ACIRS-S2D-2016

SULFUR REFERENCE MATERIAL

CERTIFICATE of ANALYSIS

Table 1: Assigned Property Values

ASSIGNED PROPERTY VALUES			
	Property Value[1]	**Standard Deviation[2]**	**Expanded Uncertainty[3]**
Ash, % d	**18.02**	**0.081**	**0.042**
Total Sulfur, % d	**2.877**	**0.0815**	**0.020**

[1] Property values are the best estimate of the true value for the measurand and are based on the robust mean of participant results from a CANSPEX proficiency testing program conducted (excluding results which did not meet ACIRS precision criteria). Ash was analysed by ISO 1171 and equivalent test methods. Biases between methods were not observed. Results from in-house methods were included when within the normal distribution of standard national and international test methods.

[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 approximately 95%.

Date of Certification:	June 2016
CoA Issue Date:	July 2023
Report Number:	ACIRS-S2D-2016-CoA-rev01
Previous ACIRS-SxD series:	This is the second in the series and supersedes ACIRS-S1D- 2011

1. Introduction

This report describes the preparation and certification of ACIRS-S2D-2016.

This reference material is a higher rank bituminous coal. Total Sulfur and Ash are intended to be used for quality control purposes for the analysis of similar coals. Total Sulfur is traceable to SI units and is therefore also suitable for calibration purposes.

2. Description of the Sample and Preparation

ACIRS-S2D-2016 comprises a sealed jar containing approximately 125 g of coal at a nominal top size of 212 µm. This sample was prepared from 250 kg of a Hunter Valley higher rank bituminous coal, obtained at -50 mm top size.

The coal was stabilised for several months before being 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 1.5 kg were obtained and then individually air dried and milled to a nominal top size of 212 µm. This pulverised material was further divided by RSD to obtain the representative samples of approximately 125 g each. Each sample was then placed into a plastic bag within sealed HDPE jars.

Homogeneity of the batch was assessed by selecting 24 bottles by stratified random sampling and tested for Ash and Total Sulfur by ISO 1171 and AS 1038.6.3.3 respectively. Satisfactory sample homogeneity for this coal was established after evaluation in accordance with ISO Guide 35, 2006.

3. Instructions for Handling and Use

Before first use, empty the sample from the inner plastic bag directly into the HDPE jar.

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 original containers with the lid tightly sealed. ACIRS cannot be held responsible for any changes that occur after the sample bottle has been opened.

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

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4. Characterisation

ACIRS-S2D-2016 was analysed as an unknown sample in the proficiency testing program CANSPEX 2016-1 conducted by Quality Associates International Ltd.

Characterisation was conducted by ACIRS using robust statistical techniques in accordance with the guideline of:

- IUPAC, 2006 International Harmonised Protocol for the Proficiency Testing of Analytical Laboratories
- ISO 13528, 2015, Statistical methods for use in proficiency testing by interlaboratory comparison, and
- ISO Guide 35, 2006, Reference Materials- General and statistical principles for certification.

NOTES:

- Assigned property values are based on the robust mean of the proficiency testing dataset.
- Proficiency testing program data set for Total Sulfur includes analyses conducted by nationally and internationally recognised test methods and in-house methods.
    - Data which did not meet ACIRS precision criteria was excluded from the proficiency testing program dataset. In-house methods were included when within the normal distribution of recognised national and international methods of analysis.
    - Where data from multiple methods have been combined, significant method biases were not detected.
    - Ash was determined by ISO 1171 and equivalent test methods.

5. Metrological Traceability

Traceability of the certified value for Total Sulfur to the SI unit of mass was confirmed through independent analysis by an ISO/IEC accredited laboratory using test method ISO 19579 and demonstrating an unbroken chain of calibrations.

Traceability for Ash has not been confirmed other than through the CANPEX 2016-1 proficiency test program.

6. Period of Validity

The stability of assigned property values given in Table 1 will be monitored by ACIRS. The minimum shelf-life until the stated period of validity (June 2026) is provided for Total Sulfur. It is the responsibility of the user to obtain the most recent Certificate of Analysis (CoA) for this reference material available at www.acirs.com.au/products/acirs-sulfur-reference-materials-/.

7. Health and Safety

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

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
ACIRS-S2D-PIL-01	Original (rev0)	08/08/2016
ACIRS-S2D-TR-01	Original (rev0)	08/08/2016
ACIRS-S2D-PIL-02	Minor editorial changes	27/03/2017
ACIRS-S2D-TR-02	Minor editorial change	27/03/2017
ACIRS-S2D-PIL-03	Addition of certified value for ash, editorial changes	15/02/2018
ACIRS-S2D-TR-03	Addition of certified value for ash, editorial changes	15/02/2018
ACIRS-S2D-2016-CoA-rev01	Removal of assigned property values for Chlorine and Mercury. Format change to align with ACIRS CoA standard, replacing ACIRS-S2D-PIL and ACIRS-S2D-TR documents. Total Sulfur values valid to June 2026 Modified expanded uncertainty definition to align with current definition	25/07/2023

10. Authorisation

Approved by Andrew Swanson (ACIRS Commercial Manager)

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