

ACIRS-A2-2024 COAL ASH REFERENCE MATERIAL

CERTIFICATE of ANALYSIS

ASH ANALYSIS ASSIGNED PROPERTY VALUES				
	Property Value ¹	Standard Deviation ²	Standard Uncertainty ³	Expanded Uncertainty ⁴
SiO ₂ , % d	58.0	0.52	0.12	0.23
Al ₂ O ₃ , % d	26.0	0.18	0.04	0.08
Fe ₂ O ₃ , % d	8.8	0.12	0.03	0.05
CaO, % d	2.61	0.026	0.006	0.012
MgO, % d	1.03	0.035	0.008	0.016
Na ₂ O, % d	0.36	0.030	0.007	0.014
K ₂ O, % d	0.90	0.021	0.005	0.010
TiO ₂ , % d	1.42	0.030	0.007	0.014
Mn ₃ O ₄ , % d	0.12	0.005	0.001	0.002
P ₂ O ₅ , % d	0.55	0.014	0.003	0.006
SO ₃ , % d	0.13	0.009	0.002	0.004
SrO, % d ⁵	0.04	0.002	0.001	0.001
BaO, % d ⁵	0.05	0.007	0.002	0.004
ZnO, % d ⁵	0.01	0.001	0.0004	0.0007
ASH FUSIBILITY ASSIGNED PROPERTY VALUES				
Reducing temperatures				
Deformation °C	1310	30	8	15
Spherical °C	1370	11	3	6
Hemispherical °C	1400	17	4	9
Flow °C	1480	27	7	15
Oxidising temperatures				
Deformation °C	1430	21	6	11
Spherical °C	1460	5	1	3
Hemispherical °C	1480	8	2	4
Flow °C	1520	18	5	10
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 (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	Standard uncertainty (u _a) is a robust value estimated at 1.25 x s*/√n.			
4	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*/√n x k where k is a coverage factor of 2 corresponding to a level of confidence of approximately 95%.			
5	Assigned property values for SrO, BaO and ZnO not included in NATA scope of accreditation.			

Date of Certification: July 2024
 CoA Issue Date: July 2024
 Report Number: ACIRS-A2-2024-CoA-rev0
 Previous ACIRS-A series: This is the second in the series and supersedes ACIRS-A1-2016



Accredited for compliance with ISO 17034

Accreditation Number 21027

*NATA is a signatory to the ILAC Mutual Recognition Arrangement for the mutual recognition of the equivalence of testing, medical testing, calibration, inspection, proficiency testing scheme providers and reference materials producers reports and certificates

1. INTRODUCTION

This certificate describes the preparation and certification of ACIRS-A2-2024. Production and certification were conducted in accordance with the technical and production requirements of ISO Guide 34 and the associated guidelines provided in ISO Guide 35 and overseen by ACIRS personnel representing the Australian Coal Preparation Society. This sample is intended to be used for quality control purposes on similar ash samples.

2. DESCRIPTION OF THE SAMPLE AND PREPARATION

ACIRS-A2-2024 comprises a sealed jar containing minimum 75g of coal ash reference material at 63µm nominal top size. This sample was prepared from a bulk subsample of approximately 165kg taken from an Australian coal fired power station. The -125µm material from this bulk sample was reignited at 815°C and then mixed by rotary divider three times. The bulk sample was then repeatedly mixed and divided by rotary sample division until samples of approximately 800g were obtained. These samples were reconstituted into 10 primary samples which were again repeatedly mixed and divided by rotary sample division until representative samples were obtained of minimum 75g each.

Homogeneity of the batch was assessed by selecting 24 bottles by stratified random sampling from the production lot. Homogeneity was assessed by analysis of SiO₂, Al₂O₃ and Fe₂O₃ by AS1038.14.3. Satisfactory sample homogeneity for this sample 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 sample. Immediately prior to weighing the subsample shall be pre-ignited to 815°C for 15 minutes, and cooled in a desiccator over freshly regenerated, self-indicating silica gel.

Note: The pre-ignition temperature is based on Australian Standard test procedures. Pre-ignition temperatures between 750°C and 815°C will not impact the characterised values of this 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 <https://acirs.com.au/products/fly-ash-reference-material/>.

4. CHARACTERISATION

ACIRS-A2-2024 was analysed as an unknown sample in an interlaboratory testing program conducted by ACIRS and through a proficiency testing program with Proficiency Testing Australia, by laboratories accredited to Australian or international standard procedures.

Characterisation was conducted by ACIRS using robust statistical techniques in accordance with the guidance 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 and proficiency testing combined dataset.
- Interlaboratory and proficiency testing data set for Ash analysis and Ash fusibility includes analyses conducted by nationally and internationally recognised test methods and in-house methods as included in Table 1 and Table 2, respectively.
- Data which did not meet ACIRS' precision criteria was excluded from the interlaboratory and proficiency 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 Ash Analysis test methods used in the interlaboratory testing for ACIRS-A2-2024

Parameter	Analysis Methods	n (total)
SiO ₂ , % d	ISO 13605* (n=14), AS1038.14.3 (n=6), AS1038.14.1 (n=1), ASTM D3682 (n=2), ASTM D4326 (n=1), ASTM D6349 (n=1), BS EN 450-1:2005+A1:2007/BS EN 196-2:2005 (n=1), in-house (n=5)	31
Al ₂ O ₃ , % d	ISO 13605* (n=14), AS1038.14.3 (n=6), AS1038.14.1 (n=1), ASTM D3682 (n=2), ASTM D4326 (n=1), BS EN 450-1:2005+A1:2007/BS EN 196-2:2005 (n=1), in-house (n=5)	30
Fe ₂ O ₃ , % d	ISO 13605* (n=14), AS1038.14.3 (n=6), AS1038.14.1 (n=1), ASTM D3682 (n=2), ASTM D4326 (n=1), ASTM D6349 (n=1), BS EN 450-1:2005+A1:2007/BS EN 196-2:2005 (n=1), in-house (n=5)	31
CaO, % d	ISO 13605* (n=13), AS1038.14.3 (n=6), ASTM D3682 (n=1), ASTM D4326 (n=1), ASTM D6349 (n=1), in-house (n=6)	28
MgO, % d	ISO 13605* (n=14), AS1038.14.3 (n=6), AS1038.14.1 (n=1), ASTM D3682 (n=2), ASTM D4326 (n=1), ASTM D6349 (n=1), BS EN 450-1:2005+A1:2007/BS EN 196-2:2005 (n=1), in-house (n=5)	31
Na ₂ O, % d	ISO 13605* (n=13), AS1038.14.3 (n=6), AS1038.14.1 (n=1), ASTM D3682 (n=2), ASTM D4326 (n=1), ASTM D6349 (n=1), BS EN 450-1:2005+A1:2007/BS EN 196-2:2005 (n=1), in-house (n=5)	30
K ₂ O, % d	ISO 13605* (n=14), AS1038.14.3 (n=6), AS1038.14.1 (n=1), ASTM D3682 (n=1), ASTM D4326 (n=1), ASTM D6349 (n=1), in-house (n=5)	29
TiO ₂ , % d	ISO 13605* (n=14), AS1038.14.3 (n=6), AS1038.14.1 (n=1), ASTM D3682 (n=1), ASTM D4326 (n=1), in-house (n=4)	27
Mn ₃ O ₄ , % d	ISO 13605* (n=13), AS1038.14.3 (n=6), AS1038.14.1 (n=1), ASTM D3682 (n=2), ASTM D4326 (n=1), ASTM D6349 (n=1), in-house (n=5)	29
P ₂ O ₅ , % d	ISO 13605* (n=14), AS1038.14.3 (n=6), AS1038.14.1 (n=1), ASTM D3682 (n=2), ASTM D4326 (n=1), ASTM D6349 (n=1), in-house (n=5)	30
SO ₃ , % d	ISO 13605* (n=14), AS1038.14.3 (n=6), AS1038.14.1 (n=1), ASTM D3682 (n=2), ASTM D4326 (n=1), in-house (n=4)	28
SrO, % d**	ISO 13605* (n=13), AS1038.14.3 (n=6), AS1038.14.1 (n=1), ASTM D3682 (n=1), ASTM D4326 (n=1), in-house (n=3)	25
BaO, % d**	ISO 13605* (n=6), AS1038.14.3 (n=6), AS1038.14.1 (n=1), ASTM D3682 (n=1), in-house (n=3)	17
ZnO, % d**	ISO 13605* (n=12), AS1038.14.3 (n=5), AS1038.14.1 (n=1), ASTM D3682 (n=1), in-house (n=4)	23

* ≤3 equivalent accredited XRF methods included.

** Not included in NATA scope of accreditation.

Table 2: Standard Ash Fusibility test methods used in the interlaboratory testing for ACIRS-A2-2024

Parameter	Analysis Methods	n (total)
Reducing temperatures		
Deformation °C	ISO 540 (n=20), AS 1038.15 (n=1), GB/T 219:2008 (n=1), Conform NEN-EN-ISO 21404 (n=1), ASTM D1857 (n=1)	24
Spherical °C	ISO 540 (n=20), AS 1038.15 (n=1), GB/T 219:2008 (n=1), Conform NEN-EN-ISO 21404 (n=1), ASTM D1857 (n=1)	24
Hemispherical °C	ISO 540 (n=20), AS 1038.15 (n=1), GB/T 219:2008 (n=1), Conform NEN-EN-ISO 21404 (n=1), ASTM D1857 (n=1)	24
Flow °C	ISO 540 (n=19), AS 1038.15 (n=1), Conform NEN-EN-ISO 21404 (n=1), ASTM D1857 (n=1)	22
Oxidising temperatures		
Deformation °C	ISO 540 (n=19), AS 1038.15 (n=1), GB/T 219:2008 (n=1), ASTM D1857 (n=1)	22
Spherical °C	ISO 540 (n=17), AS 1038.15 (n=1), GB/T 219:2008 (n=1), Conform NEN-EN-ISO 21404 (n=1), ASTM D1857 (n=1)	21
Hemispherical °C	ISO 540 (n=19), AS 1038.15 (n=1), GB/T 219:2008 (n=1), Conform NEN-EN-ISO 21404 (n=1), ASTM D1857 (n=1)	23
Flow °C	ISO 540 (n=19), AS 1038.15 (n=1), GB/T 219:2008 (n=1), ASTM D1857 (n=1)	22

5. METROLOGICAL TRACEABILITY

Property values are operationally defined by methods listed in Table 1. Traceability to SI units for SiO₂, Al₂O₃, Fe₂O₃, CaO, MgO, Na₂O, K₂O, TiO₂, Mn₃O₄, P₂O₅, SO₃, SrO, BaO, ZnO, Reducing temperature Deformation, Spherical, Hemispherical, Flow, and Oxidising temperature Deformation, Spherical, Hemispherical, Flow have not been confirmed other than through the interlaboratory study and are provided for quality control purposes on similar coal ash samples.

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 (July 2034) is provided for SiO₂, Al₂O₃, Fe₂O₃, CaO, MgO, Na₂O, K₂O, TiO₂, Mn₃O₄, P₂O₅, SO₃, SrO, BaO, ZnO, Reducing temperature Deformation, Spherical, Hemispherical, Flow, and Oxidising temperature Deformation, Spherical, Hemispherical, Flow. It is the responsibility of the user to obtain the most recent Certificate of Analysis (CoA) for this reference material available at:

<https://acirs.com.au/products/fly-ash-reference-material/>.

7. HEALTH AND SAFETY

Samples shall be handled in accordance with the Safety Data Sheet available from: <https://acirs.com.au/products/fly-ash-reference-material/>.

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
ACIRS-A2-2024	Original	22/7/2024

10. AUTHORISATION

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