

# Open Research Online

---

The Open University's repository of research publications and other research outputs

## Three New Geochemical Reference Materials for Mineral Exploration and Environmental Contamination Studies: SdARL2, SdARM2 and SdARH1

### Journal Item

How to cite:

Wilson, Stephen A.; Potts, Philip J. and Webb, Peter C. (2019). Three New Geochemical Reference Materials for Mineral Exploration and Environmental Contamination Studies: SdARL2, SdARM2 and SdARH1. *Geostandards and Geoanalytical Research* (Early Access).

For guidance on citations see [FAQs](#).

© 2019 The Authors. *Geostandards and Geoanalytical Research*; 2019 International Association of Geoanalysts

Version: Accepted Manuscript

Link(s) to article on publisher's website:  
<http://dx.doi.org/doi:10.1111/ggr.12300>

---

Copyright and Moral Rights for the articles on this site are retained by the individual authors and/or other copyright owners. For more information on Open Research Online's data [policy](#) on reuse of materials please consult the policies page.

---

[oro.open.ac.uk](http://oro.open.ac.uk)

DR. STEPHEN WILSON (Orcid ID : 0000-0002-9468-0005)

PROF. PHILIP J POTTS (Orcid ID : 0000-0002-8790-0794)

DR. PETER C WEBB (Orcid ID : 0000-0003-2467-5953)

Article type : Original Article

## **Three New Geochemical Reference Materials for Mineral Exploration and Environmental Contamination Studies: SdAR-L2, SdAR-M2 and SdAR-H1**

Stephen A. **Wilson** (1)\*, Philip J. **Potts** (2) and Peter C. **Webb** (2)

(1) Geology, Geophysics and Geochemistry Science Centre, US Geological Survey, Denver Federal Center Box 25046, MS 964. Denver, CO 80225-0046, USA

(2) Faculty of Science, Technology, Engineering and Mathematics, The Open University, Walton Hall, Milton Keynes, MK7 6AA, UK

\*Corresponding author. e-mail: [swilson@usgs.gov](mailto:swilson@usgs.gov)

### **Abstract**

The preparation and certification of three new sediment-based reference materials SdAR-L2 (Blended sediment), SdAR-M2 (Metal-rich sediment) and SdAR-H1 (Metalliferous sediment) described in this paper involved collaboration between the U.S. Geological Survey (USGS) and International

This article has been accepted for publication and undergone full peer review but has not been through the copyediting, typesetting, pagination and proofreading process, which may lead to differences between this version and the [Version of Record](#). Please cite this article as [doi: 10.1111/GGR.12300](https://doi.org/10.1111/GGR.12300)

This article is protected by copyright. All rights reserved

Association of Geoanalysts (IAG). Sediments and soils from six locations in the western United States were mixed according to a USGS-designed blending programme that calculate the proportions of source materials required to match target mass fractions and minimise the number of elements with mass fractions below instrument detection limits. For selected element mass fractions, NIST SRMs 2709–2711 provided target values. By using this approach, the three reference materials retained a natural mineralogy that is so essential for matching matrix and dissolution characteristics.

Geochemical characterisation of these materials was carried out through the IAG GeoPT proficiency testing programme. Three independent rounds of GeoPT testing were used, each providing results from over eighty laboratories. Compiled results were statistically evaluated to produce reference values for over forty elements in each material. Reference and information values as well as mineralogical compositions are presented to establish these samples as reference materials especially for use in mineral exploration and environmental contamination monitoring studies.

Keywords: United States Geological Survey, USGS, IAG, geochemical, environmental materials, geochemical reference materials, geological reference materials, sediment.

**Received 11 Jul 19 – Accepted 30 Sep 19**

The complexity of geological materials and the need for reliable quantitative analysis has led to the development of hundreds of geochemical reference materials (GRMs), each designed to matrix match specific types of geological materials (see for example, Jochum *et al.* 2016). Several international organisations such as the U.S. Geological Survey (USGS), Canadian Certified Reference Materials Project (CCRMP), National Institute for Standards and Technology (NIST), Centre de Recherches Petrographiques et Geochimiques (CRPG) and Geological Survey of Japan (GSJ) have spent considerable time and resources in the development of GRMs that are often used in quality control programmes designed to evaluate laboratory precision and accuracy. One of the limitations often encountered with single source materials is that several important trace elements are present at mass

fractions below detection limits. This limitation often requires laboratories to use large numbers of GRMs to cover the full range of elements listed in their quality control programmes. The use of so many GRMs adds significant time and expense to the measurement process. One solution to the development of GRMs with full elemental coverage is to spike a baseline material of suitable matrix with reagent grade chemicals (high purity powders or standard solutions) to achieve the desired elemental mass fractions. While this approach may be useful for spike recovery experiments, it will not adequately address possible dissolution issues associated with naturally occurring mineralogy nor other matrix-related effects. An alternative approach adopted in this investigation is to produce a composite material derived from naturally sourced geological materials from various locations that contain suitably elevated element mass fractions commonly associated with mineral resources investigations. The goal of this work is to produce reference materials that contain a range of element mass fractions typically encountered in geological investigations whilst minimising the number of elements at mass fractions at or below method detection limits.

Previous USGS investigations had identified several locations in the western United States where soil and sediment materials contained elevated mass fractions of trace elements. Several of these sites were identified as suitable candidate locations based on their accessibility, availability of large quantities of source materials and their presence on public lands.

- Sediments from the headwaters of the Animas River located near Silverton, Colorado (grid reference 37.8119°N, 107.6645°W) served as the major source of sediments containing elevated levels of trace elements. The San Juan mining district, located in south-western Colorado, produced over 200,000 tons of copper, lead, zinc and silver during its peak mining period from 1859 to 1927 (Henderson, 1926). Abandoned mining operations inundate the area, contributing significant quantities of mine waste (solid and liquid) to streams in the area. Material to prepare the reference materials described here was collected from several source locations. Several hundred kilograms of sediment material were collected along the Animas River with smaller amounts collected from the Cement and Lightner tributaries. Other areas providing important source material included
- Flat Creek near Superior, Montana;

- Stillwater mine, Nye, Montana
- Stewart Lake, Utah
- Gold ore from the Carlin Trend, Nevada
- USGS exploration reference materials GXR-3 and GXR-4.1.
- Baseline material used in material preparation came from the Platte River at a site near the entrance point to Chatfield Reservoir. This reservoir located south of Denver, Colorado is a major drinking and recreational water supply.

Determination of element mass fraction in these materials was done through the laboratory proficiency testing programme known as *GeoPT*, which is coordinated through the IAG. In this paper, the preparation and characterisation of these materials is described to establish them as reference materials especially designed for use in mineral exploration and environmental monitoring studies.

## **Experimental**

### **Evaluation of target compositions**

Having collected numerous containers of source materials, the element composition of each container was independently determined at the USGS and its contract lab (SGS Minerals, Canada). Results for each starting material were then entered into a USGS designed blending programme, which calculates the amount of each source material required to match the target composition of the blended material. Given the potential use of this material in both mineral exploration and environmental contamination studies, a target blend was required which would address both needs. Because of its widespread use the elemental profile of NIST soil reference material SRM 2710a was the target for the highest elemental mass fraction reference material, designated SdAR-H1. To produce the medium (SdAR-M2) and lower mass fraction (SdAR-L2) materials aliquots of the SdAR-H1 material were combined with additional baseline material and minor quantities of selected source materials. The objective was to broadly match elemental mass fraction in NIST SRM 2711a and SRM 2709a respectively. Minor adjustments to increase the mass fraction of specific elements in the blend for these latter materials were made when the mass fraction were otherwise predicted to be lower than method detection limits.

### **Collection and processing of materials**

Animas River sediments used in this study were collected from locations along streams in the Silverton, Colorado area in July of 2008 when low water flow provided safe access. Collection sites were selected where obvious signs (iron staining) of heavy metal mineralogy were visible. Due to stream topography sample material was often collected from shallow pools (4-10 cm) in low flow sections of the stream. Sediment material from each site was transferred to five-gallon plastic buckets using a standard shovel that was previously conditioned by digging into a sediment bed immediately downstream of the collection site. Sediments visually less than 1 cm in grain size were selected for collection. Baseline material from the Chatfield reservoir area was collected from to the top 30 cm of a sand bar located adjacent to the river bank.

Upon arrival at the USGS laboratories sediment from the Silverton area was dried for twenty-four hours in a forced air oven operating at room temperature. The drying process was designed so that the content of each bucket was dried in discrete units. The contents of each bucket were then sieved through a vibratory 2 mm sieve and the < 2mm material retained for further use. After blending calculations, specific aliquots of each component were transferred to a 10 ft<sup>3</sup> cross-flow V-blender and blended for 24 hours. Baseline material was dried in a similar manner but without the need to isolate material from individual buckets. Baseline material was combined and blended for twenty-four hours and set aside for further processing. Aliquots (20 kg) of blended Silverton and baseline material were transferred to a 30-gallon ceramic-lined ball mill, containing 80 kg of corundum grinding media (1" balls) and ground for a minimum of 15 hours per grinding period. The ground material was transferred to the V-blender and the entire lot blended for 24 hours. Blended material was then split into individual bottles using a USGS designed spinning riffler. For each of the three reference materials, a total of 2000, 80 g units were produced in this manner.

Preparation of SdAR-L2 and SdAR-M2 was performed in a slightly different manner than the SdAR-H1 material. In this process aliquots of the ground SdAR-H1 material were transferred to the ball mill along with specific amounts of the baseline material. The contents were ground for the standard

length of time, blended and split into individual bottles using the process outlined above. For each reference material produced in this manner every 100<sup>th</sup> bottle was set aside for homogeneity testing. Total element analysis of major and trace elements was performed at the USGS and its contract lab (SGS minerals, Canada). A random sample of this 100<sup>th</sup> bottle sample set was selected for mineralogical analysis at the USGS using XRD analysis

### **Characterisation using the GeoPT proficiency testing programme**

After undertaking homogeneity testing to confirm the suitability of these materials (results may be requested from the lead author), elemental composition was characterised by adopting each as a test material in successive rounds of the GeoPT proficiency testing programme, operated by the IAG. Aliquots of randomly selected bottles for each material were split into two subsamples using a standard Jones-type splitter. Each split of about 40 to 50 g was transferred into a polythene pouch which was heat sealed prior to distribution. Test samples were supplied to about 120 GeoPT participants in each round. The candidate reference material, SdAR-H1, was circulated in round 35A of the GeoPT programme, conducted in the spring of 2014 and data was reported by 89 laboratories. Statistical analysis of contributed data resulted in values being assigned for 41 measurands, as described in the relevant report (Webb *et al.* 2014). SdAR-M2 was characterised in round 36A of GeoPT, conducted in the autumn of 2014. Measurement results were reported by 84 laboratories with 48 measurands being credited with assigned status (Webb *et al.* 2015a). Finally, Round 37A of GeoPT, conducted in the spring of 2015, was used to characterise SdAR-L2. From the results reported by 95 laboratories, 49 assigned values were recognised (Webb *et al.* 2015b).

Values designated as ‘assigned’ in the GeoPT programme represent the organisers’ best estimate of the true composition of the material. Full details of the derivation of assigned values are given in the GeoPT protocol (IAG 2018b). Briefly, assigned values are consensus values obtained from a robust statistical analysis of the full data set for each measurand. A rigorous assessment is undertaken of the source data distribution, such that values are not ‘assigned’ unless at least 15 results form a unimodal distribution with about 95% of the data falling within the target uncertainty limits (U) for proficiency testing. Depending on the nature of the distribution, the mean, median or more rarely the mode may

be used as the assigned value. It must be emphasised that only when all the conditions summarised above are met is the consensus value credited with assigned status. When datasets fail to conform to all the criteria, consensus values may be regarded as an information value for the reference material. Full details of the data assessment for these three test materials may be found in the respective reports cited above.

## Results and discussion

Using information from respective *GeoPT* reports, reference material data sheets were prepared to describing the chemical and mineralogical characteristics of these materials (<http://iageo.com/sdar-reference-materials/>). It has already been established that *GeoPT* assigned values may be reliably regarded as reference values from various studies in which certified reference materials have been used as *GeoPT* test materials. In these studies, no statistically significant difference has been found between certified values and assigned values (Potts *et al.* 2015). Resultant reference values are listed in Tables 1a to 3a for SdAR-H1, SdAR-M2 and SdAR-L1. *GeoPT* consensus values that did not meet the assigned value criteria are listed as information values in Tables 1b to 3b. Uncertainties listed in these tables are the robust standard deviation of the mean or median of the respective *GeoPT* data distribution expanded by a coverage factor of two. Other metrological information may be found on the respective reference material data sheets (<http://iageo.com/sdar-reference-materials/>).

One of the targets of the present work was to prepare blended reference materials that matched the trace element composition of the well-established NIST standard reference materials SRM 2709a (San Joaquin soil), 2710a (Montana I soil) and 2711a (Montana II soil). Comparison of selected elements between SdAR-H1 and SRM 2710a, SdAR-M2 and SRM 2711a and SdAR-L2 and SRM 2709a are shown in Figures 1 to 3 respectively. Regression analysis of these data sets show correlation coefficients of 0.86 or better and slopes of the best fit line between 0.8 and 1.0. The agreement between element mass fraction in the NIST SRMs and SdAR materials suggests that the blending process used in this study was remarkably successful for these selected elements. Comparison of



uncertainties for the two sets of reference materials reveals a range of values. When elements are at a similar mass fraction the corresponding levels of uncertainty are generally comparable. It should be noted that more reference values are quoted for the SdAR materials than the combined certified and reference values of the comparable NIST materials. In particular, Be, Bi, Er, Ga, Ho, Li, Mo, Nb, Pr, Tm and Y values are available for the SdARs, but are not listed for the NIST materials.

One of the aims of the present work was to prepare reference materials with elevated levels of selected trace elements based on a blend of natural materials. Mineralogical compositions of the three materials based on USGS XRD analysis are reported in Tables 4 to 6.

## **Conclusion**

This study has presented data to show that the new reference materials, SdAR-H1, SdAR-M2 and SdAR-L2 are well characterised and fit for their intended use in the assessment of analytical measurements in support of geochemical studies, especially in the mineral exploration and environmental contamination sectors.

## **Acknowledgements**

The authors wish to thank Michael Thompson and Thomas Meisel for implementing software that permits estimation of modes by various methods and the investigation of data by analytical procedure. Estimation of modes was important in deriving information values for datasets that are significantly skewed yet exhibit a consensus.

## **References**

**Henderson C.W. (1926)**

Mining in Colorado. **U.S. Geological Survey Professional Paper, 138**, 256pp.

**IAG (2018a)**

GeoPT proficiency testing programme. International Association of Geoanalysts,  
<http://www.geoanalyst.org/overview/>

**IAG (2018b)**

Protocol for the operation of the GeoPT proficiency testing scheme. <http://www.geoanalyst.org/wp-content/uploads/2018/06/GeoPT-revised-protocol-2018.pdf>

**Jochum K.P., Weis U., Schwager B., Stoll B., Wilson S.A., Haug G.H., Andreae M.O. and Enzweiler J. (2006)**

Reference values following ISO guidelines for frequently requested rock reference materials.  
**Geostandards and Geoanalytical Research**, **40**, 333–350.

**Potts P.J., Thompson M. and Webb P.C. (2015)**

The reliability of assigned values from the GeoPT proficiency testing programme from an evaluation of data for six test materials that have been characterised as certified reference materials.

**Geostandards and Geoanalytical Research**, **39**, 407–417.

**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**. <http://www.geoanalyst.org/wp-content/uploads/2017/10/GeoPT35AReport.pdf>

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

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. <http://www.geoanalyst.org/wp->

content/uploads/2017/10/GeoPT36AReport.pdf

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

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. <http://www.geoanalyst.org/wp-content/uploads/2017/10/GeoPT37AReport.pdf>

Accepted Article

**Table 1a.****SdAR-H1 – Metalliferous sediment**

<b>Reference values</b>								
Assigned value elemental/oxide mass fractions and uncertainties from the GeoPT35a report on a dried (105 °C) basis								
Oxide / element	Reference value g 100g <sup>-1</sup>	<i>U</i> ( <i>k</i> = 2) g 100g <sup>-1</sup>	<i>n</i>		Element	Reference value mg kg <sup>-1</sup>	<i>U</i> ( <i>k</i> = 2) mg kg <sup>-1</sup>	<i>N</i>
SiO <sub>2</sub>	65.45	0.18	71		La	44.9	1.0	60
TiO <sub>2</sub>	0.560	0.004	79		Li	50.5	2.5	37
Al <sub>2</sub> O <sub>3</sub>	11.83	0.07	76		Lu	0.398	0.012	40
Fe <sub>2</sub> O <sub>3</sub> T	6.45	0.04	79		Mo	64	3	60
MnO	0.515	0.005	79		Nb	21.9	0.9	60
MgO	1.53	0.02	77		Nd	36.2	1.0	55
CaO	1.46	0.01	78		Ni	230	5	75
K <sub>2</sub> O	4.17	0.03	77		Pb	3890	80	75
P <sub>2</sub> O <sub>5</sub>	0.185	0.003	72		Pr	9.97	0.23	44
	mg kg <sup>-1</sup>	mg kg <sup>-1</sup>			Rb	152.3	2.3	68
Ba	866	12	74		Sm	6.39	0.17	47
Ce	89.3	2.1	61		Sr	182.2	2.9	76
Co	55.6	1.4	65		Ta	1.41	0.08	34
Cs	4.78	0.24	40		Tb	0.78	0.03	41
Cu	1159	16	76		Th	17.7	1.0	56
Dy	4.41	0.10	41		Tl	11.1	0.5	40
Er	2.60	0.08	42		Tm	0.394	0.013	39
Eu	1.25	0.04	43		U	4.07	0.19	53
Ga	15.6	0.7	52		V	73.2	1.7	72
Gd	5.35	0.17	41		Yb	2.60	0.09	45
Ho	0.900	0.025	41		Zn	3680	60	78

**Reference values** are the GeoPT assigned values assessed from the robust statistical analysis of results submitted to the GeoPT35a round, following an assessment of both the consistency of data distribution and the agreement between methods, where possible.

**Uncertainties**  $U$  ( $k = 2$ ) are the robust standard deviation of the mean or median of the assigned value expanded by a coverage factor of two, and rounded up.

$N$  is the number of laboratories reporting results for that element/oxide in the GeoPT35a round.

**Fe<sub>2</sub>O<sub>3</sub>T** is the total iron expressed as Fe<sub>2</sub>O<sub>3</sub>.

Table 1b.

SdAR-H1 – Metalliferous sediment

<b>Information values</b>					
Oxide / element	Value g 100g <sup>-1</sup>	<i>U</i> ( <i>k</i> = 2) g 100g <sup>-1</sup>	Element	Value mg kg <sup>-1</sup>	<i>U</i> ( <i>k</i> = 2) mg kg <sup>-1</sup>
Na <sub>2</sub> O	1.1	0.1	Hg	7	1
<i>LOI</i>	5*	–	In	9.5	0.5
	mg kg <sup>-1</sup>	mg kg <sup>-1</sup>	S	4600	330
Ag	76	6	Sb	530	50
As	390	26	Sc	8.2	0.3
Be	22	2	Se	15	3
Bi	5.1	0.4	Sn	2.9	0.6
C <sub>tot</sub>	9000	400	Te	9.5	1.0
Cd	25	1	W	13*	–
Cr	225	20	Y	25.4	1.5
<i>Ge</i>	2*	–	Zr	258	6
Hf	6.9	0.8			

**Information values** are ‘provisional’ data from the relevant *GeoPT* report with additional ‘information’ values for elements that gave a reasonably cohesive data distribution. In both cases, data distributions were not judged to be good enough to meet the criteria for designation as assigned values. These data are provided for information purposes only and **not** for the calibration of methods or the assessment of data.

**Uncertainties** *U* (*k* = 2) are the robust standard deviation of the mean or median expanded by a coverage factor of two, and rounded up.

**Indicative values** are listed in italics and marked with an asterisk (\*). These values are provided for guidance in recognition of the fact that the source *GeoPT* data set for these measurands did not provide a clear consensus.

**LOI** is the loss on ignition.

**C<sub>tot</sub>** is the total carbon.

Table 2a.  
SdAR-M2 – Metal-rich sediment

<b>Reference values</b>							
<i>Assigned value elemental/oxide mass fractions and uncertainties from the GeoPT36a report on a dried (105 °C) basis</i>							
Oxide / element	Reference value g 100g <sup>-1</sup>	<i>U</i> ( <i>k</i> = 2) g 100g <sup>-1</sup>	<i>n</i>	Element	Reference value mg kg <sup>-1</sup>	<i>U</i> ( <i>k</i> = 2) mg kg <sup>-1</sup>	<i>N</i>
SiO <sub>2</sub>	73.45	0.17	68	Ho	1.21	0.03	41
TiO <sub>2</sub>	0.300	0.003	78	La	46.6	1.0	60
Al <sub>2</sub> O <sub>3</sub>	12.47	0.06	73	Li	17.9	0.7	31
Fe <sub>2</sub> O <sub>3</sub> T	2.63	0.02	75	Lu	0.54	0.01	43
MnO	0.134	0.002	78	Mo	13.3	0.4	44
CaO	0.84	0.01	73	Nb	26.2	0.7	60
Na <sub>2</sub> O	2.58	0.03	72	Nd	39.4	0.8	56
K <sub>2</sub> O	5.00	0.03	73	Ni	48.8	1.0	70
	mg kg <sup>-1</sup>	mg kg <sup>-1</sup>		Pb	808	14	70
Ba	990	12	71	Pr	11.0	0.2	42
Be	6.6	0.2	31	Rb	149.2	1.8	69
Bi	1.05	0.03	25	Sc	4.1	0.2	45
Cd	5.1	0.2	30	Sm	7.18	0.12	48
Ce	98.8	1.7	60	Sr	143.9	2.1	73
Co	12.4	0.4	61	Ta	1.8	0.1	38
Cr	49.6	1.7	65	Tb	0.97	0.03	42
Cs	1.82	0.07	41	Th	14.2	0.4	59
Cu	236	4	69	Tm	0.54	0.02	40
Dy	5.88	0.13	43	U	2.53	0.10	52
Er	3.58	0.11	42	V	25.2	0.7	66
Eu	1.44	0.04	43	Y	32.7	0.7	66
Ga	17.6	0.4	56	Yb	3.63	0.10	47
Gd	6.28	0.14	42	Zn	760	13	71
Hf	7.29	0.23	45	Zr	259	7	69
Hg	1.44	0.09	15				

**Reference values** are the GeoPT assigned values assessed from the robust statistical analysis of results submitted to the GeoPT36a round, following an assessment of both the consistency of data distribution and the agreement between methods, where possible.

**Uncertainties** *U* (*k* = 2) are the robust standard deviation of the mean or median of the assigned value expanded by a coverage factor of two, and rounded up.

*N* is the number of laboratories reporting results for that element/oxide in the GeoPT36a round.

**Fe<sub>2</sub>O<sub>3</sub>T** is the total iron expressed as Fe<sub>2</sub>O<sub>3</sub>.

Table 2b.  
SdAR-M2 – Metal-rich sediment

<b>Information values</b>						
Oxide / element	Value	$U (k = 2)$		Element	Value	$U (k = 2)$
	g 100g <sup>-1</sup>	g 100g <sup>-1</sup>			mg kg <sup>-1</sup>	mg kg <sup>-1</sup>
MgO	0.49	0.02		In	2.1	0.2
P <sub>2</sub> O <sub>5</sub>	0.079	0.002		<i>S</i>	970	–
LOI	1.6	0.1		Sb	107	5
	mg kg <sup>-1</sup>	mg kg <sup>-1</sup>		<i>Se</i>	2.5*	–
Ag	16	3		Sn	2.4	0.2
As	76	5		Te	2.1	0.4
<i>C<sub>tot</sub></i>	2900*	–		Tl	2.8	0.2
Ge	1.5	0.2		W	3.5	0.4

**Information values** are ‘provisional’ data from the relevant *GeoPT* report with additional ‘information’ values for elements that gave a reasonably cohesive data distribution. In both cases, data distributions were judged not to be good enough to meet the criteria for designation as assigned values. These data are provided for information purposes only and **not** for the calibration of methods or the assessment of data.

**Uncertainties**  $U (k = 2)$  are the robust standard deviation of the mean or median expanded by a coverage factor of two, and rounded up.

**Indicative values** are listed in italics and marked with an asterisk (\*). These values are provided for guidance in recognition of the fact that the source *GeoPT* data set for these measurands did not provide a clear consensus.

**LOI** is the loss on ignition.

**C<sub>tot</sub>** is the total carbon.

Table 3a.

SdAR-L2 – Blended sediment

<b>Reference values</b>								
<i>Assigned value elemental/oxide and uncertainties from the GeoPT37a report on a dried (105 °C) basis</i>								
Oxide / element	Reference value g 100g <sup>-1</sup>	<i>U</i> ( <i>k</i> = 2) g 100g <sup>-1</sup>	<i>n</i>		Element	Reference value mg kg <sup>-1</sup>	<i>U</i> ( <i>k</i> = 2) mg kg <sup>-1</sup>	<i>N</i>
<b>SiO<sub>2</sub></b>	74.48	0.11	85		<b>La</b>	67.9	1.2	66
<b>TiO<sub>2</sub></b>	0.620	0.003	92		<b>Li</b>	11.8	0.5	36
<b>Al<sub>2</sub>O<sub>3</sub></b>	11.58	0.05	91		<b>Lu</b>	0.93	0.03	50
<b>Fe<sub>2</sub>O<sub>3</sub>T</b>	3.63	0.02	91		<b>Mo</b>	3.66	0.15	45
<b>MnO</b>	0.099	0.001	92		<b>Nb</b>	63.0	1.5	66
<b>CaO</b>	1.06	0.01	91		<b>Nd</b>	60.3	1.2	64
<b>Na<sub>2</sub>O</b>	2.66	0.03	90		<b>Ni</b>	14.3	0.5	69
<b>K<sub>2</sub>O</b>	4.10	0.02	91		<b>Pb</b>	183	3	72
<b>P<sub>2</sub>O<sub>5</sub></b>	0.080	0.001	85		<b>Pr</b>	16.2	0.3	54
	mg kg <sup>-1</sup>	mg kg <sup>-1</sup>			<b>Rb</b>	120	1	66
<b>As</b>	16.9	0.9	47		<b>Sb</b>	21.8	0.6	42
<b>Ba</b>	809	10	78		<b>Sc</b>	5.6	0.3	55
<b>Be</b>	3.38	0.10	37		<b>Sm</b>	11.5	0.2	59
<b>Bi</b>	0.26	0.02	26		<b>Sr</b>	150	2	75
<b>Ce</b>	140	3	68		<b>Ta</b>	3.81	0.15	44
<b>Co</b>	5.41	0.16	64		<b>Tb</b>	1.58	0.05	51
<b>Cr</b>	26.0	1.1	71		<b>Th</b>	22.0	0.6	66
<b>Cs</b>	1.14	0.04	45		<b>Tl</b>	0.99	0.04	36
<b>Cu</b>	50.8	1.1	75		<b>Tm</b>	0.92	0.04	48
<b>Dy</b>	9.83	0.28	53		<b>U</b>	3.34	0.12	58
<b>Er</b>	5.98	0.17	52		<b>V</b>	35.0	0.8	68
<b>Eu</b>	1.44	0.04	53		<b>Y</b>	54.6	1.1	71
<b>Ga</b>	17.0	0.5	63		<b>Yb</b>	6.10	0.20	57
<b>Gd</b>	9.73	0.17	54		<b>Zn</b>	201	3	77
<b>Ho</b>	2.08	0.07	48		<b>Zr</b>	618	10	75

**Reference values** are the GeoPT assigned values assessed from the robust statistical analysis of results submitted to the GeoPT37a round, following an assessment of both the consistency of data distribution and the agreement between methods, where possible.

**Uncertainties**  $U$  ( $k = 2$ ) are the robust standard deviation of the mean or median of the assigned value expanded by a coverage factor of two, and rounded up.

$N$  is the number of laboratories reporting results for that element/oxide in the GeoPT37a round.

**Fe<sub>2</sub>O<sub>3</sub>T** is the total iron expressed as Fe<sub>2</sub>O<sub>3</sub>.



Table 3b.

SdAR-L2 – Blended sediment

<b>Information values</b>					
Oxide / element	Value g 100g <sup>-1</sup>	<i>U</i> ( <i>k</i> = 2) g 100g <sup>-1</sup>	Element	Value mg kg <sup>-1</sup>	<i>U</i> ( <i>k</i> = 2) mg kg <sup>-1</sup>
<b>MgO</b>	0.43	0.01	<b>Hg</b>	0.33	0.04
<b>LOI</b>	0.92	0.07	<b>In</b>	0.47	0.03
	mg kg <sup>-1</sup>	mg kg <sup>-1</sup>	<b>S</b>	250*	–
<b>Ag</b>	3.2	0.4	<b>Se</b>	0.8*	–
<b>C<sub>tot</sub></b>	1500*	–	<b>Sn</b>	3.2	0.3
<b>Cd</b>	1.2	0.1	<b>Te</b>	0.42*	–
<b>Ge</b>	1.6	0.2	<b>W</b>	1.72	0.18
<b>Hf</b>	16	1			

**Information values** are ‘provisional’ data from the relevant *GeoPT* report with additional ‘information’ values for elements that gave a reasonably cohesive data distribution. In both cases, data distributions were judged not to be good enough to meet the criteria for designation as assigned values. These data are provided for information purposes only and **not** for the calibration of methods or the assessment of data.

**Uncertainties** *U* (*k* = 2) are the robust standard deviation of the mean or median expanded by a coverage factor of two, and rounded up.

**Indicative values** are listed in italics and marked with an asterisk (\*). These values are provided for guidance in recognition of the fact that the source *GeoPT* data set for these measurands did not provide a clear consensus.

**LOI** is the loss on ignition.

**C(tot)** is the total carbon

Table 4.

Mineralogical composition of SdAR-H1

<b>SdAR-H1</b>			
<b>Mineral</b>	<b>Normalised Conc. (%)</b>	<b>Mineral</b>	<b>Normalised Conc. (%)</b>
Quartz	39.7	Pyrite	0.3
K-feldspar	20.2	Sphalerite	0.3
Plagioclase		Total non-clay	74.2
–albite	5.7	<b>Clays</b>	
–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

Measurements were made by XRD analysis at the USGS

Table 5.

Mineralogical composition of SdAR-M2

<b>SdAR-M2</b>			
<b>Mineral</b>	<b>Normalised Conc. (%)</b>	<b>Mineral</b>	<b>Normalised Conc. (%)</b>
Quartz	36.5	Pyrite	0.1
K-feldspar	33.9		
Plagioclase		Total non-clay	93.6
–albite	17.3	<b>Clays</b>	
–bytownite	2.3	Kaolinite (disordered)	0.7
–anorthite	1.4	Illite	5.7
Calcite	0.6		
Siderite	0.5	Total clays	6.4
Amphibole	0.9	TOTAL	100

Measurements were made by XRD analysis at the USGS

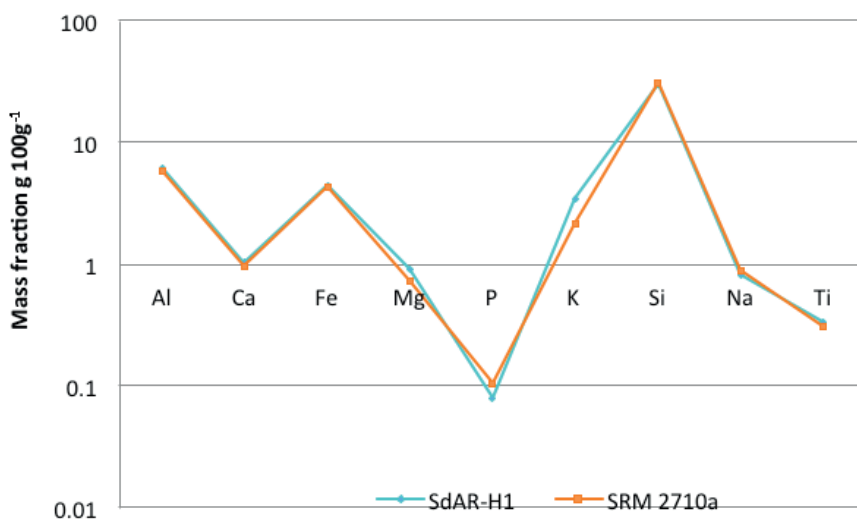
Table 6.

## Mineralogical composition of SdAR-L2

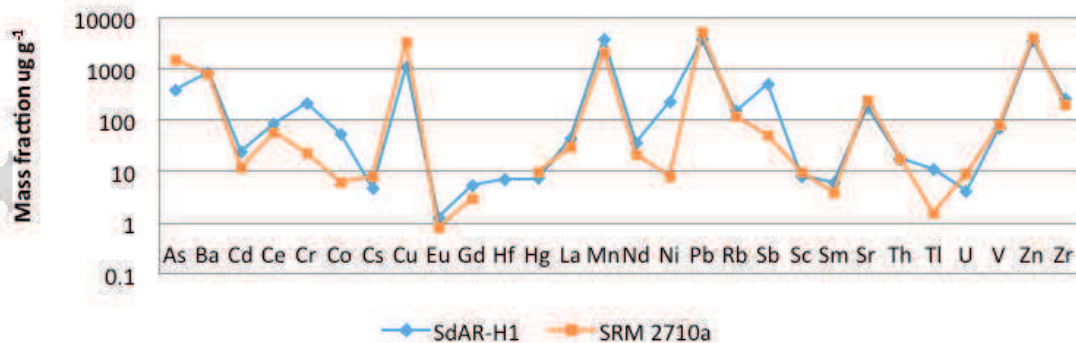
NON-CLAYS	Normalised (%)
Quartz	43.6
K feldspar (intermediate microcline)	26.2
Plagioclase (albite, var. cleavelandite)	18.9
Plagioclase (bytownite)	4.2
Calcite	0.4
Siderite	0.2
Amphibole (ferrotschermakite)	1.9
Pyrite	0.2
Total non-clays	95.4
CLAYS	
Kaolinite (disordered)	1
Illite (1Md)	3.5
Muscovite (2M1)	0
Total clays	4.6
TOTAL	100

Measurements were made by XRD analysis at the USGS

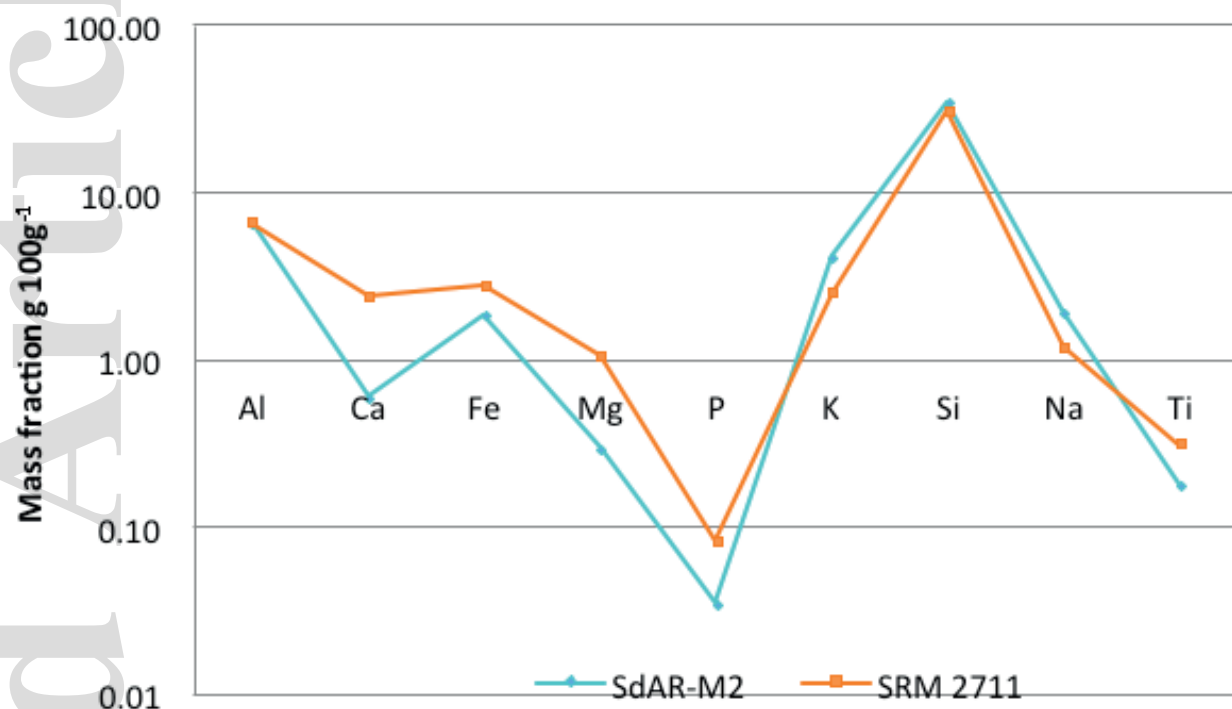
**Comparison major element mass fraction in SdAR-H1 vs SRM 2710a**



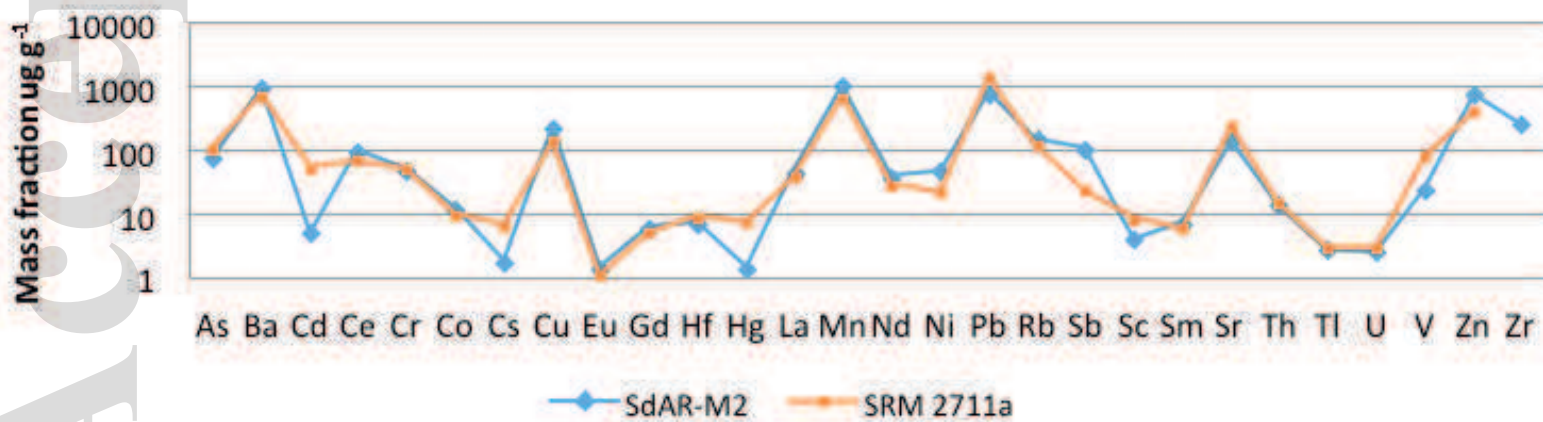
**Comparison trace element mass fraction in SdAR-H1 vs SRM 2710a**



## Comparison major element mass fraction in SdAR-M2 vs SRM 2711a



## Comparison trace element mass fraction in SdAR-M2 vs SRM 2711a

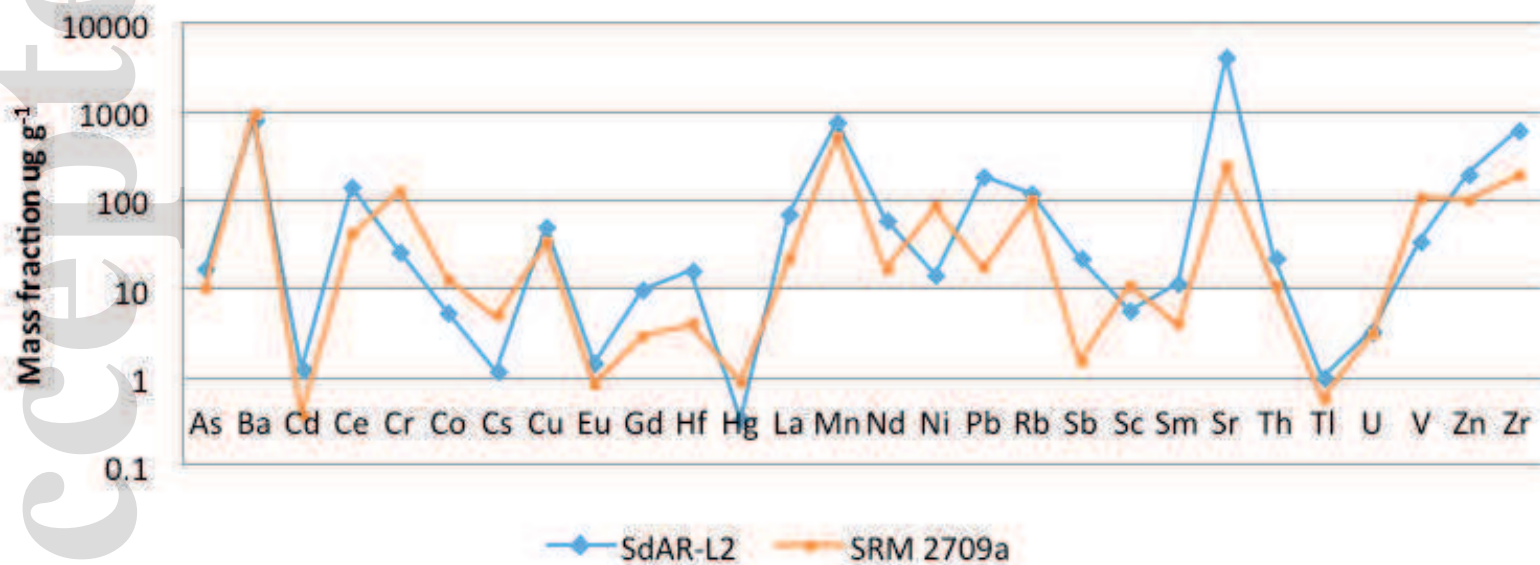


ggr\_12300\_f2.eps

## Comparison major element mass fraction in SdAR-L2 vs SRM 2709a



## Comparison trace element mass fraction in SdAR-L2 vs SRM 2709a



ggr\_12300\_f3.eps