ACIRS-S2B-2015
SULFUR REFERENCE MATERIAL

CERTIFICATE of ANALYSIS

ASSIGNED PROPERTY VALUES			
	Property Value[1]	**Standard Deviation[2]**	**Expanded Uncertainty[3]**
Total Sulfur, % d[4]	0.692	0.0237	0.005
Chlorine, % d	0.027	0.0031	-
Mercury, mg/kg d	0.057	0.0081	-
Fluorine, mg/kg d	37	8.1	-

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 an interlaboratory testing program conducted by CANSPEX (excluding results which did not meet ACIRS precision criteria). ISO and equivalent test methods were accepted and 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%.

4 Total Sulfur is a certified value and is intended for use as a calibrant. All other assigned property values on this certificate are for quality control purposes only.

Date of Certification: August, 2015
CoA Issue Date: December, 2024
Valid to: December, 2027
Report Number: ACIRS-S2B-2015-CoA-rev1
Previous ACIRS-SxB series: This is the second in the series and supersedes ACIRS-S1B-2011

1. INTRODUCTION

This report describes the preparation and certification of ACIRS-S2B-2015.

This reference material is a higher rank bituminous coal. Total Sulfur is traceable to SI units through NIST SRM 2693 and NIST SRM 1632d and is therefore suitable for calibration purposes. Other elements are intended to be used for quality control purposes for the analysis of similar coals.

2. DESCRIPTION OF THE SAMPLE AND PREPARATION

ACIRS-S2B-2015 comprises a sealed jar containing approximately 125 g of coal at a nominal top size of 212 µm. This sample was prepared from 320 kg of a Hunter Valley higher rank bituminous coal, obtained at -50 mm top size.

The coal was stabilised in storage 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-/.

Total Sulfur is traceable to SI units and is therefore suitable for calibration purposes. Other elements are intended to be used for quality control purposes for the analysis of similar coals.

4. CHARACTERISATION

ACIRS-S2B-2015 was analysed as an unknown sample in the proficiency test program CANSPEX 2015-1 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 interlaboratory testing dataset.
- Interlaboratory testing data set for Total Sulfur includes analyses conducted by nationally and internationally recognised test methods and in-house methods as included in Table 1.
    - Data which did not meet ACIRS precision criteria was excluded from the interlaboratory 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.

Table 1: *Standard test methods used in the interlaboratory testing for ACIRS-S2B-2015*

Parameter	Analysis Methods	n (total)
TS	ASTM D4239 (n=83), ASTM D5016 (n=5), ISO 19579 (n=3), ISO 29541 (n=1), AS 1038.6.3.3 (n=1), GB/T 214 (n=1), BS 1016 (n=1), DIN 51724-3 (n=2), Other including in house (n=13)	110
Cl	ASTM D4208 (n=20), ASTM D6721 (n=9), AS 1038.1 (n=1), GB/T 3558 (n=1), NF M 03-009 (n=1), DIN 51727 (n=1), Other including in house (n=15)	48
Hg	ASTM D6722 (n=27), ASTM D3684 (n=1), ASTM D6414 (n=3), BS 1016 (n=1), Other including in house (n=8)	40
F	ASTM D3761 (n=15), ASTM D5987 (n=2), AS 1038.10.4 (n=1), BS 1016 (n=1), DIN 51723 (n=1), Other including in house (n=10)	30

5. METROLOGICAL TRACEABILITY

Property values are operationally defined by methods listed in Table 1. Traceability to SI units for Total Sulfur was achieved through NIST SRM 2693 and NIST SRM 1632d. These NIST SRMs are bituminous coal reference materials listed on the Bureau International des Poids et Mesures, Key Comparison Database (BIPM-KCDB) thereby providing traceability to a higher order reference material.

Traceability for Cl, Hg and F have not been confirmed other than through the interlaboratory study and are provided for quality control purposes on similar coals.

6. Period of Validity

The stability of assigned property values in this certificate of analysis will be monitored by ACIRS. The minimum shelf-life until the stated period of validity (Dec 2027) is provided for Total Sulfur, Chlorine, Mercury and Fluorine. 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
TRI-S2B-2015_rev0	Original	27/07/2015
TRI-S2B-2015_rev1	Traceability statement added	02/12/2015
ACIRS-S2B-TR-rev2	Revision history and minor editorial change	27/03/2017
ACIRS-S2B-2015-CoA-rev0	Formatting and minor editorial changes Revised period of validity Revised Instructions for Use section Added sections for Health and Safety and Metrological Traceability Removed Additional Information section	12/12/2022
ACIRS-S2B-2015-CoA-rev1	Formatting and minor editorial changes Revised period of validity Analysis test method (Table 1) added	6/12/2024

10. Authorisation

Approved by Andrew Swanson (ACIRS Commercial Director)
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