

# ACIRS-G9-2021

## GENERAL COAL REFERENCE MATERIAL

### CERTIFICATE of ANALYSIS

Production and certification of this sample was conducted in accordance with ISO 17034.

**Table 1 Assigned Property Values**

<b>ASSIGNED PROPERTY VALUES</b>				
	<b>Certified Value<sup>1</sup></b>	<b>Standard Deviation<sup>2</sup></b>	<b>Standard Uncertainty<sup>3a</sup></b>	<b>Expanded Uncertainty<sup>3b</sup></b>
<b>Ash, % d</b>	10.69	0.071	0.013	0.026
<b>Volatile Matter, % d</b>	20.00	0.212	0.040	0.079
<b>Gross Calorific Value, MJ/kg d</b>	32.105	0.064	0.012	0.025
<b>Relative Density, d</b>	1.386	0.016	0.003	0.007
<b>Total Carbon, % d</b>	79.07	0.328	0.075	0.150
<b>Hydrogen, % d</b>	4.31	0.076	0.017	0.035
<b>Nitrogen, % d</b>	1.79	0.029	0.007	0.013
<b>Total Sulfur, % d</b>	0.601	0.007	0.001	0.003
<b>Chlorine, % d</b>	0.056	0.005	0.001	0.003
<b>Phosphorus, % d</b>	0.036	0.001	0.0003	0.001
<b>Mercury, mg/kg d</b>	0.030	0.002	0.0006	0.0011
<b>Fluorine, mg/kg d</b>	100	4.6	1.5	2.9
<b>Carbonate Carbon, % d</b>	0.063	0.006	0.002	0.004

1. Certified values are the best estimate of the true value. They are an unweighted robust mean value of an accepted dataset obtained from proficiency testing using ISO/IEC 17025 accredited laboratories each of whom tested three unique samples. The standard test methods used are detailed in Table 2. Where more than one test method was used, biases between methods were not observed
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. Uncertainty provides the user with information on the likely range of the true (but unknown) value for each parameter and has been estimated in accordance with the Guide to the Expression of Uncertainty in Measurement (GUM)
  - a. Standard uncertainty ( $u_a$ ) is a robust value estimated at  $1.25 \times s^*/\sqrt{n}$
  - b. Expanded uncertainty ( $U$ ) is calculated from  $u_a \times$  a coverage factor  $k=2$ , corresponding to a level of confidence of about 95%.

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 Previous ACIRS-G series: This sample supersedes ACIRS-G8-2019

## 1. Introduction

This report describes the preparation and certification of ACIRS–G9-2021.

This reference material is a higher rank bituminous coal and is intended to be used for quality control purposes for the analysis of similar coals.

## 2. Description of the Sample and Preparation

ACIRS–G9-2021 comprises a sealed jar containing approximately 125 g of coal at a nominal top size of 212  $\mu\text{m}$ . This sample was prepared from 350 kg of a Queensland, Bowen Basin higher rank bituminous coal, at -50 mm top size.

The bulk coal sample was crushed in a swing hammer mill to a nominal top size of 2.36 mm and stabilised over an extended period. The material was then repeatedly mixed by rotary sample division (RSD) until lots of approximately 1.4 kg were obtained which were then air dried and milled to a nominal top size of 212  $\mu\text{m}$ . This pulverised material was further divided by RSD until representative samples were obtained. Each sample was then placed into a plastic bag within sealed HDPE jars.

Between-unit homogeneity was quantified by testing ash, gross calorific value and total sulfur and assessing for sufficient homogeneity in accordance with ISO 17034.

## 3. Instructions for Use

This reference material is intended to be used as a quality control tool.

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 the original container with lids 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-G9 is established in accordance with the standard test methods listed in Table 2. Corrections to dry basis values should be in accordance with ISO 11722 or equivalent.

## 4. Characterisation

ACIRS-G9 was characterised in an interlaboratory comparison program conducted by Proficiency Testing Australia in collaboration with ACIRS. This program was conducted specifically for characterisation of ACIRS-G9.

Sixteen accredited Australian coal testing laboratories were invited to participate in the program.

Each participant was provided three unique ACIRS-G9 samples. Additionally, an ACIRS-G8-2019 sample was provided to participants for quality assurance purposes.

Participant data was accepted in the characterisation dataset when:

- The laboratory was accredited to ISO/IEC 17025 for the specific parameter;
- Testing was conducted by a national or international standard test methods; and
- When results met technical acceptability along ACIRS guidelines e.g. meeting standard method precision limits; not identified as an outlier; passed acceptance testing on ACIRS-G8.

The certified values and their associated uncertainties were calculated from an unweighted mean value of robust means in accordance with the guidelines of:

- ISO/IEC Guide 98-3, Guide to the Expression of Uncertainty in Measurement (GUM)
- ISO 13528-2015, Statistical methods for use in proficiency testing by interlaboratory comparison, and
- ISO Guide 35 -2017, Reference Materials – Guidance for characterization and assessment of homogeneity and stability

The accepted dataset included analyses tested by the standard test methods, where significant biases were not observed, as detailed in Table 2.

Notes:

- Supplementary testing on an additional three samples was conducted by ACIRS for certification of mercury.
- This sample has relatively low levels of pyritic sulfur (<0.1%).

**Table 2 Methods Used for Certification**

Parameter	Analysis Method(s) Used <sup>4</sup>	n (total) <sup>5</sup>
Ash	ISO 1171* (n=48)	48
Volatile Matte*	ISO 562* (n=45)	45
Calorific Value	ISO 1928* (n=42)	42
Relative Density	AS1038.21.1.1 (n=36)	36
Total Carbon	ISO 29541* (n=30)	30
Hydrogen	ISO 29541* (n=30)	30
Nitrogen	ISO 29541* (n=30)	30
Total Sulfur	ISO 19579 (n=27), AS 1038.6.3.3 (n=15)	42
Chlorine	ISO 587 (n=6), ISO 18806 (n=6), AS 1038.8.1 (n=6)	18
Phosphorus	ISO 622 (n=3), AS 1038.9.3 (n=3), ISO 13605 (n=6), AS 1038.14.3 (n=6)	18
Mercury	ASTM D 6722 (n=21)	21
Fluorine	ISO 11724* (n=15)	15
Carbonate Carbon	AS 1038.23 (n=9), ISO 925* (n=3)	12

<sup>4</sup> An \* indicates methods used includes the corresponding Australian Standard methods AS 1038.3-2000 (ash, VM), AS 1038.5-1998 (CV), AS 1038.6.4-2005 (C, H, N), AS 1038.10.4 (F).

<sup>5</sup> Total number of results (n) is the number of accepted results in the dataset, each being the average of duplicate results. Three unique samples were tested by each participant for all parameters except Hg where six unique samples were tested.

## 5. Metrological Traceability

Property values are operationally defined by methods listed in Table 2 using only results from laboratories accredited to ISO/IEC 17025.

## 6. Period of Validity

Property values for coal samples are subject to change due to the normal oxidation processes for coals. For this reason, the minimum shelf-life until the stated period of validity (February 2025) is provided for oxidation sensitive parameters i.e. for CV, VM, C and H. All other parameters are considered stable until February 2028.

The stability of this sample will be monitored by ACIRS. It is the responsibility of the user to obtain the most recent Certificate for this reference material available at [www.acirs.com.au/products/general-coal-reference-material](http://www.acirs.com.au/products/general-coal-reference-material).

## 7. Health and Safety

Samples shall be handled in accordance with the Safety Data Sheet available from [www.acirs.com.au/products/general-coal-reference-material](http://www.acirs.com.au/products/general-coal-reference-material).

## 8. Legal Notice

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.

## 9. Revision History

**Table 3 Revision History**

Document Number	Summary	Date
CoA-G9-2021-rev0	Original	04/06/2021
CoA-G9-2021-rev01	Updated table captions	29/6/2021
CoA-G9-2021-rev02	Clarified traceability statement and minor editorial corrections.	16/11/2021

## 10. Authorisation

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