

TECHNICAL REPORT

ACIRS-S2B-2015

Sulfur Reference Material

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| Report Number: | ACIRS-S2B-TR-rev02 |
| 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 which comprises a sealed jar containing approximately 125 g of reference coal at a nominal top size of 212 µm.

This reference material is a higher rank bituminous coal intended to be used for quality control purposes. Total sulfur is traceable to SI units through NIST SRM 2693 and NIST SRM 1632d and is therefore suitable for calibration purposes.

2. Property Values

Table 1 Assigned Property Values

| | Property Value ¹ | Standard Deviation ² | Uncertainty ³ | Number of laboratories |
|-------------------|-----------------------------|---------------------------------|--------------------------|------------------------|
| Total Sulfur, % d | 0.692 | 0.0237 | 0.0028 | 110 |
| Chlorine, % d | 0.027 | 0.0031 | 0.0006 | 48 |
| Mercury, mg/kg d | 0.057 | 0.0081 | 0.0016 | 40 |
| Fluorine, mg/kg d | 37 | 8.1 | 1.8 | 30 |

¹ Property values are the best estimate of the true value for the measurand and are based on the robust mean of participant results (outliers excluded) from a proficiency test program conducted by CANSPEX. 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 (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 property values with 95% probability.

³ The uncertainty of this value has been calculated from $1.25 \times sd/\sqrt{n}$ where n= number of laboratories.

3. Instructions for Use

This sample should 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 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 320 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 125 g samples were obtained. 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 randomly 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-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 guidelines of:

- IUPAC, 2006 International Harmonized Protocol for the Proficiency Testing of Analytical Chemical 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.

NOTES:

- Assigned property values are based on the robust mean, after exclusion of outliers. This includes the analysis results from multiple test methods including nationally and internationally recognised test methods and in-house methods.
- Where data from multiple methods have been combined, significant method biases were not detected.
- In-house methods were included in the data-set when within the normal distribution of recognised national and international methods of analysis.

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 values in table 2 are provided from results of the CANSPEX 2015- proficiency test program for informational purposes only. The purpose is to allow the user to better understand the characteristics of this sample. Oxidation sensitive parameters are subject to change due to the normal oxidation processes for coals and will not be monitored.

Table 2 Informational Data

| | Indicative Value ⁴ | Standard Deviation | Number of laboratories |
|--------------------------------|-------------------------------|--------------------|------------------------|
| Ash, % d | 9.1 | 0.1 | 123 |
| Volatile Matter, % d | 34.0 | 0.6 | 102 |
| Gross Calorific Value, MJ/kg d | 31.05 | 0.08 | 112 |
| Total Carbon, % d | 75.4 | 0.6 | 65 |
| Hydrogen, % d | 4.90 | 0.12 | 60 |
| Nitrogen, % d | 1.79 | 0.06 | 58 |
| Pyritic Sulfur, % d | 0.12 | 0.02 | 22 |
| Sulfate Sulfur, % d | 0.02 | 0.01 | 18 |
| Selenium, mg/kg d | 0.4 | 0.1 | 13 |

⁴ Informational data / Indicative values are provided for information purposes only

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.

Authorisation

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Revision History

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