



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 distributions originating from the GeoPT proficiency testing programme where they were used as test materials as follows:

SdAR-L2 GeoPT37a:2015 (Webb et al., 2015a).

SdAR-M2 GeoPT36a:2015 (Webb et al., 2015b).

SdAR-H1 GeoPT35a: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 ± 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

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Protocol for the operation of the GeoPT proficiency testing scheme (2nd Edition, 2018)
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Webb, P.C., Thompson, M., Potts, P.J and Wilson, S. (2014)

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Table 1 Reference and indicative values for IAG SdAR-L2

IAG SdAR-L2 Blended Sediment							
<i>Values for elemental/oxide mass fractions and uncertainties on a dried (105 °C) basis</i>							
Reference values				Reference values			
Oxide / element	Reference value g 100g⁻¹	Uncertainty g 100g⁻¹	p	Oxide / element	Reference value g 100g⁻¹	Uncertainty g 100g⁻¹	p
SiO ₂	74.48	0.13	85	CaO	1.06	0.01	91
TiO ₂	0.620	0.004	92	Na ₂ O	2.66	0.03	90
Al ₂ O ₃	11.58	0.05	91	K ₂ O	4.10	0.02	91
Fe ₂ O ₃ T	3.64	0.02	91	P ₂ O ₅	0.080	0.002	85
MnO	0.099	0.002	92				
	mg kg⁻¹	mg kg⁻¹			mg kg⁻¹	mg kg⁻¹	
Be	3.38	0.13	37	Pr	16.2	0.4	54
Bi	0.26	0.02	26	Rb	120	2	66
Co	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	Ta	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
Ho	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
Mo	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				Indicative values			
Oxide / element	Indicative value g 100g⁻¹	Uncertainty g 100g⁻¹	p	Element	Indicative value mg kg⁻¹	Uncertainty mg kg⁻¹	p
MgO	0.43	0.02	90	Ge	1.6	0.1	22
	mg kg⁻¹	mg kg⁻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	0.9	63	W	1.6	0.3	32

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.

Fe₂O₃T: Total mass fraction of iron expressed as Fe₂O₃.

Table 2 Reference and indicative values for IAG SdAR-M2

IAG SdAR-M2 Metal Rich Sediment							
<i>Values for elemental/oxide mass fractions and uncertainties on a dried (105 °C) basis</i>							
Reference values				Reference values			
Oxide / element	Reference value g 100g ⁻¹	Uncertainty g 100g ⁻¹	p	Element	Reference value g 100g ⁻¹	Uncertainty g 100g ⁻¹	P
SiO ₂	73.45	0.17	68	MnO	0.134	0.002	78
TiO ₂	0.300	0.003	78	CaO	0.84	0.01	73
Al ₂ O ₃	12.47	0.06	73	Na ₂ O	2.59	0.03	72
Fe ₂ O ₃ T	2.63	0.02	75	K ₂ O	5.00	0.03	73
	mg kg⁻¹	mg kg⁻¹			mg kg⁻¹	mg kg⁻¹	
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
Co	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	Ta	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
Ho	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 ⁻¹	Uncertainty g 100g ⁻¹	p	Element	Indicative value mg kg ⁻¹	Uncertainty mg kg ⁻¹	P
MgO	0.49	0.02	71	Hg	1.4	0.1	15
P ₂ O ₅	0.079	0.003	67	Mo	13.1	0.3	44
LOI	1.62	0.05	56	Sb	111	5	34
	mg kg⁻¹	mg kg⁻¹		Sn	2.4	0.3	28
As	80	3	49	Tl	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				

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.

Fe₂O₃T: Total mass fraction of iron expressed as Fe₂O₃.

LOI: Loss on ignition.

Table 3 Reference and indicative values for IAG SdAR-H1

IAG SdAR-H1 Metalliferous Sediment							
<i>Values for elemental/oxide mass fractions and uncertainties on a dried (105 °C) basis</i>							
Reference values				Reference values			
Oxide / element	Reference value g 100g ⁻¹	Uncertainty g 100g ⁻¹	p	Element	Reference value g 100g ⁻¹	Uncertainty g 100g ⁻¹	p
SiO ₂	65.45	0.22	71	MgO	1.53	0.02	77
TiO ₂	0.560	0.005	79	CaO	1.46	0.01	78
Al ₂ O ₃	11.83	0.08	76	K ₂ O	4.17	0.03	77
Fe ₂ O ₃ T	6.45	0.05	79	P ₂ O ₅	0.185	0.003	72
MnO	0.515	0.007	79				
	mg kg⁻¹	mg kg⁻¹			mg kg⁻¹	mg kg⁻¹	
Ba	866	15	74	Ni	234	8	75
Ce	89	3	61	Pb	3895	75	75
Co	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	Ta	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
Ho	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
Indicative values				Indicative values			
Element	Indicative value g 100g ⁻¹	Uncertainty g 100g ⁻¹	p	Element	Indicative value mg kg ⁻¹	Uncertainty mg kg ⁻¹	p
Na ₂ O	1.10	0.03	75	Hf	6.9	1.0	41
	mg kg⁻¹	mg kg⁻¹		In	9.5	0.6	14
Ag	76	6	4	Mo	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		Y	25.4	1.5	63

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.

Fe₂O₃T: Total mass fraction of iron expressed as Fe₂O₃.

LOI: Loss on ignition.

Appendix 1 Mineralogical details.

SdAR-L2 – Mineralogical composition (XRD)			
Mineral	Normalised %	Mineral	Normalised %
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)			
Mineral	Normalised %	Mineral	Normalised %
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)



Reference Material Data Sheet

for the selective extraction by

AQUA REGIA

of

SdAR-L2 Blended sediment

SdAR-M2 Metal-rich sediment

SdAR-H1 Metalliferous sediment

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Introduction

The extraction of selected trace elements by aqua regia is widely used, especially within the mining industry, to support geochemical exploration programmes and the management of mining operations, including the monitoring of environmental impacts. The SdAR series of three reference materials was developed to support these activities. They were each characterised as reference materials for total element contents following their distribution as test materials in the GeoPT proficiency testing programme. As a result of the statistical analysis and evaluation of contributed data, total mass fractions of major and trace elements were published in the respective reference material data sheets in 2019 (<http://iageo.com/sdar-reference-materials/>).

To extend the usefulness of these materials, new measurement results for these three reference materials were commissioned from 16 laboratories. Each laboratory was requested to provide mass fractions derived by the aqua regia selective extraction procedures that were in routine use in their laboratories. The present data sheet is designed to provide reference values for laboratories that wish to use these materials in the routine application and development of the aqua regia technique for extractions undertaken at temperatures in the range 90 to 110 °C, reflecting common procedures employed by commercial laboratories servicing the mining and geochemical exploration industries.

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 for the determination of total major and trace elements through the GeoPT37a:2015 round of the International Association of Geoanalysts' GeoPT proficiency testing scheme (Webb et al., 2015b).

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 intermediate levels of environmental contamination associated with discharges from mining operations. The material was characterised for use as a reference material for the determination of total major and trace elements through the GeoPT36a:2015 round of the International Association of Geoanalysts' GeoPT proficiency testing scheme (Webb et al., 2015a).

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 for the determination of total major and trace elements through the GeoPT35a:2014 round of the International Association of Geoanalysts' GeoPT proficiency testing scheme (Webb et al., 2014).

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

Aqua regia programme of measurement

Sixteen laboratories participated in this evaluation (**Appendix 2**). Each laboratory was sent two plastic vials each containing 5 g of the reference materials of interest (SdAR-L2, SdAR-M2 and SdAR-H1) as well as a 5 g split of a quality control certified reference material – ERM CC020 (Trace elements in contaminated river sediment). Laboratories were asked to follow their routine package of measurement based on aqua regia extraction usually with either an ICP-MS or ICP-AES finish and to report a single result for each distribution unit for relevant major and trace element mass fractions.

However, there were a number of detailed differences in the extraction procedures employed by these laboratories. For example, the mass of test portion taken for measurement varied from 0.4 to 2.0 g. Other differences included the sample to acid ratio, extraction time and temperature and even the composition of aqua regia itself (generally 3+1 : HCl+HNO₃, but some laboratories used a 2+1 or a 1+1 mixture). It is noteworthy that although extraction temperatures varied from 85 to 160 °C, the majority of laboratories (over 75%) employed a lower temperature of 90 – 110 °C, and therefore the resultant reference values are considered appropriate for extractions in this temperature range. It might be expected that all these procedural differences would affect the measurement results. However, for many measurands, an assessment of the distribution of measurement results provided a clear and well-defined consensus value that can be considered to represent a reference value characteristic of the aqua regia extraction procedure undertaken by geochemical laboratories within the stated temperature range.

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). However, it was necessary to modify these criteria to take account of the more limited number of independent aqua regia measurement results available in this study, given the provision of two results from each laboratory. In contrast to a regular GeoPT round, where participation is open to any laboratory irrespective of experience, measurement results for this study were provided by sixteen experienced laboratories following well-characterised procedures. As a consequence, there is an expectation that resultant datasets should be 'well-behaved' and characteristically unimodal with a clearly defined consensus based on a near-normal distribution of data. In these circumstances, the assessment of data for each measurand in each SdAR material also took account of, but was not in every case necessarily constrained by the following criteria to account for any incompatibility with the GeoPT protocol:

For a consensus value to be approved as **a reference value** (in addition to the routine evaluation of the quality of the data distribution):

- $n \geq 16$
- and $\geq 80\%$ values for which $|z| < 4$
- and $\geq 60\%$ values for which $|z| < 2$

For a consensus value to be approved as an **indicative value** (in addition to the routine evaluation of the quality of the data distribution):

$$n \geq 12$$

and $\geq 60\%$ values for which $|z| < 4$
and $\geq 40\%$ values for which $|z| < 2$

In these criteria, n is the total number of valid measurement results available, noting that if all participating laboratories reported results on the two distribution units provided, the maximum value of n would be 32. The parameter z is the z -score for individual results, calculated as:

$$z = (x_i - x_{pt}) / \sigma_{pt}$$

where x_i is the measurement result, x_{pt} is the organisers' best estimate of the true value of a measurand, and σ_{pt} is the corresponding standard deviation for proficiency testing, based on 'data quality 1' criteria. This approach is described in detail in the GeoPT protocol (IAG, 2020).

These additional criteria were applied in part to compensate for the fact that the evaluation provided a maximum of sixteen sets of independent results and frequently substantially fewer were available for assessment. In contrast, a minimum of fifteen independent results are required by the GeoPT protocol for the recognition of assigned values (corresponding to reference values here) and a minimum of eight independent values for the recognition of provisional values (corresponding to indicative values here) in proficiency testing.

These criteria also ensured that the distribution of measurement results generally conformed to the fitness-for-purpose criterion recognised in the GeoPT protocol for this type of measurement. This was considered essential because there were a range of detailed procedural differences in the aqua regia method employed by participating laboratories.

In combination, these criteria were designed to ensure that reference and indicative values were provided for measurands only where sufficient results are available and the distribution of results appeared to be unaffected by specific procedural differences. However, in some cases, it was necessary to apply expert judgement in the interpretation of these criteria.

For trace elements where an aqua regia extraction is expected to provide 100% or near-100% recoveries (e.g., for As, Cu, Zn), a comparison was undertaken with corresponding SdAR total mass fraction values from the reference material data sheets. This showed that none of the aqua regia data exceeded bulk compositions to a statistically significant extent.

Reference and indicative values are provided for SdAR-L2 in **Table 1**, SdAR-M2 in **Table 2** and SdAR-H1 in **Table 3**.

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 using the selective aqua regia extraction procedure undertaken within a temperature range of 90 – 110 °C that is commonly employed by geochemical laboratories. 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. The listed reference values have not been assessed for higher temperature microwave aqua regia digestion procedures, where extraction efficiencies for some elements could be higher.

Minimum sample size

After reviewing the range of test portion masses used to contribute to the present study, the minimum sample size recommended for use as a test portion is 0.5 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 ± 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 this reference material. 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).

An evaluation of potential errors in consensus values derived from results provided by participating laboratories for the co-analysed certified reference material ERM CC020 (contaminated river sediment) proved to be more problematic. This reference material (ERM, 2012) is certified for ten trace elements (As, Cd, Co, Cr, Cu, Hg, Pb, V, Ni, Zn) but is certified only for use in the assessment of results obtained using the ISO standard aqua regia extraction procedure 11466:1995. However, the majority of laboratories participating in the current characterisation study followed their own procedures and not the prescriptive requirements of ISO 11466:1995.

For the record, an assessment of results for ERM CC020 showed statistical agreement between consensus values (this study) and the certified values for As, Cd, Co, Cu, Ni and Pb, but clear bias in equivalent data for Cr, Hg, V and Zn. Because of a mismatch in the procedures used by participating laboratories and those used in the certification of ERM CC020, it was not considered appropriate to draw any further conclusions from these observations.

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, may be found in the relevant GeoPT proficiency testing reports Webb et al (2014), Webb et al (2015a) and Webb et al (2015b) which can be downloaded from <http://www.geoanalyst.org/geopt-previous-rounds/>.

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Revisions

This data sheet is Version 1.00. Any further revisions to this reference material data sheet will be made available on the IAGeo Limited web site (www.iageo.com).

Acknowledgements

The contribution of Stephen Wilson (USGS) in preparing and supplying these samples is gratefully acknowledged. The contract laboratories are thanked for providing the measurement results and supporting information on which this evaluation is based.

Approvals

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

Name	<i>Philip J Potts</i> <i>Peter C Webb</i> <i>Jennifer M Cook</i>	Date	27 September 2020
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Table 1. Reference and indicative values for the aqua regia extraction (90 – 110 °C) of IAG SdAR-L2

IAG SdAR-L2 Blended Sediment							
<i>Values for elemental/oxide mass fractions and uncertainties on a dried (105 °C) basis</i>							
Reference values				Indicative values			
Oxide / element	Reference value mg kg⁻¹	Uncertainty mg kg⁻¹	n	Element	Indicative value g 100g⁻¹	Uncertainty g 100g⁻¹	n
Ba	98	5	28	TiO₂	0.144	0.011	24
Bi	0.23	0.04	20	MnO	0.063	0.005	28
Cd	1.10	0.04	30	CaO	0.39	0.03	26
Cr	13.0	1.0	32	Na₂O	0.083	0.004	26
Cs	0.555	0.056	18	K₂O	0.27	0.02	26
Cu	52.2	1.0	32	P₂O₅	0.070	0.005	24
Er	2.83	0.26	12		mg kg⁻¹	mg kg⁻¹	
Hf	0.31	0.04	18	Ag	3.3	0.7	22
Hg	0.310	0.023	24	As	17.0	1.3	32
Ho	1.00	0.05	12	Be	1.42	0.11	28
In	0.443	0.022	18	Ce	129	9	20
La	57.7	2.6	22	Co	5.0	0.5	32
Li	8.1	0.7	20	Dy	5.5	0.7	12
Mo	3.35	0.15	30	Eu	0.69	0.08	10
Ni	12.9	0.6	32	Ga	3.8	0.5	20
Pb	168	4	32	Gd	7.2	1.1	12
Rb	14.8	1.0	22	Lu	0.33	0.04	12
Sc	2.52	0.17	22	Pr	14.4	1.5	10
Sr	14.8	0.8	24	Sb	20.0	1.9	30
Tl	0.47	0.05	22	Sm	8.8	0.6	12
U	2.01	0.17	22	Sn	1.5	0.2	26
V	26.6	1.6	30	Tb	0.91	0.12	14
W	0.60	0.04	20	Te	0.39	0.07	20
Y	24.0	1.7	22	Th	18.6	1.9	22
Yb	2.45	0.29	16				
Zn	195	4	32				

Reference value: Value that complies with the status of an ‘assigned value’ in the GeoPT protocol, taking account of the criteria listed in this document.

Indicative value: Value that complies with the status of a ‘provisional value’ in the GeoPT protocol, taking account of the criteria listed in this document.

Uncertainty: Robust standard deviation of the mean or median expanded by a coverage factor of 2 and rounded up. The number of degrees of freedom has been taken as n/2 to account for duplicate measurements in the data set. The quoted uncertainty is the best estimate of the 95% confidence limit.

n: number of measurement results noting that n includes duplicate measurements from individual laboratories.

Table 2. Reference and indicative values for the aqua regia extraction (90 – 110 °C) of IAG SdAR-M2

IAG SdAR-M2 Metal-rich Sediment							
<i>Values for elemental/oxide mass fractions and uncertainties on a dried (105 °C) basis</i>							
Reference values				Indicative values			
Oxide / element	Reference value g 100g⁻¹	Uncertainty g 100g⁻¹	n	Element	Indicative value g 100g⁻¹	Uncertainty g 100g⁻¹	n
MnO	0.118	0.005	28	TiO₂	0.064	0.005	24
CaO	0.41	0.02	26	Fe₂O₃T	2.19	0.07	30
	mg kg⁻¹	mg kg⁻¹		Na₂O	0.067	0.003	26
As	84	3	32	K₂O	0.30	0.02	26
Ba	109	5	28	P₂O₅	0.069	0.005	22
Be	4.70	0.25	28		mg kg⁻¹	mg kg⁻¹	
Bi	1.03	0.08	22	Ag	16.3	1.9	22
Cd	5.11	0.12	32	B	20.0	1.3	18
Ce	90	6	20	Cr	7.9	0.4	32
Co	12.8	0.5	32	Dy	3.5	0.5	12
Cs	0.79	0.07	18	Er	1.83	0.25	12
Cu	245	6	32	Eu	0.58	0.08	10
Hf	0.15	0.02	18	Ga	3.21	0.43	20
Hg	1.40	0.14	26	Gd	4.6	0.6	12
In	2.06	0.11	18	Ho	0.62	0.06	10
La	41.1	1.7	22	Nb	3.6	0.6	20
Li	12.9	0.8	20	Sb	99	7	30
Lu	0.208	0.012	12	Se	3.8	0.6	22
Mo	13.4	0.6	30	Sm	5.8	0.5	12
Ni	47.0	1.6	32	Tb	0.60	0.07	12
Pb	800	20	32	Th	11.3	1.2	22
Rb	15.8	0.9	22	V	14.9	1.1	30
Sc	1.91	0.12	22	Zr	4.8	0.6	20
Sn	1.23	0.14	25				
Sr	18.8	0.7	24				
Te	1.91	0.17	20				
Tl	1.94	0.16	26				
U	1.46	0.11	22				
W	1.10	0.12	22				
Y	15.4	0.7	22				
Yb	1.56	0.17	16				
Zn	792	20	32				

Reference value: Value that complies with the status of an ‘assigned value’ in the GeoPT protocol, taking account of the criteria listed in this document.

Indicative value: Value that complies with the status of a ‘provisional value’ in the GeoPT protocol, taking account of the criteria listed in this document.

Uncertainty: Robust standard deviation of the mean or median expanded by a coverage factor of 2 and rounded up. The number of degrees of freedom has been taken as n/2 to account for duplicate measurements in the data set. The quoted uncertainty is the best estimate of the 95% confidence limit.

n: number of measurement results noting that n includes duplicate measurements from individual laboratories.

Fe₂O₃T: Total extractable iron expressed as Fe₂O₃.

Table 3. Reference and indicative values for the aqua regia extraction (90 – 110 °C) of IAG SdAR-H1

IAG SdAR-H1 Metalliferous Sediment							
<i>Values for elemental/oxide mass fractions and uncertainties on a dried (105 °C) basis</i>							
Reference values				Indicative values			
Oxide / element	Reference value g 100g⁻¹	Uncertainty g 100g⁻¹	n	Element	Indicative value g 100g⁻¹	Uncertainty g 100g⁻¹	n
MnO	0.485	0.017	28	TiO₂	0.057	0.004	24
CaO	1.17	0.04	26	Fe₂O₃T	5.76	0.26	30
Na₂O	0.054	0.002	26	MgO	1.09	0.04	26
	mg kg⁻¹	mg kg⁻¹			mg kg⁻¹	mg kg⁻¹	
As	414	13	32	Ag	85	5	22
Bi	5.06	0.45	22	B	88	4	20
Cd	25.4	1.0	32	Ba	164	13	28
Ce	63.7	4.0	20	Be	21.6	1.5	28
Co	57.6	3.1	32	Dy	2.48	0.19	12
Cr	18.6	1.2	32	Er	1.16	0.11	12
Cs	2.06	0.19	20	Eu	0.75	0.07	10
Cu	1190	30	32	Gd	3.60	0.26	11
Ga	5.05	0.42	20	Hf	0.070	0.014	16
Hg	7.1	0.6	30	Ho	0.434	0.015	10
In	10.5	0.9	18	Lu	0.13	0.01	12
La	31.5	2.3	22	Nb	1.89	0.17	20
Li	41.5	2.9	20	Pb	4035	175	32
Mo	62.9	2.9	30	Pd	0.41	0.04	8
Ni	222	6	32	Pr	7.6	0.7	10
Re	0.022	0.002	14	Rb	26.0	1.9	22
S	4745	145	18	Sb	485	36	29
Sc	3.10	0.17	22	Sm	4.34	0.22	12
Se	18.8	1.3	26	Tb	0.44	0.07	12
Sn	1.77	0.16	24	Th	10.9	1.0	24
Sr	50.1	2.0	24	Tl	9.7	1.0	26
Te	9.8	0.7	20	Tm	0.16	0.02	8
U	2.01	0.14	22	V	35.0	2.3	30
W	3.91	0.41	22	Zr	3.60	0.37	20
Y	11.2	0.4	22				
Yb	0.99	0.09	16				
Zn	3780	75	32				

Reference value: Value that complies with the status of an ‘assigned value’ in the GeoPT protocol, taking account of the criteria listed in this document.

Indicative value: Value that complies with the status of a ‘provisional value’ in the GeoPT protocol, taking account of the criteria listed in this document.

Uncertainty: Robust standard deviation of the mean or median expanded by a coverage factor of 2 and rounded up. The number of degrees of freedom has been taken as n/2 to account for duplicate measurements in the data set. The quoted uncertainty is the best estimate of the 95% confidence limit.

n: number of measurement results noting that n includes duplicate measurements from individual laboratories.

Fe₂O₃T: Total extractable iron expressed as Fe₂O₃.

Appendix 1. Mineralogical details

SdAR-L2 – Mineralogical composition (XRD)			
Mineral	Normalised %	Mineral	Normalised %
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)			
Mineral	Normalised %	Mineral	Normalised %
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)

Appendix 2. Laboratories participating in the aqua regia study

Laboratory	Address
Analytisches Zentrum Berlin-Adlershof GmbH (AZBA)	Berlin, Germany
Activation Laboratories Ltd	Ancaster, Ontario, Canada
Agat Laboratories	Thunder Bay, Ontario, Canada
AGROLAB (Agar und Umwelt GmbH)	Kiel, Germany
ALS Chemex	North Vancouver, British Columbia, Canada
ALS Life Sciences Limited	Hawarden, Flintshire, UK
ALS Perth	Malaga, WA, Australia
Bundesanstalt für Materialforschung und -prüfung (BAM)	Berlin, Germany
Bureau Veritas	Vancouver, Canada
Eurofins Labtium Oy	Kuopio, Finland
Intertek Genalysis	Maddington, WA, Australia
OMAC Laboratories Limited	Loughrea, Co. Galway, Ireland
SGS North America	Denver, CO, USA
SGS Perth	Perth, WA, Australia
SGS Townsville	Townsville, Qld, Australia
SYNLAB Analytics & Services	Spremberg, Germany