



TECHNICAL REPORT

ACIRS-M1-2014

General Coal Reference Material

Date of Issue: November, 2014
Valid to: November, 2016+
Report Number: TR1-M1-2014
Previous ACIRS-M series: This is the first in the ACIRS-Mx-yyyy series

1. Introduction

This report describes the preparation and certification 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 to support the validity of analysis results for those parameters where certified values have been provided; and as a quality control tool.

2. Certified Values

Table1 Characterisation of ACIRS-M1-2014

	Certified Values ¹	Standard Deviation ²	Uncertainty ³	Number of laboratories
Ash, % d ^{1a}	18.68	0.065	0.011	38
Volatile Matter, % d ^{1b}	32.85	0.286	0.045	40
Gross Calorific Value, MJ/kg d	28.356	0.1082	0.0093	135
Relative Density, d ^{1c}	1.417	0.0108	0.0029	14
Total Sulfur, % d	2.392	0.0924	0.0077	145
Total Carbon, % d	67.40	0.463	0.054	73
Hydrogen, % d	4.732	0.0961	0.0114	71
Nitrogen, % d	1.335	0.0490	0.0059	69
Phosphorus, % d	0.0980	0.0042	0.0010	17
Carbonate Carbon, % d ^{1d}	0.037	0.0017	0.0008	5
Mercury, mg/kg d	0.120	0.0108	0.0016	46
<i>Indicative Values</i> ⁴				
Chlorine, % d	0.014	0.0036	0.0005	57
Pyritic Sulfur, % d	0.071	0.0093	0.0021	19
Sulfate Sulfur, % d	0.209	0.0186	0.0043	19

¹ Certified values are the best estimate of the true value for the measurand and are based on the robust mean of participant results (outliers excluded). Unless otherwise specified, parameters have been certified from results from multiple analysis methods from proficiency test programs results. Biases between methods were not observed.

^{1a} Ash certified by ISO 1171 and equivalent methods

^{1b} Volatile Matter certified by ISO 562 and equivalent methods

^{1c} Relative Density certified by AS 1038.21.1.1/1038.21.1.2.

^{1d} Carbonate carbon certified by AS 1038.23.

² 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 certified value with 95% probability.

³ The uncertainty of this value has been calculated from sd/\sqrt{n} where n= number of laboratories.

⁴ 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 shall not to be used as certified values.

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. Certification

Blind samples of ACIRS-M1-2014 were analysed through two proficiency test programs which were conducted by independent providers. Characterisation of ACIRS-M1-2014, based on results from this program, 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 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.

Certified values are based on the robust mean, after exclusion of outliers, of proficiency test program participant results. Certified values 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 shall not to be used as certified values.

+ The shelf-life of ACIRS-M1-2014, until the stated period of validity (November, 2016), is provided for oxidation sensitive parameters, primarily calorific value.

The calorific value 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 Technical Report and Certificate of Analysis for this reference material available at www.acirs.com.au/products/general-coal-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 the certified 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.

Approved by: Mark Bennetts

Australian Coal Industry Reference Samples (ACIRS)

PO Box 2315, DANGAR NSW 2309, AUSTRALIA

Phone +61 (2) 4926 4870

Fax +61 (2) 4926 4902

Email acpsnational@acps.com.au