TECHNICAL REPORT

ACIRS-S2D-2016

Sulfur Reference Material

With Mercury and Chlorine

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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 which comprises a sealed jar containing approximately 125 g of reference coal at a nominal top size of 212 µm.

Production and characterisation of this sample has been conducted in accordance with the technical and production requirements of ISO Guide 34.

2. Property Values

This reference material is a higher rank bituminous coal.

Total sulfur is traceable to the certified value for sulfur on NIST 1632d and is therefore suitable for calibration or quality control purposes on similar coals.

Mercury and chlorine values are provided for quality control purposes on similar coals.

Table 1 Assigned Property Values

	Property Value[1]	Standard Deviation[2]	Uncertainty[3]	Number of laboratories
Total Sulfur, % d	2.877	0.0815	0.0205	99
Chlorine, % d	0.010	0.0014	0.0005	41
Mercury, mg/kg d	0.281	0.0184	0.0077	36

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). Parameters have been assigned from results of multiple analysis 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 is a robust value 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 standard deviations of the assigned property values with 95% probability.

3 Uncertainty is an expanded uncertainty of the property value is the likely range of the true (but unknown) value for each parameter with a coverage factor of 2 at 95% confidence limits. This includes contribution from variations in measurement results, inhomogeneity and instability. It is calculated from 2 x 1.25 x robust sd/√n.

3. Instructions for Use

This sample **must** be thoroughly mixed by end-over-end rotation each time before sub-sampling. To minimise the risk of compositional changes due to oxidation, store in a cool, dark place in original containers with the lid tightly sealed.

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

4. Sample Source and Preparation

Approximately 250 kg of a Hunter Valley higher rank bituminous coal was 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 until representative samples were obtained of approximately 125 g each. Each sample was then placed into a plastic bag within sealed HDPE jars.

5. Homogeneity testing

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 Guide 35, 2006.

6. 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 guidelines of:

- IUPAC, 2006 International Harmonized Protocol for the Proficiency Testing of Analytical Chemical 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 program dataset.
- The proficiency testing program dataset includes analyses conducted by nationally and internationally recognised test methods and in-house methods.
    - Data was excluded from the proficiency testing program dataset which did not meet ACIRS precision criteria. 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.

7. Period of Validity

The stability of assigned property values given in Table 1 will be monitored by ACIRS. It is the responsibility of the user to obtain the most recent Technical Report and Product Information Leaflet for this reference material available at www.acirs.com.au/products/acirs-sulfur-reference-materials-/

8. Additional Information

The indicative values in Table 2 are provided to allow the user to better understand the characteristics of this sample and not for quality control or calibration purposes. Values provided are derived from CANSPEX Q1, 2016 proficiency testing program and are subject to change due to normal coal oxidation processes.

Table 2 Informational Data

Parameter	Indicative Value
Ash, % d	18.0
Volatile Matter, % d	35.5
Gross Calorific Value, MJ/kg d	28.02
Total Carbon, % d	66.6
Hydrogen, % d	4.81
Nitrogen, % d	1.21
Pyritic Sulfur, % d	0.57
Sulfate Sulfur, % d	0.65
Fluorine, mg/kg d	80
Selenium, mg/kg d	1.6
Phosphorus, % d	0.004

Revision History

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ACIRS-S2D-TR-01	Original (rev0)	08/08/21016

Disclaimer

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.

Approval

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