

TECHNICAL REPORT

ACIRS-M1-2014

General Coal Reference Material

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Valid to: November, 2017
Report Number: TR1-M1-2014_rev2
Previous ACIRS-M series: This is the first in the ACIRS-Mx-yyyy series

1. Introduction

This report describes the preparation and characterisation of ACIRS–M1-2014 which comprises a sealed jar containing approximately 125 g of reference coal at a nominal top size of 212 µm.

This reference material is intended to be used for quality control purposes.

2. Property Values

Table1 Characterisation of ACIRS-M1-2014

	Property Values ¹	Standard Deviation ⁴	Uncertainty ⁵	Number of laboratories
Ash, % d ^{1a}	18.68	0.065	0.013	38
Volatile Matter, % d ^{1b}	32.85	0.286	0.057	40
Gross Calorific Value, MJ/kg d	28.356	0.1082	0.0116	135
Relative Density, d ^{1c}	1.417	0.0108	0.0036	14
Total Sulfur, % d	2.392	0.0924	0.0096	145
Total Carbon, % d	67.40	0.463	0.068	73
Hydrogen, % d	4.73	0.096	0.014	71
Nitrogen, % d	1.34	0.049	0.007	69
Phosphorus, % d	0.098	0.0042	0.0013	17
Carbonate Carbon, % d ^{1d}	0.037	0.0017	0.0010	5
Mercury, mg/kg d	0.120	0.0108	0.0020	46
Fluorine, mg/kg d ²	149	4.0	1.5	12
<i>Indicative Values</i> ³				
Chlorine, % d	0.014	0.0036	0.0006	57
Pyritic Sulfur, % d	0.07	0.009	0.003	19
Sulfate Sulfur, % d	0.21	0.019	0.005	19

- 1 Property values are the best estimate of the true value for the measurand and are based on the robust mean of participant results (outliers excluded) from proficiency test programs conducted by CANSPEX and Proficiency Testing Australia. Unless otherwise specified, parameters have been assigned from results of multiple analysis methods. Biases between methods were not observed. Further information can be obtained from the full technical report available at www.acirs.com.au/products/general-coal-reference-material/

1a Ash – property value assigned by ISO 1171 and equivalent methods

1b Volatile Matter - property value assigned by ISO 562 and equivalent methods

1c Relative Density – property value assigned by AS1038.21.1.1/1038.21.1.2

1d Carbonate carbon – property value assigned by AS 1038.23

- 2 Fluorine - assigned in accordance with ISO 11724 by 2 independent laboratories who tested 6 replicates on duplicate samples over 2 days (n=12). Fluorine results have been verified by, and are traceable to, IRRM BCR-460.
- 3 Indicative values have been provided for chlorine, pyritic and sulfate sulfur where the relative uncertainty of the robust mean, or distribution of data, was considered unacceptably high from the results of proficiency testing. These values are best estimates and are not considered to be property values.
- 4 Standard deviation (sd) is used to derive the likely range of results - the value for a measurand from a randomly chosen laboratory would be expected to lie within 2 sd of the property values with 95% probability.
- 5 The uncertainty of this value has been calculated from $sd/\sqrt{n}$ where n= number of laboratories.

3. Instructions for Use

ACIRS-M1-2014 does not require further preparation prior to analysis however **must always be thoroughly mixed** by end-over-end rotation before sub-sampling.

To minimise the risk of compositional changes due to oxidation store in a cool, dark place in original containers with lids tightly sealed.

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

4. Sample Source and Preparation

ACIRS-M1-2014 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 -212 μm . This material was re-blended in 2014 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 in a HDPE jars.

5. Homogeneity testing

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

6. Characterisation

a. Parameters other than fluorine

Blind samples of ACIRS-M1-2014 were analysed through two proficiency test programs i.e. CANSPEX 2014-3 and Proficiency Testing Australia- round 31. Characterisation of ACIRS-M1-2014, based on results from these programs, was conducted by ACIRS. Robust statistical techniques were 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.

Assigned property values are based on the robust mean, after exclusion of outliers, of proficiency test program participant results and are based on analysis by multiple test methods*. Significant method biases were not detected.

*The exceptions were ash, volatile matter, carbonate carbon and relative density which were certified by AS/ISO equivalent methods.

Indicative values have been provided for chlorine, pyritic and sulfate sulfur where the relative uncertainty of the robust mean, or distribution of data, was considered unacceptably high from the results of proficiency testing. These values are best estimates and are not assigned property values.

b. Fluorine

The property value for fluorine was assigned in accordance with ISO 11724 by 2 independent accredited laboratories who tested 6 replicates on duplicate samples over 2 days (n=12).

Fluorine results have been verified by, and are traceable to, IRRM BCR-460.

7. Period of validity

The stated period of validity for ACIRS-M1-2014, until the (November, 2017), is provided for oxidation sensitive parameters. Property values will be subject to change should this reference material deteriorate due to the normal oxidation processes for coals. This will be monitored by stability test programs conducted by ACIRS. It is the responsibility of the user to use the most recent documentation for this reference material.

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.

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