

## **Reference Material Data Sheet**

for

# **SdAR-L2 Blended sediment**

## **SdAR-M2** Metal-rich sediment

# **SdAR-H1** Metalliferous sediment

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## Introduction

These SdAR reference materials were originally characterised by the assessment of data distributionsoriginating from the GeoPT proficiency testing programme where they were used as test materials as follows:SdAR-L2GeoPT37a:2015 (Webb et al., 2015a).SdAR-M2GeoPT36a:2015 (Webb et al., 2015b).SdAR-H1GeoPT35a:2014 (Webb et al., 2014).

Reference and indicative values were obtained after a detailed evaluation of data distributions as set out in the earlier 2018 IAG protocol (IAG, 2018).

However, since that time, further advances have taken place in the evaluation of GeoPT datasets as included in the updated 2020 GeoPT protocol (IAG, 2020). Specifically, this revised protocol facilitates the option (where appropriate) of using a mode as an estimate of the central location of a data distribution, in addition to the mean and median. This protocol also incorporates a revised procedure to evaluate the uncertainty of median values that provides a larger and more realistic estimate of this parameter.

Taking account of the observation that some data distributions of these reference materials were not as regular as in most silicate rocks and would, in some cases, be better served by estimation of the mode, it was considered appropriate to re-evaluate the original GeoPT datasets according to the current 2020 GeoPT protocol.

This revised data sheet, therefore, presents a re-evaluation of the original GeoPT measurement results for these three reference materials, based on the 2020 IAG GeoPT protocol. It is noted that the majority of values quoted

here are not statistically different from those on the original data sheets. However, for consistency, values included in this data sheet are all based on the re-evaluation. For convenience, and to match the recently issued SdAR aqua regia data sheets (see http://iageo.com/reference-materials/), values for all three reference materials are included in this one document.

#### **Description of the reference materials**

SdAR-L2 is a blended material produced by the US Geological Survey under the direction of Dr Stephen Wilson and designed to resemble sediment sampled when monitoring low levels of environmental contamination associated with discharges from mining operations. The material was characterised for use as a reference material through the GeoPT37a:2015 round of the International Association of Geoanalysts' GeoPT proficiency testing scheme (Webb et al., 2015a). The Proficiency Testing Steering Committee for this round was Prof. M. Thompson (statistician), Dr P.C. Webb (results coordinator), Prof. P.J. Potts and Dr S.A. Wilson. The material was tested for homogeneity by the US Geological Survey Laboratories and at The Open University and results indicated it was suitable for use in the GeoPT proficiency testing programme.

**SdAR-M2** is a blended material produced by the US Geological Survey under the direction of Dr Stephen Wilson and designed to resemble sediment sampled when monitoring moderately high levels environmental contamination associated with discharges from mining operations. The material was characterised for use as a reference material through the GeoPT36a:2015 round of the International Association of Geoanalysts' GeoPT proficiency testing scheme (Webb et al., 2015b). The Proficiency Testing Steering Committee for this round was Prof. M. Thompson (statistician), Dr P.C. Webb (results coordinator), Prof. P.J. Potts and Dr S.A. Wilson. The material was tested for homogeneity by the US Geological Survey Laboratories and at The Open University and results indicated it was suitable for use in the GeoPT proficiency testing programme.

SdAR-H1 is a blended material produced by the US Geological Survey under the direction of Dr Stephen Wilson and designed to resemble sediment sampled when monitoring high levels of environmental contamination associated with discharges from mining operations. The material was characterised for use as a reference material through the GeoPT35a:2014 round of the International Association of Geoanalysts' GeoPT proficiency testing scheme ((Webb et al., 2014). The Proficiency Testing Steering Committee for this round was Prof. M. Thompson (statistician), Dr P.C. Webb (results coordinator), Prof. P.J. Potts and Dr S.A. Wilson. The material was tested for homogeneity by the US Geological Survey Laboratories and at The Open University and results indicated it was suitable for use in the GeoPT proficiency testing programme.

An estimate of the mineralogical composition of these three materials can be found in Appendix 1.

#### Analysis of measurement results

Analysis of measurement results was based on procedures developed for the well-established GeoPT proficiency testing programme as described in the GeoPT protocol (IAG, 2020). In comparison with earlier assessments of data distributions, the revised 2020 protocol facilitated the selection of the mode as an estimate of the central location of the distribution, if that was judged to be more appropriate than the mean or median, together with a revised and more reliable estimate of the uncertainty in median values.

#### **Reference and indicative values**

*Reference and indicative values are provided for SdAR-L2 in Table 1, SdAR-M2 in Table 2 and SdAR-H1 in Table 3. These values replace those published in the earlier SdAR reference material data sheets.* 

#### Intended use

These reference materials are designed for use by laboratories undertaking environmental monitoring of the major and trace element mass concentration fractions of sediments and equivalent matrices. Intended uses include the calibration of a measurement system, the assessment of a measurement procedure, assigning values to other materials, and quality control. Note that each material may be used only for a single purpose in the same measurement process. For example, it must not be used for calibration and method validation at the same time.

#### Minimum sample size

After reviewing the range of test portion masses used to contribute measurement results to the original GeoPT proficiency testing rounds, the minimum sample size recommended for use as a test portion is 0.2 g.

#### Period of validity

Provided the storage and handling conditions are met, this reference material is not expected to deteriorate with time. On exposure to air, the material may absorb moisture, and instructions for handling must be followed.

#### **Storage information**

Store in a sealed container in a cool dry environment.

### **Instructions for handling**

Before any measurements are made, every portion of the test sample must be dried at  $105 \pm 5$  °C for at least 2 hours. Avoid contamination and cross-contamination of the test material.

### Metrological traceability and quality control

Traceability was not formally demonstrated for these reference materials. However, traceability could be demonstrated through laboratories contributing measurement results to this study through their use of certified reference materials or standard solutions as calibrators or for data validation (although this information was not recorded in this study). Traceability is also implied by the overall agreement between datasets for individual elements/oxides submitted to this programme that led to the identification of a well-defined consensus value for reasons discussed further in Potts et al. (2019).

### Safety information

Silicate powders containing heavy metals can cause harm especially if inhaled or in contact with the skin. User organisations must undertake a health and safety risk assessment and ensure that the appropriate procedures are followed in the handling and use of this material. Further details are available on the relevant Material Safety Data Sheet. The attention of users is particularly drawn to the relatively high concentration of trace elements in some of these reference materials.

## **Further information**

Further information about the SdAR materials, including some mineralogical details, may be found in the relevant GeoPT proficiency testing reports (SdAR L2: GeoPT37a:2015; SdAR M2: GeoPT36a:2015; SdAR H1: GeoPT35a:2014) (see http://www.geoanalyst.org/geopt-previous-rounds/).

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## Revisions

This information sheet is Version 2.00. Any further revisions to this reference material data sheet will made available on the IAGeo Ltd web site (www.iageo.com).

#### Acknowledgements

The contribution of Stephen Wilson (USGS) in preparing and supplying these samples is gratefully acknowledged in addition to the laboratories, who provided the measurement results to the GeoPT proficiency testing scheme on which this re-evaluation is based.

#### Approvals

This reference material data sheet was approved on behalf of the International Association of Geoanalysts.

Name Philip J Potts

Peter C Webb

Date 27th September 2020

### References

#### IAG (2018)

Protocol for the operation of the GeoPT proficiency testing scheme (2nd Edition, 2018) International Association of Geoanalysts (Keyworth, Nottingham), 18pp.

#### IAG (2020)

Protocol for the operation of the GeoPT proficiency testing scheme (2nd Edition 2018, revised 2020). International Association of Geoanalysts (Keyworth, Nottingham), 19pp.

#### Potts P.J., Webb P.C. and Thompson M. (2019)

*GeoPT proficiency testing programme as a scheme for the certification of geological reference materials. Geostandards and Geoanalytical Research, 43, 409-418* 

#### Webb, P.C., Thompson, M., Potts, P.J and Wilson, S. (2014)

*GeoPT35A - an international proficiency test for analytical geochemistry laboratories - report on round 35A (Metalliferous sediment, SdAR-H1) / August 2014. International Association of Geoanalysts: Unpublished report.* 

#### Webb, P.C., Thompson, M., Potts, P.J, Gowing, C.J.B. and Wilson, S. (2015a)

*GeoPT37A* - an international proficiency test for analytical geochemistry laboratories - report on round 37A (Blended sediment, SdAR-L2) / July 2015. International Association of Geoanalysts: Unpublished report.

#### Webb, P.C., Thompson, M., Potts, P.J and Wilson, S. (2015b)

*GeoPT36A - an international proficiency test for analytical geochemistry laboratories - report on round 36A (Metal-rich sediment, SdAR-M2) / January 2015. International Association of Geoanalysts: Unpublished report.* 

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	IAG	SdAR-	L2	Blended	Sedime	nt		
	Values for eleme	ntal/oxide mass j	fraction	ns and uncertainti	es on a dried (10	5 °C) basis		
<b>Reference values</b>					Reference values			
Oxide / element	Reference value g 100g <sup>-1</sup>	Uncertainty g 100g <sup>-1</sup>	р	Oxide / element	Reference value g 100g <sup>-1</sup>	Uncertainty g 100g <sup>-1</sup>	р	
SiO <sub>2</sub>	74.48	0.13	85	CaO	1.06	0.01	91	
TiO <sub>2</sub>	0.620	0.004	92	Na <sub>2</sub> O	2.66	0.03	90	
Al <sub>2</sub> O <sub>3</sub>	11.58	0.05	91	K <sub>2</sub> O	4.10	0.02	91	
Fe <sub>2</sub> O <sub>3</sub> T	3.64	0.02	91	P <sub>2</sub> O <sub>5</sub>	0.080	0.002	85	
MnO	0.099	0.002	92					
	mg kg <sup>-1</sup>	mg kg <sup>-1</sup>			mg kg <sup>-1</sup>	mg kg <sup>-1</sup>		
Be	3.38	0.13	37	Pr	16.2	0.4	54	
Bi	0.26	0.02	26	Rb	120	2	66	
Со	5.4	0.2	64	Sb	21.8	0.8	42	
Cr	26	2	71	Sc	5.6	0.3	55	
Cs	1.14	0.04	45	Sm	11.5	0.3	59	
Cu	50.8	1.1	75	Sr	150	2	75	
Dy	10.0	0.3	53	Та	3.81	0.22	44	
Er	6.19	0.55	52	Tb	1.58	0.07	51	
Eu	1.43	0.06	53	Th	22.0	0.7	66	
Gd	9.73	0.22	54	Tl	0.99	0.05	36	
Но	2.12	0.09	48	Tm	0.95	0.03	48	
La	67.9	1.4	66	U	3.34	0.12	58	
Li	11.8	0.6	36	V	35.0	0.9	68	
Lu	0.945	0.021	50	Y	55.5	2.0	71	
Мо	3.7	0.2	45	Yb	6.24	0.13	57	
Nd	60.3	1.5	64	Zn	201	3	77	
Ni	14.3	0.9	69	Zr	626	14	75	
Pb	183	4	72					
]	Indicative	values		II	ndicative v	alues		
Oxide / element	Indicative value g 100g <sup>-1</sup>	Uncertainty g 100g <sup>-1</sup>	р	Element	Indicative value mg kg <sup>-1</sup>	Uncertainty mg kg <sup>-1</sup>	р	
MgO	0.43	0.02	90	Ge	1.6	0.1	22	
-	mg kg <sup>-1</sup>	mg kg <sup>-1</sup>	1	Hf	16.7	0.9	53	
As	17	1	47	Hg	0.33	0.05	14	
Ba	812	20	78	In	0.47	0.03	11	
Cd	1.1	0.2	41	Nb	63	2	64	
Ga	17.5	0.2	63	W	1.6	0.3	32	
				of an 'assigned v				

Table 1 Reference and indicative values for IAG SdAR-L2

**Reference value:** Value that complies with the status of an 'assigned value' in the GeoPT protocol (2020). **Indicative value:** Value that complies with the status of a 'provisional value' in the GeoPT protocol (2020). **Uncertainty:** Robust standard deviation of the mean, median or mode expanded by a coverage factor of 2 and rounded up. The quoted uncertainty is the best estimate of the 95% confidence limit.

**p:** number of measurement results contributing to the quoted value.

 $Fe_2O_3T$ : Total mass fraction of iron expressed as  $Fe_2O_3$ .

IAG SdAR-M2 Metal Rich Sediment								
	Values for eleme	ntal/oxide mass	fractio	ns and uncertainti	ies on a dried (105	5 °C) basis		
]	Reference	values			<b>Reference values</b>			
Oxide / element	Reference value g 100g <sup>-1</sup>	Uncertainty g 100g <sup>-1</sup>	р	Element	Reference value g 100g <sup>-1</sup>	Uncertainty g 100g <sup>-1</sup>	р	
SiO <sub>2</sub>	73.45	0.17	68	MnO	0.134	0.002	78	
TiO <sub>2</sub>	0.300	0.003	78	CaO	0.84	0.01	73	
Al <sub>2</sub> O <sub>3</sub>	12.47	0.06	73	Na <sub>2</sub> O	2.59	0.03	72	
Fe <sub>2</sub> O <sub>3</sub> T	2.63	0.02	75	K <sub>2</sub> O	5.00	0.03	73	
	mg kg <sup>-1</sup>	mg kg <sup>-1</sup>			mg kg <sup>-1</sup>	mg kg <sup>-1</sup>		
Ba	990	15	71	Nd	40.0	0.6	56	
Be	6.6	0.2	31	Ni	48.8	1.3	70	
Bi	1.05	0.04	25	Pb	808	17	70	
Cd	5.1	0.3	30	Pr	11.0	0.2	42	
Ce	100	2	60	Rb	149	3	69	
Со	12.4	0.5	61	Sc	4.08	0.21	45	
Cs	1.80	0.06	41	Sm	7.18	0.15	48	
Dy	5.88	0.13	43	Sr	144	2	73	
Er	3.58	0.14	42	Та	1.76	0.12	38	
Eu	1.44	0.05	43	Tb	0.95	0.07	42	
Gd	6.3	0.2	42	Th	14.2	0.4	59	
Hf	7.3	0.3	45	Tm	0.54	0.02	40	
Но	1.21	0.03	41	U	2.53	0.10	52	
La	46.6	1.3	60	v	25.2	0.9	66	
Li	18.1	0.8	31	W	3.45	0.30	30	
Lu	0.54	0.02	43	Y	32.7	0.7	66	
Nb	26.2	0.9	60	Zn	772	19	71	
]	Indicative	values			Indicative	values		
Oxide / element	Indicative value g 100g <sup>-1</sup>	Uncertainty g 100g <sup>-1</sup>	р	Element	Indicative value mg kg <sup>-1</sup>	Uncertainty mg kg <sup>-1</sup>	р	
MgO	0.49	0.02	71	Hg	1.4	0.1	15	
P2O5	0.079	0.003	67	Мо	13.1	0.3	44	
LOI	1.62	0.05	56	Sb	111	5	34	
	mg kg <sup>-1</sup>	mg kg <sup>-1</sup>		Sn	2.4	0.3	28	
As	80	3	49	TI	2.9	0.3	30	
Cr	51	3	65	Yb	3.5	0.1	47	
Cu	239	6	69	Zr	259	8	69	
Ga	17.6	0.4	56			_		
				of an 'assigned y			<u> </u>	

Table 2 Reference and indicative values for IAG SdAR-M2

**Reference value:** Value that complies with the status of an 'assigned value' in the GeoPT protocol (2020). **Indicative value:** Value that complies with the status of a 'provisional value' in the GeoPT protocol (2020). **Uncertainty:** Robust standard deviation of the mean, median or mode expanded by a coverage factor of 2 and rounded up. The quoted uncertainty is the best estimate of the 95% confidence limit.

**p**: number of measurement results contributing to the quoted value.

 $Fe_2O_3T$ : Total mass fraction of iron expressed as  $Fe_2O_3$ .

LOI: Loss on ignition.

IAG SdAR-H1 Metalliferous Sediment									
Values for elemental/oxide mass fractions and uncertainties on a dried (105 °C) basis									
R	eference	values		F	Reference values				
Oxide / element	Reference value g 100g <sup>-1</sup>	Uncertainty g 100g <sup>-1</sup>	р	Element	Reference value g 100g <sup>-1</sup>	Uncertainty g 100g <sup>-1</sup>	р		
SiO <sub>2</sub>	65.45	0.22	71	MgO	1.53	0.02	77		
TiO <sub>2</sub>	0.560	0.005	79	CaO	1.46	0.01	78		
Al <sub>2</sub> O <sub>3</sub>	11.83	0.08	76	K <sub>2</sub> O	4.17	0.03	77		
Fe <sub>2</sub> O <sub>3</sub> T	6.45	0.05	79	P2O5	0.185	0.003	72		
MnO	0.515	0.007	79						
	mg kg <sup>-1</sup>	mg kg <sup>-1</sup>			mg kg <sup>-1</sup>	mg kg <sup>-1</sup>			
Ba	866	15	74	Ni	234	8	75		
Ce	89	3	61	Pb	3895	75	75		
Со	55.6	1.7	65	Pr	10.0	0.3	44		
Cs	4.78	0.24	40	Rb	154	3	68		
Cu	1170	12	76	Sc	8.2	0.3	48		
Dy	4.50	0.21	41	Sm	6.42	0.13	47		
Er	2.67	0.11	42	Sr	182	3	76		
Eu	1.25	0.05	43	Та	1.41	0.08	34		
Ga	15.6	0.7	52	Tb	0.78	0.03	41		
Gd	5.35	0.25	41	Tl	11.1	0.7	40		
Но	0.91	0.05	41	Tm	0.405	0.014	39		
La	44.9	1.3	60	V	73.2	2.1	72		
Li	50.5	2.5	37	Yb	2.66	0.02	45		
Lu	0.41	0.02	40	Zn	3725	60	78		
Nd	36.2	1.2	55	Zr	262	3	68		
In	dicative	values		Ι	ndicative	values			
Element	Indicative value g 100g <sup>-1</sup>	Uncertainty g 100g <sup>-1</sup>	р	Element	Indicative value mg kg <sup>-1</sup>	Uncertainty mg kg <sup>-1</sup>	р		
Na <sub>2</sub> O	1.10	0.03	75	Hf	6.9	1.0	41		
	mg kg <sup>-1</sup>	mg kg <sup>-1</sup>		In	9.5	0.6	14		
Ag	76	6	4	Мо	66	3	60		
Be	21.8	1.5	34	Nb	22	1	60		
Bi	5.1	0.4	32	Th	17.7	1.2	56		
Cd	24.4	1.1	48	U	4.2	0.3	53		
Cr	225	12	-	Ŷ	25.4	1.5	63		

Table 3 Reference and indicative values for IAG SdAR-H1

**Reference value:** Value that complies with the status of an 'assigned value' in the GeoPT protocol (2020). **Indicative value:** Value that complies with the status of a 'provisional value' in the GeoPT protocol (2020). **Uncertainty:** Robust standard deviation of the mean, median or mode expanded by a coverage factor of 2 and rounded up. The quoted uncertainty is the best estimate of the 95% confidence limit. **p:** number of measurement results contributing to the quoted value.

 $Fe_2O_3T$ : Total mass fraction of iron expressed as  $Fe_2O_3$ .

LOI: Loss on ignition.

Appendix	1	Mineralogical	details.
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SdAR-L2 – Mineralogical composition (XRD)						
	Normalised		Normalised			
Mineral	%	Mineral	%			
Quartz	43.6	Pyrite	0.2			
K-feldspar	26.2	Sphalerite	0			
Plagioclase		Total non-clay	95.4			
-albite	18.9	Clays				
-bytownite	4.2	Kaolinite (disordered)	1			
-anorthite	0	Illite	3.5			
Calcite	0.4	Muscovite	0			
Siderite	0.2	Total clays	4.6			
Amphibole	1.9	TOTAL	100			

SdAR-M2 – Mineralogical composition (XRD)						
	Normalised		Normalised			
Mineral	%	Mineral	%			
Quartz	36.5	Pyrite	0.1			
K-feldspar	33.9	Sphalerite	0			
Plagioclase		Total non-clay	93.6			
-albite	17.3	Clays				
-bytownite	2.3	Kaolinite (disordered)	0.7			
-anorthite	1.4	Illite	5.7			
Calcite	0.6	Muscovite	0			
Siderite	0.5	Total clays	6.4			
Amphibole	0.9	TOTAL	100			

SdAR-H1 – Mineralogical composition (XRD)						
Mineral	Normalised %	Mineral	Normalised %			
Quartz	39.7	Pyrite	0.3			
K-feldspar	20.2	Sphalerite	0.3			
Plagioclase		Total non-clay	74.2			
-albite	5.7	Clays				
-bytownite	0.8	Kaolinite (disordered)	1.4			
-anorthite	3.6	Illite	20.7			
Calcite	1.1	Muscovite	3.6			
Siderite	0.9	Total clays	25.8			
Amphibole	1.5	TOTAL	100			

XRD data courtesy of Dr Stephen Wilson (USGS)