

GENERAL COAL REFERENCE MATERIAL ACIRS-G8-2019

CERTIFICATION REPORT

Date of Issue: March, 2019
 Report Number: CR-G8-2019-rev01
 Previous ACIRS-G series: This is the sixth in the series and supersedes ACIRS-G6-2014-Lot#2

Production and certification of this sample was conducted in accordance with ISO 17034 and ISO Guide 35.

Table 1

ASSIGNED PROPERTY VALUES				
	Property Values¹	Standard Deviation²	Expanded Uncertainty³	Number of results
Ash, % d ^{1a}	10.14	0.08	0.03	50
Volatile Matter, % d ^{1b}	20.23	0.18	0.06	48
Gross Calorific Value, MJ/kg d	32.357	0.089	0.036	38
Relative Density, d	1.382	0.008	0.004	20
Total Carbon, % d	79.44	0.36	0.19	22
Hydrogen, % d	4.40	0.05	0.03	17
Nitrogen, % d	1.79	0.03	0.02	21
Total Sulfur, % d	0.590	0.013	0.005	41
Chlorine, % d	0.052	0.002	0.001	13
Phosphorus, % d	0.027	0.001	0.001	20
Mercury, mg/kg d	0.02	0.005	0.004	9
Fluorine, mg/kg d	85	5.9	3.8	15

1. Notes

1 Property values are the best estimate of the true value. They are based on the robust mean of technically valid results from proficiency testing using standard test methods where biases between methods were not observed. Methods used are detailed in Table 2.

2 Standard deviation (sd) is a robust value used to derive the likely range of results. For normally distributed data, the value for a measurand from a randomly chosen laboratory would be expected to lay within 2 standard deviations of the certified value with 95% probability.

3 The expanded uncertainty provides the user with information on the likely range of the true (but unknown) value for each parameter and has been estimated in accordance with the Guide to the Expression of Uncertainty in Measurement (GUM) with a coverage factor $k=2$, corresponding to a level of confidence of about 95%. For Hg, a k -factor of 2.2 was chosen according to the t -distribution for sets of results.

2. Description of the Sample and Preparation

ACIRS-G8-2019 comprises a sealed jar containing approximately 125 g of coal at a nominal top size of 212 μm . This sample was prepared from 550 kg of a Queensland, Bowen Basin higher rank bituminous coal at -50 mm top size.

The bulk coal sample was crushed in a swing hammer mill to a nominal top size of 2.36 mm and stabilised over an extended period. The material was then repeatedly mixed by rotary sample division (RSD) until lots of approximately 1.4 kg were obtained which were then air dried and milled to a nominal top size of 212 μm . This pulverised material was further divided by RSD until representative 125 (± 5) g samples were obtained. Each sample was then placed into a plastic bag within sealed HDPE jars.

Between-unit homogeneity was quantified by testing ash, calorific value, volatile matter and total sulfur and assessed in accordance with ISO Guide 35.

3. Instructions for Use

This reference material is intended to be used as a quality control tool.

Before the bottle is opened, it **must** be thoroughly mixed by end-over-end rotation so that the material is re-homogenised.

To minimise the risk of compositional changes due to oxidation store in a cool, dark place in original containers with lids tightly sealed. ACIRS cannot be held responsible for any changes that occur after the sample bottle has been opened.

The minimum sample intake for ACIRS-G8 is established in accordance with the standard test methods listed in Table 2.

4. Characterisation

With the exception of fluorine, ACIRS-G8 was characterised through Proficiency Testing Australia's (PTA) Round 38 proficiency testing program in which each participant was provided two samples for testing.

Fluorine was characterised by an independent test program conducted by ACIRS in which each participant was provided three samples to be tested by ISO 11724 and equivalent test methods.

Only technically valid data was included for characterisation. Generally, data was excluded when:

- Reported from in-house/unknown methods;
- Reported from laboratories not accredited to ISO/IEC 17025; or
- When results failed to meet technical acceptability along ACIRS guidelines e.g. failing to meet standard method precision limits or identified as outliers from the dataset.

Property values and their associated uncertainties were calculated by robust statistical techniques in accordance with the guidelines of:

- ISO/IEC Guide 98-3, Guide to the Expression of Uncertainty in Measurement (GUM)
- IUPAC, 2006 International Harmonized Protocol for the Proficiency Testing of Analytical Chemical Laboratories
- ISO 13528-2015, Statistical methods for use in proficiency testing by interlaboratory comparison, and
- ISO Guide 35 -2017, Reference Materials – Guidance for characterization and assessment of homogeneity and stability.

The accepted data-set included analyses tested by the standard test methods, where significant biases were not observed, as detailed in Table 2.

Table 2

Parameter	Analysis Methods used in Certification Testing*			
Ash	ISO 1171 (n=48)	AS 1038.3 (n=2)		
Volatile Matter	ISO 562 (n=48)	AS 1038.3 (n=2)		
Calorific Value	ISO 1928 (n=30)	AS 1038.5 (n=6)	ASTM D5865 (n=2)	
Relative Density	AS 1038.21.1.1 (n=24)	AS 1038.21.1.2 (n=4)		
Total Carbon	ISO 29541 (n=12)	AS 1038.6.1 (n=4)	AS 1038.6.4 (n=2)	ASTM D5373 (n=4)
Hydrogen	ISO 29541 (n=11)	AS 1038.6.1 (n=4)	ASTM D5373 (n=2)	
Nitrogen	ISO 29541 (n=11)	AS 1038.6.2 (n=6)	AS 1038.6.4 (n=2)	ASTM D5373 (n=2)
Total Sulfur	AS 1038.6.3.3 (n=20)	ISO 19579 (n=17)	ASTM D4239 (n=4)	
Chlorine	AS 1038.8.1 (n=6)	ISO/TS 18806 (n=4)	ISO 587 (n=1)	ASTM D4205 (n=2)
Phosphorus	AS 1038.14.3 (n=14)	ISO/TS 13865 (n=4)	AS 1038.9.3 (n=2)	
Mercury	ASTM D6722 (n=9)			
Fluorine	ISO 11724 (n=12)	AS 1038.10.4 (n=3)		

*Or their technical equivalents

5. Period of Validity

Property values for coal samples are subject to change due to the normal oxidation processes for coals. For this reason, the minimum shelf-life until the stated period of validity (January 2023) is provided for oxidation sensitive parameters i.e. for CV, VM, C and H. All other parameters are considered stable until January 2026. The stability of this sample will be monitored by ACIRS. It is the responsibility of the user to obtain the most recent Certification Report for this reference material available at www.acirs.com.au/products/general-coal-reference-material/

6. Metrological Traceability

Property values are operationally defined by methods listed in Table 2. Traceability of the results is based on traceability of all relevant input factors and was achieved by using only results from laboratories accredited to ISO/IEC 17025. Certified property values are based on agreement amongst technically valid results each traceable to the SI through stringent requirements of accreditation.

Note: Fluorine is traceable to the property value assigned to BCR 460.

7. Health and Safety

Samples shall be handled in accordance with the Safety Data Sheet available from www.acirs.com.au/products/general-coal-reference-material/

8. Legal Notice

To the extent permitted by law, ACIRS disclaims all warranties whether expressed or implied with regard to merchantability, non-infringement, or fitness for a particular purpose. In no event will ACIRS be liable for incidental damage or consequential loss arising from the use of this product.

Where the product does not conform to assigned property values, giving due consideration to the stated uncertainties and accepted tolerances, the total liability of ACIRS shall be limited at ACIRS' absolute discretion to either replacement of the product or refund of the purchase price.

9. Revision History

Document Number	Summary	Date
CR-G8-2019-rev0	Original (rev0)	20/03/2019
CR-G8-2019-rev01	Addition of fluorine, removal of CSN	31/07/2019

10. Authorisation

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