

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

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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

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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.

A ash

AI abrasion index

c maximum contraction

C carbon (total, including carbon in minerals)

C_m carbonate carbon from minerals

C_o organic carbon

CRI coke reactivity index

CSR coke strength after reaction

CSN crucible swelling number

CV See *q*

FC fixed carbon ($100 - A - M - VM$)

F_s specific flow time

H hydrogen (total, excluding moisture content of the air-dry sample, but including hydrogen from water of hydration in minerals)

d 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).

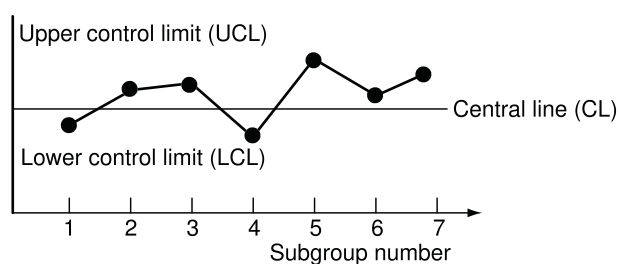


FIGURE 1 OUTLINE OF A CONTROL CHART

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

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

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

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: