

TRACE ELEMENTS in COAL REFERENCE MATERIAL ACIRS-M1-2014-Lot#2

CERTIFICATION REPORT

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 Previous ACIRS-M series: ACIRS-M1-2014-Lot#2 supersedes ACIRS-M1-2014

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

Table 1 Assigned Property Values

	Assigned Property Values ¹	Standard Deviation ²	Expanded Uncertainty ³	Number of laboratories
Ash, % d	18.68	0.06	0.03	38
Relative Density, d	1.417	0.011	0.007	14
Total Sulfur, % d	2.39	0.09	0.02	145
Chlorine, % d	0.014	0.003	0.001	65
Phosphorus, % d	0.098	0.004	0.003	17
Fluorine, mg/kg d	160	7	4	22
Mercury, mg/kg d	0.122	0.010	0.004	41
Arsenic, mg/kg d	3.9	0.4	0.3	12
Selenium, mg/kg d	1.5	0.1	0.06	12

Table 2 Indicative Values

INDICATIVE VALUES				
	Indicative Value ⁴	Standard Deviation ²	Expanded Uncertainty ³	Number of laboratories
Cadmium, mg/kg d	0.18	0.01	0.01	9

Table 3 Informational Values

Parameter	Information Values ⁵	Parameter	Information Values ⁵
Volatile Matter, % d	32.8	Gross Calorific Value, MJ/kg d	28.36
Total Carbon, % d	67.4	Pyritic Sulfur, % d	0.07
Hydrogen, % d	4.73	Sulfate Sulfur, % d	0.21
Nitrogen, % d	1.34	Carbonate Carbon, % d	0.04

1. Notes

1 Assigned property values (Table 1) are the best estimate of the true value and are based on the robust mean of technically valid results from proficiency test programs using data from the following analysis methods;

- Ash: ISO 1171 and equivalent methods
- Relative Density: AS1038.21.1.1/1038.21.1.2
- Fluorine: ISO 11724 and equivalent methods
- Mercury: ASTM D6722 and equivalent methods
- Arsenic and Selenium: ISO 11723 and equivalent methods
- All other parameters were assigned from the results of multiple analysis methods where biases between methods were not observed.

2 Standard deviation (sd) is a robust value used to derive the likely range of results. 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 Cadmium, k -factor of 2.3 was chosen according to the t -distribution for sets of results.

4 Indicative values (Table 2) are provided where the relative uncertainty of the robust mean, or distribution of data, was considered unacceptably high and are not considered to be assigned property values.

5. Informational values (Table 3) are provided for the purpose of allowing the user to better understand the characteristics of this sample. They are not considered to be assigned property values.

2. Description of the Sample and Preparation

ACIRS-M1-2014-Lot#2 comprises a sealed jar containing approximately 125 g of coal at a nominal top size of 212 μm .

This sample was prepared from a high volatile bituminous coal sourced from the Hunter Valley, New South Wales in 2011. This material, at 50 mm top size, was air-dried and crushed to a nominal top size of 4 mm before being milled to 250 g sub-samples at -212 μm . This material was re-blended by repeated rotary sample division (RSD) processes before being divided, by RSD, until representative 125 g samples were obtained. Each sample was then placed into a plastic bag within sealed HDPE jars.

Homogeneity of the batch was confirmed by comparison of the dry ash and total sulfur values of each sample against the repeatability criteria of ISO 1171 and ISO 19579 respectively.

NOTE: This reference material (RM) was previously marketed as General Coal Reference Materials ACIRS-M1-2014 which reached the end of the period of validity for oxidation sensitive parameters in November 2017. Market demand for a trace element RM resulted in additional testing focussing on those parameters with re-brand to ACIRS-M1-2014-Lot#2.

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.

4. Characterisation

This sample was originally tested during CANSPEX 2014-3 and Proficiency Testing Australia (Round 31) proficiency testing programs. Values for Ash, Relative Density, Total Sulfur, Chlorine and Phosphorus were derived from this dataset.

Further testing was conducted in ACARP research project C25044 during 2016-18. This resulted in inclusion of certified values for Arsenic and Selenium; amendment of the certified values for Fluorine and Mercury and the addition of an indicative value for Cadmium.

Assigned and indicative property values (Tables 1 and 2) are based on datasets from the following analysis methods;

- Ash: ISO 1171 and equivalent methods
- Relative Density: AS1038.21.1.1/1038.21.1.2
- Total Sulfur, Chlorine and Phosphorus: various equivalent methods
- Fluorine: ISO 11724 and equivalent methods
- Mercury: ASTM D6722 and equivalent methods
- All other parameters were assigned from the results of multiple analysis methods where biases between methods were not observed.

All results were evaluated for technical competency before inclusion in the final certification dataset e.g. meeting standard method precision limits and by outlier identification. Robust statistical techniques were then used in the characterisation process in accordance with the guidelines of:

- IUPAC, 2006 International Harmonized Protocol for the Proficiency Testing of Analytical Chemical Laboratories
- ISO 13528-2005, Statistical design for use in proficiency testing by interlaboratory comparison, and
- ISO Guide 35 -2006, Reference Materials – General and statistical principles for certification.

Information values (Table 3) are provided for the purpose of allowing the user to better understand the characteristics of this sample. These values are from analyses conducted in 2014 and are subject to changes due to the normal oxidation process for coals.

5. Period of Validity

Assigned property values are considered stable until February 2023.

The stability of assigned property values for this sample will be monitored by ACIRS and it is the responsibility of the user to obtain the most recent documentation for this reference material available at www.acirs.com.au/products/. Informational values will not be monitored.

6. 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/

7. 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.

8. Revision History

Document Number	Summary	Date
CR-M1-2014-Lot-2	Original	01-06-2018

9. Authorisation

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