysis of the mineral matter composition due to the oxidation of siderite and pyrite, and significant irreversible loss of water of constitution from the mineral matter of high-ash or high-gypsum coal. This method is generally not suitable for lower rank coal, except where there is a negligible amount of non-mineral inorganic matter.

7.2.8 Oxygen

Although oxygen is a significant component of coal and coke, there has been insufficient demand for its direct determination to justify preparation of an Australian Standard. International Standard ISO 1994 presents a high-temperature pyrolysis method that recommends demineralization of the sample, according to ISO 602, if the ash exceeds approximately 5%. All necessary corrections for oxygen in moisture and mineral matter are presented in the Standard for calculation of organic oxygen. An Australian implementation of these principles has been published.*

An indication of organic oxygen in coal may be obtained 'by difference'.

$$O_{o,dmmf} = 100 - (C_{o,dmmf} + H_{o,dmmf} + N_{dmmf} + S_{o,dmmf}) \quad \dots 7(9)$$

Caution needs to be exercised with the approximate result derived for 'oxygen by difference'. It incorporates the summation of errors in the results for total carbon, carbonate carbon, total hydrogen, mineral matter, moisture, nitrogen, total sulfur, pyritic and sulfate sulfur, and water of constitution. The derived result should not be compared strictly with a result from a direct determination of oxygen, and should be used only as a guide to the magnitude of organic oxygen in the sample. Because lower rank coals contain much of the ash-forming matter as inorganic ions, dmif corrections for carbon, hydrogen, nitrogen and sulfur have to be used to calculate 'oxygen by difference' (see Clause 7.3.5).

* KINSON, K. and BELCHER, C.B. 'Determination of oxygen in coal and coke using a radio-frequency heating method.' *Fuel*, 1975, 54, 205–209.

Oxygen content of coke is usually between 0.4% and 0.8% on a dmmf basis, at which level the calculated ‘oxygen by difference’ is of limited value.

7.3 Reporting analysis to different bases

7.3.1 Dry basis

Because the dry basis serves to eliminate the effect of moisture variations from time to time within a laboratory, as well as between laboratories, it enables valid comparison of results from analysis of samples of coal and coke.

$$\text{Example: } A_d = A_{ad} \frac{100}{100 - M_{ad}}$$

The factors of Table 1 refer to results expressed on a ‘by mass’ basis and are not, therefore, suitable for converting relative density results to the dry basis. Such conversion is achieved by using the following equation derived in Appendix A:

$$RD_d = \frac{RD_{ad} (1 - 0.01 M_{ad})}{1 - (RD_{ad} \times 0.01 M_{ad})} \quad \dots 7(10)$$

NOTE: See Appendix A, Paragraph A10 for the derivation of this equation.

7.3.2 Dry, ash-free basis

The dry, ash-free basis provides a useful approximation to the dmmf basis for comparison of organic components in coals having low ash values. The accuracy of this approximation becomes increasingly degraded, however, as mineral matter content or proportion of readily decomposed minerals increases. If results are reported on this basis, it needs to be indicated, by use of the appropriate subscript, whether or not corrections for inorganic components have been made.

$$\text{Example: } VM_{o,daf} = VM_{o,ad} \frac{100}{100 - M_{ad} + A_{ad}}$$

The reporting of a volatile matter result exemplifies the anomalies that can be present with the use of the dry, ash-free basis. For example, VM_{daf} is, by convention, reported according to the following equation:

$$VM_{daf} = (VM_{ad} - 3.66 C_{m,ad}) \frac{100}{100 - (M_{ad} + A_{ad})} \quad \dots 7(11)$$

without incorporation of correction factors for S_p and W_{ad} , which also are volatilized during the preparation of ash. Similarly, ambiguity will result if S_{daf} is reported, without consideration of S_p and S_s , but there is no convention available for such a quantity.

7.3.3 Dry, mineral matter-free basis

The dry, mineral matter-free basis is the correct basis upon which to compare results for the organic components of higher rank coal and coke. It is imperative, therefore, for any quantity calculated to this basis to be corrected, as necessary, to represent organic matter only.

$$\text{Example: } C_{o,dmmf} = C_{o,ad} \frac{100}{100 - (M_{ad} + MM_{ad})}$$

Equations are available for the estimation of mineral matter in higher rank coal when a result from the direct determination according to AS 1038.22 has not been carried out.

Where a factor has been used for calculation of mineral matter or water of constitution or both, these and any analytical results expressed on a dry, mineral matter-free basis have to be qualified by the subscript 'est'. The equation used should be stated, and such a result should not be published as complying with an Australian Standard.

Factors presented in BS 1016.100 for this purpose have been shown to be inappropriate for Australian coals.* It is recommended that the following equation be used:

$$MM_{ad} = bA_{ad} \quad \dots 7(12)$$

Where insufficient information on the value of b has been accumulated to provide optimal accuracy to the estimation, a value of 1.15 may be used to achieve an approximate result. However, factors ranging between 1.05 and 1.25 are applicable to various coal sources. This variability is primarily due to differences in the water of constitution, carbonates, pyrite, volatile matter, minor inorganic components, and susceptibility to sulfur oxide fixation by basic components as the coal is ashed.

Water of constitution is predominantly associated with aluminosilicate minerals in coals, such as the various clays, and is important for corrections to other analysed values before conversion to a dmmf basis. It is determined directly by the procedures of AS 1038.22, as an intrinsic component of mineral matter. In similar fashion to the estimation of mineral matter, water of constitution may be estimated also by use of an equation as follows:

$$W_{ad} = cA_{ad} \quad \dots 7(13)$$

where c will generally be a function of the silica/alumina ratio for a particular coal source. In the absence of sufficient information about the value of c , 0.1 may be used to achieve an approximate result.

Finally, a mineral matter value may be estimated by use of the following equation:

$$MM_{ad,est} = A_{ad} + W_{ad} + 3.3C_{m,ad} + 0.62S_{p,ad} \quad \dots 7(14)$$

Cokes are generally resistant to the complete removal of the organic components by low temperature oxidation procedures, and no method has been developed for direct determination of mineral matter in coke. It is assumed that mineral matter and ash percentages are equivalent. Water of constitution is volatilized during the coking process, and is considered to be absent from mineral matter of coke.

7.3.4 Dry, organic matter-free basis

The dry, organic matter-free basis is applicable only to the expression or comparison of components that are known to be completely contained within the mineral matter of coal or coke.

Example: $P_{domf} = P_{ad} \frac{100}{MM_{ad}}$

7.3.5 Dry, minerals and inorganics-free basis

The dry, minerals and inorganics-free basis is relevant only to lower rank coals in which some inorganic elements, including aluminium, calcium, iron, magnesium and sodium, associated with carboxylate groups of the coal or dissolved in the water phase, are present. Allowance has to be made for this in calculating results to what is otherwise equivalent to a

* BROWN, H.R., DURIE, R.A. and SCHAFER, H.N.S. 'The inorganic constituents in Australian coals. I—The direct determination of total mineral-matter content.' *Fuel*, 1959, 38, 295–308; Ibid. 'The inorganic constituents in Australian coals. II—Combined acid-digestion-low-temperature oxidation procedure for determination of total mineral-matter content, water of hydration of silicate minerals and composition of carbonate minerals.' *Fuel*, 1960, 39, 59–70; BROWN, N.A., BELCHER, C.B. and CALLCOTT, T.G. 'Mineral matter in N.S.W. coke-making coals; composition, determination and effects.' *J. Inst. Fuel*, 1965 38, 198–206.

dmmf basis. This basis refers to a hypothetical condition in which lower rank coal is assumed to be free of moisture, inorganic ions and mineral matter.

In contrast to higher rank coals, where most of the ash comes from minerals present in the coal, many of the ash-forming minerals present in lower rank Australian (particularly Latrobe Valley) coals occur as inorganic ions bound to oxygen functional groups of the coal matrix and soluble in the water phase. These ions (termed inorganics) include sodium, magnesium, calcium, iron and an aluminium hydroxo complex.

Sodium chloride may also be present in the water phase. Inorganics are clearly distinguished from particulate or crystalline minerals which may be present also in the coal, usually at a much lower concentration. Minerals comprise oxides, e.g. of silicon or aluminium, as in kaolinite, but also include minerals such as siderite and pyrite.

Hence,

Minerals \equiv $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{TiO}_2 + \text{K}_2\text{O} + \text{FeS}_2$

and Inorganics \equiv $\text{Na} + \text{Mg} + \text{Ca} + \text{Fe} + (\text{Al}) + \text{NaCl}$

MI is the sum of the minerals and inorganics, expressed on a dry basis*.

Before conversion of determined results to the dry, minerals and inorganics-free basis, appropriate corrections should be applied (see Clause 7.2) so that only organic components are included. Analyses may be calculated to the dmif basis by using appropriate factors shown in Table 1.

$$\text{Example: } C_{\text{o,dmif}} = C_{\text{o,d}} \frac{100}{100 - MI_{\text{d}}}$$

Without taking account of the concept of organically associated elements and of the presence of sodium chloride as distinct from organo-chlorine species, an ash determination will overestimate the actual non-organic components, owing to a mass increase resulting from conversion of the elements to sulfates and oxides during the ashing procedure. The effect on an organic carbon result may be seen from the example of a coal reporting as 68.6% on a daf basis but only 67.2% on a dmif basis; the values for *A* and *MI* were 5.5% and 3.6% respectively.

8 REPORTING OF TEST RESULT

8.1 General

A test result should be reported in the manner required by the test method.

8.2 Number of decimal places

The number of decimal places to which the test result is rounded should be stipulated in each Standard. The test result should be rounded in accordance with AS 2706.

NOTE: Table C1 indicates the number of decimal places at which the test result should be reported.

8.3 Test report

The test report should contain the following information:

- (a) Full name and location of the testing laboratory.
- (b) Unique identification of the sample and test report.
- (c) Reference to the sample's history, e.g. sampled by . . ., or 'as received'.

* KISS, L.T. and KING, T.N. 'The expression of results of coal analysis: the case for brown coals.' *Fuel*, 1977, 56, 340--341; Ibid. 'Reporting of low rank coal analysis: the distinction between minerals and inorganics. *Fuel*, 1979, 58, 547-548.

- (d) Date of receipt of the sample.
- (e) Date of test report.
- (f) Reference to the test method used.
- (g) The analysis basis on which the result is reported.
- (h) Signature of the responsible officer.
- (i) Any other important information that will assist the recipient of the report in the interpretation of the test results (e.g. recovery data), test method (if given).

TABLE 1
FACTORS FOR CALCULATION OF RESULTS TO DIFFERENT BASES

Given basis	Desired basis							
	As sampled (as received) (as despatched) (as fired)	Air-dry	Dry	Dry, ash-free	Dry, mineral matter-free	Ash-free, moist	Dry, organic matter-free	Dry, minerals and inorganics- free
As sampled (as received) (as despatched) (as fired)	—	$\frac{100 - M_{ad}}{100 - M_{as}}$	$\frac{100}{100 - M_{as}}$	$\frac{100}{100 - (M_{as} + A_{as})}$	$\frac{100}{100 - (M_{as} + MM_{as})}$	$\frac{100 - MHC}{100 - (M_{as} + A_{as})}$	$\frac{100}{MM_{as}}$	$\frac{100}{100 - (M_{as} + MI_{as})}$
Air-dry	$\frac{100 - M_{as}}{100 - M_{ad}}$	—	$\frac{100}{100 - M_{ad}}$	$\frac{100}{100 - (M_{ad} + A_{ad})}$	$\frac{100}{100 - (M_{ad} + MM_{ad})}$	$\frac{100 - MHC}{100 - (M_{ad} + A_{ad})}$	$\frac{100}{MM_{ad}}$	$\frac{100}{100 - (M_{ad} + MI_{ad})}$
Dry	$\frac{100 - M_{as}}{100}$	$\frac{100 - M_{ad}}{100}$	—	$\frac{100}{100 - A_d}$	$\frac{100}{100 - MM_d}$	$\frac{100 - MHC}{100 - A_d}$	$\frac{100}{MM_d}$	$\frac{100}{100 - MI_d}$
Dry, ash-free	$\frac{100 - (M_{as} + A_{as})}{100}$	$\frac{100 - M_{ad} + A_{ad}}{100}$	$\frac{100 - A_d}{100}$	—	$\frac{100 - A_d}{100 - MM_d}$	$\frac{100 - MHC}{100}$	$\frac{100 - A_d}{MM_d}$	$\frac{100 - A_d}{100 - MI_d}$
Dry, mineral matter-free	$\frac{100 - (M_{as} + MM_{as})}{100}$	$\frac{100 - (M_{ad} + MM_{ad})}{100}$	$\frac{100 - MM_d}{100}$	$\frac{100 - MM_d}{100 - A_d}$	—	—	$\frac{100 - MM_d}{MM_d}$	—
Ash-free, moist	$\frac{100 - (M_{as} + A_{as})}{100 - MHC}$	$\frac{100 - (M_{ad} + A_{ad})}{100 - MHC}$	$\frac{100 - A_{ad}}{100 - MHC}$	$\frac{100}{100 - MHC}$	—	—	—	—
Dry, organic matter-free	$\frac{MM_{as}}{100}$	$\frac{MM_{ad}}{100}$	$\frac{MM_d}{100}$	$\frac{MM_d}{100 - A_d}$	$\frac{MM_d}{100 - MM_d}$	—	—	$\frac{MI_d}{100 - MI_d}$
Dry, minerals and inorganics- free	$\frac{100 - (M_{as} + MI_{as})}{100}$	$\frac{100 - (M_{ad} + MI_{ad})}{100}$	$\frac{100 - MI_d}{100}$	$\frac{100 - MI_d}{100 - A_d}$	—	—	$\frac{100 - MI_d}{MM_d}$	—

NOTE: Some conversions may require corrections prior to application of the above factors (see Clause 7.2).

APPENDIX A

DERIVATION OF EQUATIONS USED FOR METHOD CALCULATIONS

(Informative)

A1 GENERAL

Equations used to calculate results in the AS 1038 and AS 2434 series of Standards are derived below. For the purposes of this Appendix, atomic or molecular weights are represented by symbols contained within square brackets.

A2 TOTAL MOISTURE (AS 1038.1, AS 1038.2 and AS 2434.1)

m mass of sample used, as-sampled basis, in grams

M_f free moisture; percentage loss in achieving air-dry condition

M_{as} percentage of total moisture in coal as sampled

M_r percentage of moisture in air-dry sample

m_1 mass of free moisture = $\frac{mM_f}{100}$

m_2 mass of air-dry sample = $m - m_1$

m_3 mass of moisture in air-dry sample = $m_2 \frac{M_r}{100}$

m_4 mass of total moisture = $m_1 + m_3$

$M_{as} = \frac{100}{m} m_4$

$= M_f + M_r \left(1 - \frac{M_f}{100} \right)$

A3 CARBON AND HYDROGEN (AS 1038.6.1 and AS 2434.6)

NOTE: For lower rank coals, carbon and hydrogen are usually determined on a dry basis (rather than air-dry). The equations for calculating carbon and hydrogen on a dry basis are given in AS 2434.6.

A3.1 Carbon

m_1 mass of air-dry sample used, in grams

m_3 increase in mass of soda-asbestos tube for sample (grams of carbon dioxide formed)

m_5 increase in mass of soda-asbestos tube for blank

$$\frac{[C]}{[CO_2]} = \frac{12.011}{44.010} = 0.2729$$

$$C_{ad} = \frac{[C]}{[CO_2]} \times \frac{m_3 - m_5}{m_1} \times 100$$

$$= \frac{27.29(m_3 - m_5)}{m_1}$$

A3.2 Hydrogen

m_1 mass of air-dry sample used, in grams

m_2 increase in mass of magnesium perchlorate tube for sample (grams of water formed)

m_4 increase in mass of magnesium perchlorate tube for blank

M_{ad} percentage of moisture in air-dry sample

$$\frac{[H_2]}{[H_2O]} = \frac{2.016}{18.015} = 0.1119$$

$$\begin{aligned} H_{ad} &= \left[\frac{[H_2]}{[H_2O]} \times \frac{m_2 - m_4}{m_1} \times 100 \right] - \left[\frac{[H_2]}{[H_2O]} \times M_{ad} \right] \\ &= 11.19 \left[\frac{m_2 - m_4}{m_1} - 0.01 M_{ad} \right] \end{aligned}$$

A4 NITROGEN (AS 1038.6.2 and AS 2434.6)

m mass of air-dry sample used, in grams

V_1 volume of 0.005 mol/L sulfuric acid used in sample determination, in millimetres

V_2 volume of 0.005 mol/L sulfuric acid used in blank determination, in millimetres

mol(N) moles nitrogen in 1 mL of 0.005 mol/L sulfuric acid

$$= (\text{moles equivalent to mole acid}) \times \text{molarity} \times (\text{mL}/1000)$$

$$= 2 \times 0.005 \times 0.001 = 0.00001$$

$m(N)$ mass of nitrogen per millilitre of 0.005 mol/L sulfuric acid, in grams

$$= [N] \times \text{mol(N)} = 0.00014 \text{ g}$$

$$\begin{aligned} N_{ad} &= m(N) \times (V_1 - V_2) \times \frac{100}{m} \\ &= \frac{0.014(V_1 - V_2)}{m} \end{aligned}$$

A5 TOTAL SULFUR (ESCHKA METHOD) (AS 1038.6.3.1, and AS 2434.6)

m mass of air-dry sample used, in grams

m_1 mass of barium sulfate precipitate in sample determination, in grams

m_2 mass of barium sulfate precipitate in blank determination, in grams

m_3 mass per litre of added potassium sulfate solution, in grams

25 mL of potassium sulfate solution

$$= \left[\frac{BaSO_4}{K_2SO} \times \frac{25 m_3}{1000} \right]$$

$$= \frac{233.39}{174.25} \times 0.025 \times m_3$$

$$= 0.03348 m_3 \text{ grams of barium sulfate}$$

$$S_{\text{ad}} = \frac{100}{m} \times \left(\left[\frac{[\text{S}]^-}{[\text{BaSO}_4]} \right] \times m_1 - \left[\frac{[\text{S}]^-}{[\text{BaSO}_4]} \right] \times (m_2 - 0.03348 m_3) \right)$$

$$= \frac{100}{m} \times \frac{32.06}{233.39} \times [m_1 - (m_2 - 0.03348 m_3)]$$

$$= \frac{13.74}{m} (m_1 - m_2 + 0.03348 m_3)$$

A6 TOTAL SULFUR OR CHLORINE (HIGH TEMPERATURE COMBUSTION/TITRATION METHOD) (AS 1038.6.3.2, AS 1038.8.2 and AS 2434.6)

m mass of air-dry sample used, in grams

V_1 volume of 0.025 mol/L sodium borate used in sample determination, in millimetres

V_2 volume of 0.025 mol/L sodium borate used in blank determination, in millimetres

V_3 volume of 0.0125 mol/L sulfuric acid used in sample determination, in millimetres

V_4 volume of 0.0125 mol/L sulfuric acid used in blank determination, in millimetres

Chemical reactions upon which the method is based assume quantitative liberation of sulfur and chlorine from samples so that they will yield sulfuric and hydrochloric acids in solution:



mol(S) moles sulfur per millilitre of 0.025 mol/L sodium borate

= (moles equivalent to mole sodium borate)

× molarity × (mL/1000)

= $1 \times 0.025 \times 0.001 = 0.000025$

$m(\text{S})$ mass of sulfur per millilitre of 0.025 mol/L sodium borate

= $[\text{S}] \times \text{mol(S)}$

= $32.06 \times 0.000025 = 0.0008015 \text{ g}$

Since 1 mL of 0.0125 mol/L sulfuric acid is equivalent to 0.5 mL of 0.025 mol/L sodium borate then,

V volume of 0.025 mol/L sodium borate required to neutralize the sulfuric acid produced from the sample corrected for the hydrochloric acid produced, in millilitres

= $(V_1 - V_2) - 0.5(V_3 - V_4)$

$$S_{\text{ad}} = m(\text{S}) \times V \times \frac{100}{m}$$

$$= \frac{0.08015V}{m}$$

mol(Cl) moles chlorine per millilitre of 0.0125 mol/L sulfuric acid
 = (moles equivalent to mole acid) \times molarity \times (mL/1000)
 = $2 \times 0.0125 \times 0.001 = 0.000025$

m(Cl) mass of chlorine per millilitre of 0.0125 mol/L sulfuric acid
 = [Cl] \times mol (Cl)
 = $35.45 \times 0.000025 = 0.0008862$ g

$$\begin{aligned} Cl_{\text{ad}} &= m(\text{Cl}) \times (V_3 - V_4) \times \frac{100}{m} \\ &= \frac{0.08862 (V_3 - V_4)}{m} \end{aligned}$$

A7 CHLORINE (ESCHKA METHOD) (AS 1038.8.1)

m mass of air-dry sample used, in grams

V_1 volume of 0.025 mol/L potassium thiocyanate used in sample determination, in millilitres

V_2 volume of 0.025 mol/L potassium thiocyanate used in blank determination, in millilitres

V_3 volume of 0.025 mol/L silver nitrate added, in millilitres
 = 20.0 mL



mol(Cl) moles chlorine per millilitre of 0.025 mol/L silver nitrate
 = (moles equivalent to mole silver) \times molarity \times (mL/1000)
 = $1 \times 0.025 \times 0.001 = 0.000025$

m(Cl) mass of chlorine per millilitre of 0.025 mol/L silver nitrate, in grams
 = [Cl] \times mol (Cl)
 = $35.45 \times 0.000025 = 0.0008862$ g

Cl equiv in sample = $[(V_3 - V_1) - (V_3 - V_2)]$ mL
 0.025 mol/L silver nitrate
 = $(V_2 - V_1)$ mL of 0.025 mol/L silver nitrate

$$\begin{aligned} Cl_{\text{ad}} &= m(\text{Cl}) \times [\text{Cl equiv}] \times \frac{100}{m} \\ &= \frac{0.08862 (V_2 - V_1)}{m} \end{aligned}$$

A8 PHOSPHORUS**A8.1 Ash digestion/molybdenum blue method (AS 1038.9.1)**

- m mass of sample used, in grams
 A_1 absorbance of test solution
 A_2 absorbance of blank test solution
 A_3 absorbance of calibration solution
 A_4 absorbance of reagent blank solution
 V volume of sample solution taken for colour development, in millilitres

$$P_{\text{ad}} = \frac{A_1 - A_2}{10 \times V \times m (A_3 - A_4)}$$

A8.2 Coal extraction method (AS 1038.9.2)

- m mass of sample used, in grams
 A_1 absorbance of test solution
 A_2 absorbance of blank test solution
 A_3 absorbance of calibration solution
 A_4 absorbance of blank calibration solution
 V volume of test solution taken for colour development, in millilitres

$$P_{\text{ad}} = \frac{A_1 - A_2}{Vm (A_3 - A_4)}$$

A8.3 Ash digestion method (AS 1038.9.3)

- m mass of sample used, in grams
 m_p Mass of phosphorus in the test solution, in milligrams
 V volume of test solution taken, in millilitres

$$P_{\text{ad}} = \frac{m_p}{m \times V}$$

A8.4 Phosphorus in ash (AS 1038.14.1 and AS 1038.14.2)

$$P_2O_5 (\text{ash})_d = P_d \frac{100}{A_d} \times 2.291$$

where

- $P_2O_5 (\text{ash})_d$ = phosphorus in ash (dry basis) as $P_2O_5\%$
 P_d = phosphorus in coal (dry basis) as $P\%$
 A_d = ash in coal (dry basis) %
2.291 = Conversion factor for P to P_2O_5

A9 FORMS OF SULFUR (AS 1038.11)**A9.1 Sulfate sulfur**

- m mass of air-dry sample used, in grams
 m_1 mass of barium sulfate precipitate in sample determination, in grams

m_2 mass of barium sulfate precipitate in blank determination, in grams

$$\begin{aligned} S_{s,ad} &= \frac{[S]}{\text{BaSO}_4} \times (m_1 - m_2) \times \frac{100}{m} \\ &= \frac{13.74 (m_1 - m_2)}{m} \end{aligned}$$

A9.2 Pyritic sulfur

A9.2.1 General

There are three measurement techniques in the method, all of which are based on determining the iron component of pyrite (FeS_2), from which the associated pyritic sulfur may be calculated.

$$\frac{[S_2]}{[Fe]} = \frac{2 \times 32.06}{55.85} = 1.148$$

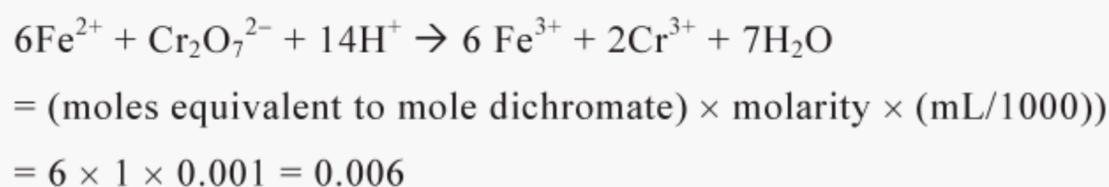
A9.2.2 Titrimetric

m mass of air-dry sample used, in grams

m_1 mass of potassium dichromate per litre in solution, in grams

V volume of m_1 g/L potassium dichromate (blank corrected) used in sample determination, in millilitres

mol(Fe) moles of iron per millilitre of 1 mol/L potassium dichromate based on:



m(Fe) mass of iron per millilitre of m_1 g/L potassium dichromate, in grams

$$\begin{aligned} &= [Fe] \times \frac{\text{mol(Fe)}}{[\text{K}_2\text{Cr}_2\text{O}_7]} \times m_1 \\ &= 55.85 \times \left[\frac{0.006}{294.19} \right] \times m_1 = 0.001139 m_1 g \end{aligned}$$

$$\begin{aligned} S_{p,ad} &= \frac{[S_2]}{[Fe]} \times V \times m(\text{Fe}) \times \frac{100}{m} \\ &= 1.148 \times V \times 0.001139 \times m_1 \frac{100}{m} \\ &= \frac{0.1308 V m_1}{m} \end{aligned}$$

A9.2.3 Spectrophotometric

m mass of air-dry sample used, in grams

V volume of aliquot for colour development, taken from original 250 mL sample solution, in millilitres

m_1 mass of iron in aliquot V , in micrograms

$$S_{p,ad} = \frac{[S_2]}{[Fe]} \times \frac{250}{V} \times \frac{m_1}{10^6} \times \frac{100}{m}$$

$$= \frac{0.02870 m_1}{mV}$$

A9.2.4 Atomic absorption spectrometric

m mass of air-dry sample used, in grams

m_1 mass of iron per millilitre of sample solution made to 250 mL, in micrograms

$$S_{p,ad} = \frac{[S_2]}{[Fe]} \times 250 \times \frac{m_1}{10^6} \times \frac{100}{m}$$

$$= \frac{0.02870 m_1}{m}$$

A10 RELATIVE DENSITY (AS 1038.21.1.1 and AS 1038.21.1.2)

Although not intrinsic to AS 1038.21.1, the conversion of air-dry sample relative densities to a dry basis for special comparison may be achieved through the derivation below. The factor of Table 1 cannot be used.

m mass of air-dry sample taken, in grams

V volume occupied by mass m of sample, in millilitres

Then

$$\text{mass of dry sample} = m(1 - 0.01M_{ad})$$

$$\text{volume of dry sample} = V - (m \times 0.01 M_{ad})$$

(taking density of water as 1)

$$RD_{db} = \frac{m(1 - 0.01 M_{as})}{V - (m \times 0.01 M_{ad})}$$

$$= \frac{m \times (1 - 0.01 M_{as})}{V \left(1 - \frac{m}{V} \times 0.01 M_{ad}\right)}$$

$$= \frac{RD_{ad} (1 - 0.01 M_{as})}{1 - RD_{ad} \times 0.01 M_{ad}}$$

A11 CARBONATE CARBON (AS 1038.23)

m mass of air-dry sample taken, in grams

m_1 increase in mass of absorption tube, blank corrected, in grams

$$C_{m,ad} = \frac{[C]}{[CO_2]} \times m_1 \times \frac{100}{m}$$

$$= \frac{27.3 m_1}{m}$$

APPENDIX B

SUGGESTED HIERARCHY OF PARAMETERS FOR A TEST REPORT

(Informative)

Proximate and general analysis	Total moisture	
	Moisture	
	Ash	
	Volatile matter	
	Fixed carbon	
	Total sulfur	
	Forms of sulfur (pyritic, sulfate, organic)	
	Chlorine	
	Phosphorus	
	Carbonate carbon	
	Calorific value	
	Ultimate analysis	Carbon*
		Hydrogen
Nitrogen		
Sulfur*		
Oxygen		
Mineral matter	Mineral matter	
Caking and coking properties	Crucible swelling number	
	Gieseler plasticity	
	Dilatation	
	Gray-King coke type	
	Coke strength	
	Coke reactivity	
	Petrographics	Reflectance
Maceral analysis		
Physical properties	Moisture-holding capacity	
	Relative density	
	Abrasion index	
	Hardgrove grindability index	
	Size distribution	
	Float-and-sink analysis	
	Durham cone handleability	

* Organic carbon and organic sulfur are sometimes used, but should be qualified as such.

Ash analysis

Silicon as SiO₂
Aluminium as Al₂O₃
Iron as Fe₂O₃
Calcium as CaO
Magnesium as MgO
Sodium as Na₂O
Potassium as K₂O
Titanium as TiO₂
Manganese as Mn₃O₄
Phosphorus as P₂O₅
Sulfur as SO₃
Barium as BaO
Strontium as SrO
Zinc as ZnO

Trace elements

Arsenic (As)
Boron (B)
Barium (Ba)
Beryllium (Be)
Cadmium (Cd)
Cobalt (Co)
Chromium (Cr)
Copper (Cu)
Fluorine (F)
Mercury (Hg)
Lithium (Li)
Manganese (Mn)
Molybdenum (Mo)
Nickel (Ni)
Lead (Pb)
Antimony (Sb)
Selenium (Se)
Tin (Sn)
Strontium (Sr)
Thorium (Th)
Thallium (Tl)
Uranium (U)
Vanadium (V)
Zinc (Zn)

Ash fusibility (reducing and oxidizing)

Deformation temperature

Sphere temperature

Hemisphere temperature

Flow temperature

APPENDIX C
PRECISION OF TEST METHODS AND SCHEDULE
FOR REPORTING OF RESULTS

(Informative)

Precision statistics for analysis methods presented in this Standard are detailed in Table C1. Reference should be made to Clauses 5 and 6 for explanation of their use.

Reference should be made to the latest edition of the relevant Standard to verify the data in Table C1.

The procedure adopted to establish precision statistics for presentation in Table C1 follows general recommendations of AS 2850. Reference should be made to that Standard for additional information on statistical methods used.

Table C1 also contains a schedule for the reporting of test results.

TABLE C1
REPEATABILITY AND REPRODUCIBILITY OF TEST METHODS

Australian Standard	Corresponding ISO Standard	Material	Determination	<i>r</i>	<i>R</i>	See Note I	Report to nearest
AS 1038.1	589	coal	M_{as} total moisture %	0.5	1.5	A	0.1
AS 1038.2	579	coke	M_{as} total moisture %	0.5	0.7	A	0.1
AS 1038.3	331	coal	M_{ad} analysis moisture %	0.1	—	A	0.1
				0.15	—	A	0.1
			A_{ad} ash %	0.10	0.15	A	0.1
				0.15	0.25	A	0.1
				0.20	0.60	A	0.1
AS 1038.4	562	coke	VM_{ad} volatile matter %	0.2	0.5	A	0.1
				0.2	1.0	A	0.1
AS 1038.5	1928	coal, coke	M_{as} analysis moisture %	0.10	—	A	0.1
			A_{as} ash %	0.15	0.30	A	0.1
				0.20	0.40	A	0.1
AS 1038.6.1	609	coal	VM_{as} volatile matter %	0.20	—	A	0.1
				0.13	0.30	A	0.01
AS 1038.6.2	334	coal, coke	$q_{gr,v,ad}$ gross calorific value MJ/kg (gross specific energy)	0.13	—	A	0.01
				0.05	0.10	A	0.01
AS 1038.6.3.1	351	coal, coke	C_{ad} carbon (total) %	0.3	0.6	A	0.1
			H_{ad} hydrogen %	0.10	0.20	A	0.01
AS 1038.6.3.2	351	coal, coke	N_{ad} nitrogen %	0.03	0.08	A	0.01
			S_{ad} sulfur (total) % (Eschka)	0.05	0.10	A	0.01
AS 1038.6.3.3	587	coal, coke	S_{ad} sulfur (total) % (high temperature combustion)	0.10	0.20	A	0.01
				0.03	0.08	A	0.01
AS 1038.8.1	352	coal, coke	S_{ad} sulfur (total) % (Infrared)	2%	10%	A	0.01
				0.03	0.05	A	0.01
AS 1038.8.2	352	coal, coke	Cl_{ad} chlorine (Eschka) %	2%	8%	A	0.01
			Cl_{ad} chlorine (high temperature combustion) %	0.01	0.02	A	0.01
				0.01	0.02	A	0.01

(continued)

TABLE C1 (continued)

Australian Standard	Corresponding ISO Standard	Material	Determination	<i>r</i>	<i>R</i>	See Note 1	Report to nearest
AS 1038.10.2	601	coal, coke	<i>A</i> _{s,ad} arsenic mg/kg* <i>S</i> _{b,ad} antimony mg/kg* <i>S</i> _{e,ad} selenium mg/kg*	0.1 10% 0.1 10% 0.1 10%	0.2 20% 0.2 20% 0.2 20%	A A A A A A	0.1
AS 1038.10.3		coal, coke	boron mg/kg*	5 10%	10 20%	A A	1
AS 1038.10.4		ash, coal, coke	fluorine µg/g*	10 20	20 15%	A A	<100 1 ≥100 < 500 5 ≥500 10
AS 1038.10.5.1	15237	ash, coal, coke	mercury µg/g*	0.01 10%	0.02 15%	A A	0.01
AS 1038.11	157	coal	<i>S</i> _{s,ad} sulfate sulfur % <i>S</i> _{p,ad} pyritic sulfur %	0.02 0.05 0.07	0.03 0.10 0.15	A A A	0.01
AS 1038.12.1	501	coal	<i>CSN</i> crucible swelling number 3 determinations 5 determinations		1 1	A A	½
AS 1038.12.2	502	coal	Gray-King coke type—	one letter, or one unit in the subscript	one letter, or one unit in the subscript	A	N/A
AS 1038.12.3	8264	coal	dilatometer characteristics: <i>T</i> ₁ , <i>T</i> ₂ , <i>T</i> ₃ temperature °C <i>c</i> max. contraction % <i>d</i> max. dilatation, negative % positive %	7 5 5 5[1+(d/100)]	15 8 8 5[2+(d/100)]	A A A A	5 See Standard

* mg/kg, µg/kg and ppm are all equivalent terms, but mg/kg is the preferred unit.

(continued)

TABLE C1 (continued)

Australian Standard	Corresponding ISO Standard	Material	Determination	r	R	See Note 1	Report to nearest
AS 1038.13		coke	CSR coke strength after reaction %	2.5 5.0	—		
AS 1038.14.1	1171	ash	Ash analysis (fusion/AAS)			A	See Standard
			SiO ₂ %	1.4	2.5		
			Al ₂ O ₃ %	0.6	0.7		
			Fe ₂ O ₃	0.8	1.5		
			CaO%	0.3	0.5		
			MgO%	0.05	0.08		
			Na ₂ O%	0.15	0.25		
			K ₂ O%	0.05	0.1		
			TiO ₂ %	0.05	0.2		
			Mn ₃ O ₄ %	0.05	0.06		
			P ₂ O ₅	0.15	0.3		
			SO ₃ %	0.08	0.12		
			BaO%	10%	15%		
			SrO%	0.08	0.20		
			ZnO%	2%	10%		
AS 1038.14.2		ash	Ash analysis (acid digestion/AAS)				See Standard
			SiO ₂ %	1.30	1.76	A	
			Al ₂ O ₃ %	1.49	1.76		

(continued)

TABLE C1 (continued)

Australian Standard	Corresponding ISO Standard	Material	Determination	r	R	See Note 1	Report to nearest
AS 1038.14.2		ash	Fe ₂ O ₃ %	1 to 10	0.233	0.393	
			CaO%	0.1 to 5	0.077	0.146	
			MgO%	0.1 to 1	0.053	0.093	
			Na ₂ O%	0.1 to 1	0.058	0.066	
			K ₂ O%	1 to 5	0.178	0.309	
			TiO ₂ %	0.5 to 1.5	0.106	0.141	
			Mn ₃ O ₄ %	0.01 to 0.1	0.017	0.021	
			P ₂ O ₃ %	0.01 to 2	0.053	0.077	
			SO ₃ %	< 4	0.08	0.20	
				4.0 to 10.0	2%	10%	
			AS 1038.14.3		ash	Ash analysis (XRF)	
SiO ₂ %	45 to 70	0.42				1.44	
Al ₂ O ₃ %	20 to 35	0.25				1.01	
Fe ₂ O ₃ %	1.5 to 13	0.007X + 0.035				0.027X + 0.063	*
CaO%	0.5 to 3.5	0.035				0.089	
MgO%	1.0 to 2.0	0.073				0.13	
Na ₂ O%	0.1 to 1.0	0.063				0.11	
K ₂ O%	0.5 to 2.0	0.012X + 0.009				0.062X + 0.016	*
TiO ₂ %	1.0 to 2.5	0.037				0.10	
Mn ₃ O ₄ %	0.02 to 0.25	0.010				0.017	
P ₂ O ₅ %	0.05 to 1.0	0.022X + 0.010				0.078X + 0.014	*
SO ₃ %	0.5 to 1.5	0.049X + 0.001				0.16	*
BaO%	0.04 to 0.2	0.021				0.043	
							See Standard

(continued)

TABLE C1 (continued)

Australian Standard	Corresponding ISO Standard	Material	Determination	<i>r</i>	<i>R</i>	See Note I	Report to nearest
AS 1038.14.3		ash	SrO% ZnO%	0.004 0.006	0.195 0.011		
AS 1038.15	540	ash	Ash fusion temperature °C deformation sphere hemisphere flow	30 50 30 30 40	80 150 60 60 80	A	10
AS 1038.17	1018	coal	MHC Moisture-holding capacity %	0.6	1.2	A	0.1
AS 1038.18	1953	coke	Size analysis	2.5 0.60 0.12 0.06	— — — —	B	0.1
AS 1038.19	12900	coal	AI Abrasion index	2 10%	— —	C	1
AS 1038.20	5074	coal	HGI Hardgrove grindability	2	5	C	1
AS 1038.21.1.1		coal, coke	RD Relative density—Analysis sample/density bottle	0.03 0.04	0.08 0.08	A	0.01
AS 1038.21.1.2		coal, coke	RD Relative density—Analysis sample/volumetric	0.03 0.04	0.10 0.12	A	0.01

(continued)

TABLE C1 (continued)

Australian Standard	Corresponding ISO Standard	Material	Determination	r	R	See Note I	Report to nearest
AS 1038.22	602	coal	MM_{ad} mineral matter %: radio-frequency ashing	<10 ≥10 to ≤20 >20	0.25 0.70 0.70	A	0.1
			air oxidation	<10 ≥10 to ≤20	0.30 0.70		0.1
			W_{ad} water of constitution %: radio-frequency ashing	<2 ≥2	0.20 0.40	A	0.1
			air oxidation	<2 ≥2	0.30 0.65		
AS 1038.23	925	coal	$C_{m,ad}$ carbonate carbon %	0.01	0.02	A	0.01
AS 1038.25		coal	F_s Handleability s/kg	<1 ≥1	10% 20%		0.1 1
AS 2434.1	5068	Lower rank coal	M_{as} Moisture (total) %	0.5	—	A	0.1
AS 2434.2	5071-1	Lower rank coal	VM_{ad} Volatile matter %	0.7	2.0	A	0.1
AS 2434.3		Lower rank coal	MHC Moisture holding capacity of lower rank coal %	0.5	1.2	A	0.5
AS 2434.4		Lower rank coal and char	Apparent density kg/m ³	14	—	A	1
AS 2434.5		Lower rank coal char	M_{ad} Moisture in bulk samples and in analysis samples of lower rank coal char %	0.30 0.50	— —		0.1

(continued)

TABLE C1 (continued)

Australian Standard	Corresponding ISO Standard	Material	Determination	<i>r</i>	<i>R</i>	See Note 1	Report to nearest
AS 2434.6		brown coal	Ultimate analysis of lower rank coal— Classical methods <i>C</i> _{ad} carbon % <i>H</i> _{ad} hydrogen % <i>N</i> _{ad} nitrogen % <i>S</i> _{ad} sulfur (total) Eschka % High temperature %	0.3 0.1 0.03 0.05 0.1 0.03 2%	0.6 0.2 0.08 0.1 0.2 0.08 10%	A	0.1 0.01 0.01 0.01 0.01 0.01
AS 2434.7		brown coal	<i>M</i> _{ad} Moisture in analysis sample %	<5 ≥5	— —	A	0.1
AS 2434.8		brown coal	Ash %	0.1	0.3	A	0.1
AS 2434.9		brown coal	Sodium, magnesium, calcium, iron %	0.025 5%	0.050 10%	A	0.01

* X in the equations = concentration of analyte

NOTES:

- Recent precision statistics from Australian interlaboratory test programs are designated by an 'A' in the final column, whereas those adapted from earlier versions of BS 1016-100 and which are due for revision are designated 'B'. Precision data obtained from International Standards are designated 'C'. The allocation of precision statistics is based primarily upon the results of the test program, but consideration is given also to results from recent NATA surveys as well as the corresponding ISO, BS and ASTM statistics where advisable.
- Strictly, results obtained under reproducibility conditions should be compared only on a dry basis.
- '—' denotes the unavailability of sufficient information, or that statistics are not applicable in this instance.
- '%' values quoted in precision columns are percentages relative to the mean result, not absolute percentages as is otherwise the case where this symbol is applicable.

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