CERTIFICATE OF ANALYSIS
ACIRS-S2D-2016
Sulfur Certified Reference Material

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Table 1 Certified property values

Element			
Sulfur (S)	2.877	+/-	0.021
Element	**mg/kg, d**		
Antimony (Sb)	0.167	+/-	0.022
Cobalt (Co)	4.59	+/-	0.18
Lead (Pb)	6.42	+/-	0.18
Manganese (Mn)	50.7	+/-	0.5
Nickel (Ni)	12.4	+/-	0.3
Selenium (Se)	1.77	+/-	0.09
Strontium (Sr)	38.0	+/-	0.6
Vanadium (V)	41.2	+/-	1.9

Table 2 Non-certified property values

Parameter			
Ash	17.81	+/-	0.04
Chlorine (Cl)	0.0092	+/-	0.0002
Phosphorus (P)	0.0074	+/-	0.0004
Element	**mg/kg, d**		
Arsenic (As)	(4.15)		
Barium (Ba)	93.3	+/-	2.5
Boron (B)	41.5	+/-	2.1
Cadmium (Cd)	0.090	+/-	0.009
Chromium (Cr)	(19.6)		
Copper (Cu)	26.7	+/-	0.8
Fluorine (F)	97	+/-	3
Lithium (Li)	(14.3)		
Mercury (Hg)	0.281	+/-	0.008
Molybdenum (Mo)	(1.01)		
Zinc (Zn)	33.2	+/-	1.7

Values are expressed as robust mean +/- expanded uncertainty (U). Property values are the best estimate of the true value and are an unweighted robust mean value of an accepted dataset obtained from an interlaboratory study using ISO/IEC 17025 accredited laboratories, each of whom tested a minimum of five 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.

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 x $s^*/\sqrt{n}$ x k where k is a coverage factor of 2 corresponding to a level of confidence of approximately 95%.

Values in parentheses () indicate an informational value.

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1. Purpose

This reference material is a higher rank bituminous coal. The certified values for Total Sulfur and specified trace elements (Table 1) are traceable to SI units and are suitable for calibration purposes. All non-certified values (Table 2) are intended to be used for quality control purposes for the analysis of similar coals. Informational values are provided for reference only.

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.

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.

The minimum sample intake for ACIRS-S2D-2016 is established in accordance with the standard test methods listed in section 4. Corrections to dry basis values should be in accordance with ISO 1170 or equivalent.

4. Homogeneity Assessment and Characterisation

Homogeneity of the batch was assessed by selecting 24 bottles by stratified random sampling and testing 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 33405:2024.

ACIRS-S2D-2016 was analysed as an unknown sample in the proficiency testing program CANSPEX 2016-1 conducted by Quality Associates International Ltd and again in an interlaboratory study by ACIRS in 2025. The accepted characterisation dataset included testing by analytical methods in Table 3.

Table 3 Analysis Methods

Parameter	Analysis Method	Number of Results
Ash, % d	ISO 1171 (20), ASTM D3174-12 (5)	25
Total Sulfur, % d	ASTM D4239 (67), ASTM D5016 (4), ISO 19579 (5), in-house (13), various other (10)	99
Chlorine, % d	ISO 18806 (14), ASTM D6721-24 (5)	19
Phosphorus, % d	ISO 13605 (10), ASTM D6349-21 (10)	20
Antimony, mg/kg d	ASTM D6357-21b (10), In-house method (10)	20
Arsenic, mg/kg d	ISO 11723 (5), ASTM D6357-21b (10), In-house method (9)	24
Barium, mg/kg d	ASTM D6357-21b (5), ASTM D6349-21 (10), in-house method (5)	20
Boron, mg/kg d	AS 1038.10.3 (10), ASTM D8213-18 (5), In-house method (9)	24
Chromium, mg/kg d	ASTM D6357-21b (10), In-house method (12)	22
Cadmium, mg/kg d	ASTM D6357-21b (10), In-house method (15)	25
Cobalt, mg/kg d	ASTM D6357-21b (10), In-house method (15)	25
Copper, mg/kg d	ASTM D6357-21b (10), In-house method (15)	25
Fluorine, mg/kg d	ISO 11724 (12), In-house method (5)	17
Lead, mg/kg d	ASTM D6357-21b (10), In-house method (15)	25
Lithium, mg/kg d	ASTM D6357-21b (5), In-house method (10)	15
Manganese, mg/kg d	ISO 13605 (5), ASTM D6357-21b (10), In-house method (10)	25
Mercury, mg/kg d	ASTM D6722 (20), In-house method (5)	25
Molybdenum, mg/kg d	ASTM D6357-21b (15), In-house method (7)	22
Nickel, mg/kg d	ASTM D6357-21b (10), In-house method (15)	25
Selenium, mg/kg d	ISO 11723 (3), ASTM D4606-21 (5), In-house method (9)	17
Strontium, mg/kg d	ISO 13605 (5), ASTM D6349-21 (10), In-house method (2)	17
Vanadium, mg/kg d	ASTM D6357-21b (10), In-house method (15)	25
Zinc, mg/kg d	ASTM D6357-21b (15), In-house method (10)	25

Results were evaluated for technical competency before inclusion in the characterisation dataset, e.g., by 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 13528:2022, Statistical methods for use in proficiency testing by interlaboratory comparison, and
- ISO 33405:2024 Reference Materials – Approaches for characterization and assessment of homogeneity and stability

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.

Property values are operationally defined by methods listed in section 4 using only results from laboratories accredited to ISO/IEC 17025. Certified values in Table 1 include metrological traceability with primary (NMI) reference standard NIST SRM 1635a.

6. PERIOD OF VALIDITY

Property values are considered stable until June 2036. The stability of assigned property values and informational 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 or informational 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 Cl and Hg and informational values table. 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
ACIRS-S2D-2016-CoA-rev02	Added Hg back on certificate per original certified values as stability confirmed. Adjusted significant figures in assigned property value, standard deviation, and expanded uncertainty.	23/11/2023
ACIRS-S2D-2016-CoA-rev03	Formatting, design changes to document. Change to wording in Note 1 in Table 1. Minimum sample intake added to Section 3. Methods specified in Section 4. Ash value removed from the certificate. Period of validity extended to June 2029.	16/04/2026
ACIRS-S2D-2016-CoA-rev04	Updated Ash value with changes since initial characterisation due to oxidative processes. Added 20 trace element property values. Period of validity extended to June 2036. Formatting, minor editorial changes.	30/06/2026

10. Authorisation

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