ACIRS-M2-2018
TRACE ELEMENTS in COAL REFERENCE MATERIAL
CERTIFICATE of ANALYSIS

Table 1 Assigned Property Values

CERTIFIED PROPERTY VALUES			
	Property Value[1]	**Standard Deviation[2]**	**Expanded Uncertainty[3]**
Ash, % d	9.32	0.09	0.06
Phosphorus (in coal), % d	0.1109	0.0053	0.0030
Arsenic, mg/kg d	0.3	0.09	0.07
Fluorine, mg/kg d	176	6	4
Mercury, mg/kg d	0.041	0.003	0.002
Selenium, mg/kg d	0.3	0.04	0.03

[1] Certified property values are the best estimate of the true value. They are an unweighted robust mean value of an accepted dataset obtained from proficiency testing using ISO/IEC 17025 accredited laboratories, each of whom tested a minimum of three unique samples. The standard test methods used are detailed in section 4. Where more than one test method was used, significant biases between methods were not observed.

[2] Standard deviation (s*) 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] Expanded Uncertainty (*U*) is a robust value estimated on the likely range of the true value of each parameter, including estimation of the uncertainty due to characterisation and heterogeneity, and is calculated from $1.25 \times s^*/\sqrt{n} \times k$ where k is a coverage factor of 2 corresponding to a level of confidence of about 95%. For Arsenic, a k-factor of 2.3 was chosen according to the t-distribution for sets of results.

Table 2 Informational Values

INFORMATIONAL VALUES[4]			
	Mean	**Range of Results**	**Number of Laboratories**
Chlorine, % d	0.031	0.022 - 0.037	15
Cadmium, mg/kg d	0.024	0.020 – 0.034	8

[4] Informational values are provided where the relative uncertainty of the robust mean or distribution of data was considered unacceptably high, or the number of participant results are low. These are not considered to be certified property values.

Date of Issue: 28 February 2025
Report Number: ACIRS-M2-2018-CoA-rev02
Previous ACIRS-M series: This sample is the first in the series.

1. Introduction

This report describes the preparation and certification of ACIRS-M2-2018.

This reference material is a higher rank bituminous coal and is intended to be used for quality control purposes for the analysis of similar coals.

2. Description of the Sample and Preparation

ACIRS-M2-2018 comprises a sealed jar containing 50 g (minimum) of coal at a nominal top size of 212 µm. This sample was prepared from 25 kg of a Queensland, Bowen Basin higher rank bituminous coal, at -50 mm top size.

This sample was prepared from a 25 kg bulk sample of a higher rank bituminous coal sourced from the Queensland, Bowen Basin in 2017. This material was air-dried and then crushed in a swing hammer mill to a nominal top size of 2.36 mm. The material was then repeatedly mixed by rotary sample division (RSD) until lots of approximately 0.5 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 50 g samples were obtained. Each sample was then placed into sealed amber glass jars.

Homogeneity of the batch was confirmed by comparison of the dry ash value of each sample against the ash repeatability criteria of ISO 1171.

3. Instructions for Handling and Use

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

Before each use, the bottle **must** be thoroughly mixed by end-over-end rotation to re-homogenise the coal sample.

To minimise the risk of compositional changes due to oxidation, store in a cool, dark place in the original container with lid 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-M2-2018 is established in accordance with the standard test methods listed in section 4. Corrections to dry basis values should be in accordance with ISO 11722 or equivalent.

4. Characterisation

ACIRS-M2-2018 was originally certified from the outcomes of ACARP research project C25044 during 2017-18.

Further testing was conducted in 2023 through an independent proficiency test program conducted by ACIRS. This testing provided sufficient data to add phosphorus and (update) selenium as assigned property values on this Certificate and extend the product shelf life.

The accepted certification dataset included analyses tested by the following analytical methods:

- From the 2017-18 dataset:
    - Ash – by ISO 1171 and equivalent methods (n = 18)
    - Fluorine – by ISO 11724 and equivalent methods (n = 16)
    - Arsenic – by ISO 11723 and equivalent methods (n = 9)
    - Mercury – by ASTM D6722 and equivalent methods (n = 11).
- From the 2023 dataset:
    - Phosphorus in coal – by ISO 622 and equivalent methods (n = 8) and by ISO 13605 and equivalent methods (n=12)
    - Selenium – by ISO 11723 and equivalent methods (n = 16).
- From the 2023/2024-25 datasets (Cl) and 2017-18/2024-25 datasets (Cd):
    - Chlorine – by ISO 18806 and equivalent methods (n=11) and ISO 587 and equivalent methods (n=4)
    - Cadmium – by ISO 23380 and equivalent methods (n=8)

Results were evaluated for technical competency before inclusion in the 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.

5. Metrological Traceability

Property values are operationally defined by methods listed in section 4 using only results from laboratories accredited to ISO/IEC 17025.

6. Period of Validity

Property values are considered stable until February 2028.

The stability of assigned property values 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/.

7. Health and Safety

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

8. Legal Notice

To the extent permitted by law, ACIRS disclaims all warranties whether expressed or implied regarding 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-M2-2018-rev0	- Original (rev0)	01/06/2018
ACIRS-M2-2018-CoA-rev01	- Update Table 1 & Notes: added P, updated Se from Indicative to Certified Value, removed Cd, n laboratories transferred to s4. - Added s1 (Introduction) & s5 (Traceability), updated s3 (Handling & Use) & s4 (Characterisation). - Extended shelf life to 02/2025. - Formatting, minor editorial corrections.	21/03/2023
ACIRS-M2-2018-CoA-Rev02	- Added Table 2: Cl and Cd as informational values. - Extended shelf life to 02/2028. - Formatting, minor editorial corrections.	28/02/2025

10. Authorisation

Approved by Andrew Swanson (ACIRS Commercial Manager)

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