

# Additional Provisional Elemental Values for LKSD-1, LKSD-2, LKSD-3, LKSD-4, STSD-1, STSD-2, STSD-3 and STSD-4

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Provisional values related to element concentrations derived from partial extractions as well as some additional total concentration data are reported for eight CCRMP sediment reference materials. These values are intended to supplement previously published data for these samples. The partial extractions for which data are reported include dilute  $\text{HNO}_3$  - dilute  $\text{HCl}$ , concentrated  $\text{HNO}_3$  - concentrated  $\text{HCl}$  and concentrated  $\text{HNO}_3$  - concentrated  $\text{HClO}_4$ .

Keywords: partial extractions, sediments, reference materials, compilation of data, CCRMP, LKSD/STSD.

*Des valeurs provisoires de concentrations d'éléments, mesurées après extraction partielle, ainsi que des valeurs complémentaires de concentrations totales sont présentées pour 8 matériaux sédimentaires de référence (SRM) du CCRMP. Ces valeurs ont pour but de compléter les données déjà publiées sur ces SRM. Les extractions partielles ont été faites avec les mélanges suivants:  $\text{HNO}_3$  -  $\text{HCl}$  dilué,  $\text{HNO}_3$  -  $\text{HCl}$  concentré et  $\text{HNO}_3$  -  $\text{HClO}_4$  concentré.*

*Mots-clés : extractions partielles, sédiments, matériaux de référence, compilation de données, CCRMP, LKSD/STSD.*

A large number of provisional values for the CCRMP reference materials LKSD-1, 2, 3 and 4 and STSD-1, 2, 3 and 4 have been published by Lynch (1990). These provisional values include major and minor elements expressed as oxides, total elements and elements determined using partial extractions. The partial extractions involved the use of concentrated  $\text{HNO}_3$ - $\text{HCl}$  acids and dilute  $\text{HNO}_3$ - $\text{HCl}$  acids. For the dilute  $\text{HNO}_3$ - $\text{HCl}$  extraction, data for nine elements in only the LKSD series were published. In this present compilation, provisional values for the same nine elements are reported for the STSD series.

Interest has been expressed in additional elemental data by partial extraction procedures. Provisional values for Al, Ba, Ca, Mg, P, Sr and Ti have been calculated for the eight samples using concentrated  $\text{HNO}_3$ - $\text{HCl}$  extractions. Provisional values for Al, Ba, Ca, Mg, P, Sr, As, Cr and Mo have been calculated using dilute  $\text{HNO}_3$ - $\text{HCl}$  extractions. Data for As, Cr and Mo using concentrated  $\text{HNO}_3$ - $\text{HCl}$  extractions were reported previously by Lynch (1990). Provisional values for Ag, As, Cd, Co, Cu, Mo, Ni, Pb and Zn are reported for the eight sediment samples using concentrated  $\text{HNO}_3$ - $\text{HClO}_4$  extractions.

## Analytical methods

In evaluating the data presented here, it should be noted that both partial and total digestions used by the participating laboratories (Appendix 1) varied in the conditions employed (e.g. the relative proportions of acids, sample mass taken for analysis, temperature and time of digestion). There is currently no international agreement on the standardisation of these analytical conditions and hence protocols vary from one laboratory to another.

General comments on the schemes of analysis used are as follows:

### Dilute $\text{HNO}_3$ - dilute $\text{HCl}$ (Tables 1 and 2)

Several laboratories employed 3:1  $\text{HCl}:\text{HNO}_3$  mixtures diluted with various volumes of water (usually 1 or 2 times the volume of the acid). Other laboratories used a mixture of 4 mol  $\text{l}^{-1}$   $\text{HNO}_3$  - 1 mol  $\text{l}^{-1}$   $\text{HCl}$ . In some instances the acid mixture was in contact overnight. Sample weights were either 1.0 g or 0.5 g. Heating times at 90 °C - 95 °C varied from 2 to 4 hours. Except for two laboratories which used AAS, all others employed ICP-AES as the analytical method.

Text continues on page 259

**Table 1.**  
 Partial extraction elements; dilute  
 HNO<sub>3</sub> - dilute HCl for STSD-1, 2, 3 and 4

Sample number	N	n	$\bar{x}$	s	Qualified	Outliers
<b>Ag (<math>\mu\text{g g}^{-1}</math>)</b>						
STSD-1	9	49	0.2	0.1	4	3
STSD-2	9	58	0.5	0.1	0	3
STSD-3	8	49	0.3	0.2	3	3
STSD-4	8	49	0.3	0.1	4	1
<b>Cd (<math>\mu\text{g g}^{-1}</math>)</b>						
STSD-1	6	36	0.6	0.2	0	0
STSD-2	6	35	0.7	0.1	0	1
STSD-3	6	36	0.8	0.3	0	0
STSD-4	4	24	0.3	0.1	6	2
<b>Co (<math>\mu\text{g g}^{-1}</math>)</b>						
STSD-1	9	60	14	2	0	0
STSD-2	9	55	16	2	0	4
STSD-3	9	59	14	2	0	1
STSD-4	8	58	11	1	0	0
<b>Cu (<math>\mu\text{g g}^{-1}</math>)</b>						
STSD-1	10	60	37	3	0	2
STSD-2	10	61	46	2	0	0
STSD-3	9	56	40	2	0	6
STSD-4	8	53	68	2	0	7
<b>Fe (% m/m)</b>						
STSD-1	10	59	3.3	0.3	0	3
STSD-2	10	60	3.8	0.5	0	1
STSD-3	10	62	3.2	0.4	0	0
STSD-4	9	60	2.6	0.5	0	0
<b>Mn (<math>\mu\text{g g}^{-1}</math>)</b>						
STSD-1	10	60	3760	300	0	2
STSD-2	9	59	670	80	0	2
STSD-3	9	59	2520	220	0	3
STSD-4	8	56	1160	80	0	4
<b>Ni (<math>\mu\text{g g}^{-1}</math>)</b>						
STSD-1	8	58	20	1	0	4
STSD-2	8	56	49	3	0	5
STSD-3	7	54	28	2	0	8
STSD-4	7	54	25	2	0	6
<b>Pb (<math>\mu\text{g g}^{-1}</math>)</b>						
STSD-1	10	60	35	3	0	2
STSD-2	10	57	67	4	0	4
STSD-3	10	62	42	4	0	0
STSD-4	8	58	14	4	0	2
<b>Zn (<math>\mu\text{g g}^{-1}</math>)</b>						
STSD-1	8	47	165	9	0	5
STSD-2	8	45	220	10	0	5
STSD-3	7	46	195	10	0	6
STSD-4	7	46	84	6	0	4

**Table 2.**  
 Partial extraction elements; dilute HNO<sub>3</sub> - dilute HCl  
 for LKSD-1, 2, 3 and 4 and STSD-1, 2, 3 and 4

Sample number	N	n	$\bar{x}$	s	Qualified	Outliers
<b>Al (% m/m)</b>						
LKSD-1	5	28	0.44	0.06	0	0
LKSD-2	5	28	1.57	0.15	0	0
LKSD-3	5	28	1.8	0.16	0	0
LKSD-4	5	25	1.3	0.11	0	3
STSD-1	5	26	1.13	0.1	0	2
STSD-2	5	27	3.34	0.29	0	0
STSD-3	5	28	1.69	0.15	0	0
STSD-4	4	26	1.16	0.14	0	0
<b>As (<math>\mu\text{g g}^{-1}</math>)</b>						
LKSD-1	7	45	33	2	0	5
LKSD-2	7	47	10	2	0	3
LKSD-3	7	44	25	2	0	6
LKSD-4	7	45	14	1	0	5
STSD-1	6	44	20	2	0	6
STSD-2	7	44	34	2	0	5
STSD-3	8	47	24	2	0	3
STSD-4	6	42	12	1	0	6
<b>Ba (<math>\mu\text{g g}^{-1}</math>)</b>						
LKSD-1	4	25	85	4	0	3
LKSD-2	4	26	206	9	0	2
LKSD-3	4	24	158	5	0	4
LKSD-4	5	26	126	8	0	2
STSD-1	4	23	260	6	0	5
STSD-2	4	26	100	5	0	1
STSD-3	3	23	589	17	0	3
STSD-4	3	23	906	35	0	3
<b>Ca (% m/m)</b>						
LKSD-1	5	25	734	0.38	0	2
LKSD-2	4	25	0.63	0.06	0	3
LKSD-3	4	24	0.66	0.04	0	4
LKSD-4	4	24	0.94	0.05	0	4
STSD-1	4	25	1.74	0.07	0	3
STSD-2	3	23	1.19	0.05	0	4
STSD-3	4	24	1.33	0.06	0	4
STSD-4	3	23	1.21	0.05	0	3
<b>Cr (<math>\mu\text{g g}^{-1}</math>)</b>						
LKSD-1	5	28	11	1	0	0
LKSD-2	5	28	26	3	0	0
LKSD-3	5	26	46	4	0	2
LKSD-4	5	26	19	2	0	2
STSD-1	5	28	24	3	0	0
STSD-2	5	25	43	4	0	2
STSD-3	5	28	29	3	0	0
STSD-4	4	26	28	3	0	0
<b>Mg (% m/m)</b>						
LKSD-1	5	28	0.6	0.03	0	0
LKSD-2	4	26	0.67	0.03	0	2
LKSD-3	4	26	0.88	0.04	0	2
LKSD-4	5	27	0.39	0.03	0	1
STSD-1	4	26	0.8	0.04	0	2
STSD-2	4	25	1.35	0.05	0	2
STSD-3	4	25	0.83	0.03	0	3
STSD-4	3	24	0.73	0.04	0	2

**Table 2 (continued).**  
 Partial extraction elements; dilute HNO<sub>3</sub> - dilute HCl  
 for LKSD-1, 2, 3 and 4 and STSD-1, 2, 3 and 4

Sample number	N	n	$\bar{x}$	s	Qualified	Outliers
<b>Mo (<math>\mu\text{g g}^{-1}</math>)</b>						
LKSD-1	8	58	10	2	0	0
LKSD-2	7	37	1	1	0	3
LKSD-3	6	36	1	<1	0	4
LKSD-4	6	36	2	1	0	4
STSD-1	6	36	1	<1	0	4
STSD-2	7	45	12	1	0	4
STSD-3	7	46	6	1	0	4
STSD-4	6	35	1	<1	0	3
<b>P (<math>\mu\text{g g}^{-1}</math>)</b>						
LKSD-1	3	24	575	28	0	2
LKSD-2	4	25	1010	38	0	1
LKSD-3	3	23	803	32	0	3
LKSD-4	4	23	1080	51	0	3
STSD-1	4	26	1260	64	0	0
STSD-2	4	23	1040	47	0	3
STSD-3	4	25	1150	42	0	1
STSD-4	4	26	718	40	0	0
<b>Sr (<math>\mu\text{g g}^{-1}</math>)</b>						
LKSD-1	5	25	62	2	0	3
LKSD-2	4	25	29	1	0	3
LKSD-3	4	23	28	1	0	5
LKSD-4	5	26	40	1	0	2
STSD-1	5	26	29	1	0	2
STSD-2	5	25	133	4	0	2
STSD-3	5	25	65	2	0	3
STSD-4	4	23	62	2	0	3

**Table 3.**  
 Partial extraction elements; concentrated  
 HNO<sub>3</sub> - concentrated HCl for LKSD-1, 2, 3  
 and 4 and STSD-1, 2, 3 and 4

Sample number	N	n	$\bar{x}$	s	Qualified	Outliers
<b>Al (% m/m)</b>						
LKSD-1	4	20	0.54	0.09	0	0
LKSD-2	4	19	1.87	0.25	0	0
LKSD-3	4	20	2.1	0.29	0	0
LKSD-4	4	20	1.44	0.19	0	0
STSD-1	4	19	1.28	0.14	0	0
STSD-2	3	17	3.78	0.39	0	1
STSD-3	4	20	1.98	0.28	0	0
STSD-4	3	18	1.37	0.22	0	0
<b>Ba (<math>\mu\text{g g}^{-1}</math>)</b>						
LKSD-1	5	21	85	4	0	3
LKSD-2	5	21	211	12	0	2
LKSD-3	5	24	169	10	0	0
LKSD-4	4	21	135	4	0	3
STSD-1	5	20	261	9	0	4
STSD-2	5	22	110	5	0	1
STSD-3	5	24	692	62	0	0
STSD-4	3	18	1280	81	0	4

**Table 3 (continued).**  
 Partial extraction elements; concentrated  
 HNO<sub>3</sub> - concentrated HCl for LKSD-1, 2, 3  
 and 4 and STSD-1, 2, 3 and 4

Sample number	N	n	$\bar{x}$	s	Qualified	Outliers
<b>Ca (% m/m)</b>						
LKSD-1	5	18	6.54	0.18	0	4
LKSD-2	4	16	0.67	0.06	0	2
LKSD-3	4	20	0.74	0.06	0	0
LKSD-4	4	20	0.89	0.05	0	0
STSD-1	4	18	1.64	0.07	0	2
STSD-2	3	17	1.37	0.08	0	2
STSD-3	4	18	1.33	0.06	0	2
STSD-4	3	18	1.23	0.08	0	0
<b>Mg (% m/m)</b>						
LKSD-1	4	20	0.65	0.07	0	0
LKSD-2	4	20	0.69	0.06	0	0
LKSD-3	4	20	0.92	0.1	0	0
LKSD-4	4	20	0.41	0.05	0	0
STSD-1	4	20	0.82	0.08	0	0
STSD-2	4	18	1.42	0.12	0	1
STSD-3	4	19	0.87	0.09	0	0
STSD-4	3	18	0.75	0.07	0	0
<b>P (<math>\mu\text{g g}^{-1}</math>)</b>						
LKSD-1	5	20	731	47	0	4
LKSD-2	6	23	1290	99	0	2
LKSD-3	6	25	1110	160	0	0
LKSD-4	6	24	1440	182	0	1
STSD-1	6	31	1680	221	0	1
STSD-2	6	26	1450	131	0	4
STSD-3	6	32	1610	221	0	0
STSD-4	4	26	979	90	0	2
<b>Sr (<math>\mu\text{g g}^{-1}</math>)</b>						
LKSD-1	3	18	62	4	0	4
LKSD-2	6	26	32	6	0	0
LKSD-3	6	25	36	8	0	0
LKSD-4	5	24	41	5	0	0
STSD-1	5	24	28	6	0	2
STSD-2	6	22	144	7	0	3
STSD-3	6	23	67	5	0	3
STSD-4	5	24	71	10	0	0
<b>Ti (<math>\mu\text{g g}^{-1}</math>)</b>						
LKSD-1	5	24	620	130	0	0
LKSD-2	5	24	1010	170	0	0
LKSD-3	5	24	1410	240	0	0
LKSD-4	5	24	660	86	0	0
STSD-1	5	24	540	130	0	0
STSD-2	4	22	1720	320	0	1
STSD-3	5	24	610	130	0	0
STSD-4	4	22	1070	200	0	0

**Table 4.**  
 Partial extraction elements; concentrated HNO<sub>3</sub> - concentrated HClO<sub>4</sub> for LKSD-1, 2, 3 and 4 and STSD-1, 2, 3 and 4

Sample number	N	n	$\bar{x}$	s	Qualified	Outliers
<b>Ag (<math>\mu\text{g g}^{-1}</math>)</b>						
LKSD-1	4	16	0.6	0.2	1	3
LKSD-2	4	17	0.9	0.2	0	2
LKSD-3	4	17	2.9	0.3	0	2
LKSD-4	4	16	0.2	0.1	0	3
STSD-1	4	17	0.3	0.1	1	2
STSD-2	4	16	0.6	0.2	1	3
STSD-3	3	16	0.4	0.1	0	3
STSD-4	4	16	0.4	0.1	1	3
<b>As (<math>\mu\text{g g}^{-1}</math>)</b>						
LKSD-1	5	21	33	6	0	0
LKSD-2	3	17	10	1	0	1
LKSD-3	5	21	26	4	0	0
LKSD-4	5	21	13	3	0	0
STSD-1	5	21	20	4	0	0
STSD-2	5	21	36	6	0	0
STSD-3	5	20	25	5	0	1
STSD-4	3	16	13	1	0	5
<b>Cd (<math>\mu\text{g g}^{-1}</math>)</b>						
LKSD-1	6	21	1.1	0.2	0	0
LKSD-2	4	17	0.9	0.1	0	2
LKSD-3	3	13	0.9	0.2	0	6
LKSD-4	5	19	1.9	0.2	0	2
STSD-1	4	18	0.9	0.1	1	5
STSD-2	4	19	0.9	0.1	0	2
STSD-3	5	20	1	0.1	0	3
STSD-4	4	20	0.8	0.3	0	0
<b>Co (<math>\mu\text{g g}^{-1}</math>)</b>						
LKSD-1	3	15	10	1	0	2
LKSD-2	4	17	15	2	0	0
LKSD-3	3	16	28	2	0	1
LKSD-4	3	15	10	1	0	2
STSD-1	4	16	14	1	0	1
STSD-2	4	16	17	1	0	1
STSD-3	4	17	14	1	0	0
STSD-4	4	17	11	1	0	0
<b>Cu (<math>\mu\text{g g}^{-1}</math>)</b>						
LKSD-1	7	25	42	3	0	0
LKSD-2	6	23	34	2	0	2
LKSD-3	7	23	33	2	0	2
LKSD-4	7	25	29	3	0	0
<b>Cu (<math>\mu\text{g g}^{-1}</math>) (cont.)</b>						
STSD-1	7	22	35	1	0	3
STSD-2	6	23	44	2	0	2
STSD-3	7	24	36	3	0	1
STSD-4	7	25	65	4	0	0
<b>Mo (<math>\mu\text{g g}^{-1}</math>)</b>						
LKSD-1	6	21	11	4	2	2
LKSD-2	4	19	2	1	1	0
LKSD-3	4	17	1	<1	0	0
LKSD-4	5	17	2	<1	0	2
STSD-1	5	19	2	<1	1	0
STSD-2	6	22	11	2	0	1
STSD-3	6	21	7	1	2	0
STSD-4	5	18	1	1	1	1
<b>Ni (<math>\mu\text{g g}^{-1}</math>)</b>						
LKSD-1	5	21	12	3	0	3
LKSD-2	7	25	23	4	0	0
LKSD-3	7	25	45	4	0	0
LKSD-4	7	25	31	5	0	0
STSD-1	6	21	21	2	0	2
STSD-2	6	22	50	4	0	3
STSD-3	7	25	29	4	0	0
STSD-4	7	25	24	4	0	0
<b>Pb (<math>\mu\text{g g}^{-1}</math>)</b>						
LKSD-1	6	23	82	5	0	2
LKSD-2	6	21	38	2	0	4
LKSD-3	4	20	24	2	0	5
LKSD-4	6	23	89	4	0	2
STSD-1	6	23	35	3	0	2
STSD-2	5	21	65	2	0	4
STSD-3	5	20	40	1	0	5
STSD-4	5	21	12	2	0	4
<b>Zn (<math>\mu\text{g g}^{-1}</math>)</b>						
LKSD-1	5	22	311	10	0	3
LKSD-2	5	21	188	7	0	4
LKSD-3	5	22	132	6	0	3
LKSD-4	5	22	174	7	0	3
STSD-1	5	22	161	8	0	3
STSD-2	6	24	222	18	0	1
STSD-3	5	22	184	10	0	3
STSD-4	5	22	85	5	0	3

**Table 5.**  
 "Total" elements

Sample number	N	n	$\bar{x}$	s	Qualified	Outliers
<b>Cd (<math>\mu\text{g g}^{-1}</math>)</b>						
LKSD-1	7	34	1.4	0.3	0	1
LKSD-2	6	31	1	0.5	1	0
LKSD-3	4	19	0.8	0.6	3	1
LKSD-4	8	30	2	0.2	0	4
STSD-1	7	30	0.8	0.2	2	4
STSD-2	7	26	0.7	0.3	0	3
STSD-3	7	29	1.1	0.3	2	4
STSD-4	5	20	0.5	0.3	0	1
<b>Cl (<math>\mu\text{g g}^{-1}</math>)</b>						
LKSD-1	3	29	556	67	0	1
LKSD-2	3	22	333	47	6	2
LKSD-3	3	17	222	20	8	5
LKSD-4	3	27	269	49	0	3
STSD-1	3	19	283	53	9	2
STSD-2	3	24	213	26	5	1
STSD-3	3	23	381	30	3	3
STSD-4	2	18	171	35	0	2

**Table 5 (continued).**  
**"Total" elements**

Sample number	N	n	$\bar{x}$	s	Qualified	Outliers	Sample number	N	n	$\bar{x}$	s	Qualified	Outliers
<b>Ga (<math>\mu\text{g g}^{-1}</math>)</b>							<b>Ho (<math>\mu\text{g g}^{-1}</math>)</b>						
LKSD-1	6	41	10	4	4	1	LKSD-1	4	23	1	0.3	0	4
LKSD-2	6	39	18	3	0	3	LKSD-2	4	25	2.1	0.8	0	2
LKSD-3	6	42	17	4	0	0	LKSD-3	3	16	1.1	0.2	0	1
LKSD-4	5	30	12	3	0	2	LKSD-4	4	26	1.6	0.8	0	1
STSD-1	5	29	16	3	0	3	STSD-1	4	23	1.4	0.3	0	4
STSD-2	6	38	24	4	0	4	STSD-2	4	24	2.1	1	0	3
STSD-3	6	40	17	4	0	2	STSD-3	3	15	1.3	0.4	0	2
STSD-4	6	38	16	4	0	4	STSD-4	3	15	0.9	0.1	0	2
<b>Gd (<math>\mu\text{g g}^{-1}</math>)</b>							<b>Tm (<math>\mu\text{g g}^{-1}</math>)</b>						
LKSD-1	3	17	3.6	0.7	0	0	LKSD-1	3	15	0.35	0.07	1	2
LKSD-2	3	15	9.4	1.2	0	2	LKSD-2	3	16	0.79	0.19	0	1
LKSD-3	3	17	7.5	1.1	0	0	LKSD-3	3	17	0.57	0.18	0	0
LKSD-4	3	17	5.1	0.7	0	0	LKSD-4	3	17	0.44	0.13	0	0
STSD-1	3	16	5.9	1	0	1	STSD-1	3	17	0.7	0.23	0	0
STSD-2	3	17	7.4	1.3	0	0	STSD-2	3	17	0.75	0.27	0	0
STSD-3	3	17	6.5	0.9	0	0	STSD-3	3	16	0.67	0.25	0	1
STSD-4	3	17	4.3	0.8	0	0	STSD-4	3	17	0.49	0.16	0	0

**Table 6.**  
**Compilation of data by the different extraction procedures for the LKSD and STSD series reference materials**

Sample number	Total		Conc. HCl-conc. HNO <sub>3</sub>		Dilute HCl-dilute HNO <sub>3</sub>		Conc. HNO <sub>3</sub> -HClO <sub>4</sub>	
	$\bar{x}$	s	$\bar{x}$	s	$\bar{x}$	s	$\bar{x}$	s
<b>Ag (<math>\mu\text{g g}^{-1}</math>)</b>								
LKSD-1	0.6	0.05	0.6	0.2	0.6	0.2	0.6	0.2
LKSD-2	0.8	0.06	0.8	0.2	0.8	0.1	0.9	0.2
LKSD-3	2.7	0.11	2.4	0.4	2.8	0.3	2.9	0.3
LKSD-4	< 0.5	-	0.2	0.1	0.2	0.1	0.2	0.1
STSD-1	< 0.5	-	0.3	0.1	0.2	0.1	0.3	0.1
STSD-2	0.5	0.08	0.5	0.2	0.5	0.1	0.6	0.2
STSD-3	< 0.5	-	0.4	0.1	0.3	0.2	0.4	0.1
STSD-4	< 0.5	-	0.3	0.1	0.3	0.1	0.4	0.1
<b>Al (% m/m)</b>								
LKSD-1	4.13	0.11	0.54	0.09	0.44	0.06	no data	-
LKSD-2	6.51	0.21	1.87	0.25	1.57	0.15	no data	-
LKSD-3	6.61	0.21	2.10	0.29	1.80	0.16	no data	-
LKSD-4	3.12	0.21	1.44	0.19	1.30	0.11	no data	-
STSD-1	4.76	0.16	1.28	0.14	1.13	0.10	no data	-
STSD-2	8.52	0.21	3.78	0.39	3.34	0.29	no data	-
STSD-3	5.77	0.11	1.98	0.28	1.69	0.15	no data	-
STSD-4	6.40	0.05	1.37	0.22	1.16	0.14	no data	-
<b>As (<math>\mu\text{g g}^{-1}</math>)</b>								
LKSD-1	40	2	30	6	33	2	33	6
LKSD-2	11	1	9	3	10	2	10	1
LKSD-3	27	2	23	6	25	2	26	4
LKSD-4	16	1	12	3	14	1	13	3
STSD-1	23	2	17	5	20	2	20	4
STSD-2	42	3	32	8	34	2	36	6
STSD-3	28	2	22	6	24	2	25	5
STSD-4	15	1	11	3	12	1	13	1
<b>Ba (<math>\mu\text{g g}^{-1}</math>)</b>								
LKSD-1	430	40	85	4	85	4	no data	-
LKSD-2	780	75	211	12	206	9	no data	-
LKSD-3	680	55	169	10	158	5	no data	-
LKSD-4	330	55	135	4	126	8	no data	-

**Table 6 (continued).**  
Compilation of data by the different extraction procedures for the LKSD and STSD series reference materials

Sample number	Total		Conc. HCl-conc. HNO <sub>3</sub>		Dilute HCl-dilute HNO <sub>3</sub>		Conc. HNO <sub>3</sub> - HClO <sub>4</sub>	
	$\bar{x}$	s	$\bar{x}$	s	$\bar{x}$	s	$\bar{x}$	s
<b>Ba (<math>\mu\text{g g}^{-1}</math>) (cont.)</b>								
STSD-1	630	51	261	9	260	6	no data	-
STSD-2	540	43	110	5	100	5	no data	-
STSD-3	1490	120	692	62	589	17	no data	-
STSD-4	2000	222	1280	81	906	35	no data	-
<b>Ca (% m/m)</b>								
LKSD-1	7.72	0.43	6.54	0.18	7.34	0.38	no data	-
LKSD-2	1.57	0.14	0.67	0.06	0.63	0.06	no data	-
LKSD-3	1.64	0.14	0.74	0.06	0.66	0.04	no data	-
LKSD-4	1.29	0.14	0.89	0.05	0.94	0.05	no data	-
STSD-1	2.57	0.21	1.64	0.07	1.74	0.07	no data	-
STSD-2	2.86	0.29	1.37	0.08	1.19	0.05	no data	-
STSD-3	2.36	0.14	1.33	0.06	1.33	0.06	no data	-
STSD-4	2.86	0.21	1.23	0.08	1.21	0.05	no data	-
<b>Cd (<math>\mu\text{g g}^{-1}</math>)</b>								
LKSD-1	1.4	0.3	1.2	0.3	1.2	0.1	1.1	0.2
LKSD-2	1.0	0.5	0.8	0.2	0.6	0.2	0.9	0.1
LKSD-3	0.8	0.6	0.6	0.3	0.4	0.2	0.9	0.2
LKSD-4	2.0	0.2	1.9	0.5	1.9	0.2	1.9	0.2
STSD-1	0.8	0.2	0.8	0.2	0.6	0.2	0.9	0.1
STSD-2	0.7	0.3	0.8	0.3	0.7	0.1	0.9	0.1
STSD-3	1.1	0.3	1.0	0.2	0.8	0.3	1.0	0.1
STSD-4	0.5	0.3	0.6	0.3	0.3	0.1	0.8	0.3
<b>Co (<math>\mu\text{g g}^{-1}</math>)</b>								
LKSD-1	11	1	9	1	8	1	10	1
LKSD-2	17	1	17	1	16	2	15	2
LKSD-3	30	2	30	2	30	3	28	2
LKSD-4	11	1	11	1	9	1	10	1
STSD-1	17	1	14	2	14	2	14	1
STSD-2	19	2	17	1	16	2	17	1
STSD-3	16	1	14	1	14	2	14	1
STSD-4	13	1	11	1	11	1	11	1
<b>Cr (<math>\mu\text{g g}^{-1}</math>)</b>								
LKSD-1	31	3	12	2	11	1	no data	-
LKSD-2	57	8	29	3	26	3	no data	-
LKSD-3	87	8	51	5	46	4	no data	-
LKSD-4	33	6	21	2	19	2	no data	-
STSD-1	67	9	28	3	24	3	no data	-
STSD-2	116	13	50	9	43	4	no data	-
STSD-3	80	10	34	6	29	3	no data	-
STSD-4	93	14	30	6	28	3	no data	-
<b>Cu (<math>\mu\text{g g}^{-1}</math>)</b>								
LKSD-1	44	5	44	5	44	2	42	3
LKSD-2	37	4	36	3	36	2	34	2
LKSD-3	35	3	34	3	34	2	33	2
LKSD-4	31	4	30	3	31	2	29	3
STSD-1	36	4	36	2	37	3	35	1
STSD-2	47	5	43	3	46	2	44	2
STSD-3	39	4	38	2	40	2	36	3
STSD-4	65	6	66	5	68	2	65	4
<b>Fe (% m/m)</b>								
LKSD-1	2.9	0.1	1.8	0.3	1.8	0.1	no data	-
LKSD-2	4.4	0.2	3.5	0.3	3.7	0.5	no data	-
LKSD-3	4.0	0.2	3.5	0.3	3.6	0.5	no data	-
LKSD-4	2.9	0.2	2.7	0.3	2.6	0.3	no data	-

**Table 6 (continued).**  
**Compilation of data by the different extraction procedures for the LKSD and STSD series reference materials**

Sample number	Total		Conc. HCl-conc. HNO <sub>3</sub>		Dilute HCl-dilute HNO <sub>3</sub>		Conc. HNO <sub>3</sub> - HClO <sub>4</sub>	
	$\bar{x}$	<i>s</i>	$\bar{x}$	<i>s</i>	$\bar{x}$	<i>s</i>	$\bar{x}$	<i>s</i>
<b>Fe (% m/m) (cont.)</b>								
STSD-1	4.5	0.5	3.5	0.2	3.3	0.3	no data	-
STSD-2	5.2	0.3	4.1	0.4	3.8	0.5	no data	-
STSD-3	4.3	0.4	3.4	0.1	3.2	0.4	no data	-
STSD-4	4.0	0.4	2.6	0.3	2.6	0.5	no data	-
<b>Mg (% m/m)</b>								
LKSD-1	1.04	0.07	0.65	0.07	0.60	0.03	no data	-
LKSD-2	1.01	0.08	0.69	0.06	0.67	0.03	no data	-
LKSD-3	1.20	0.06	0.92	0.10	0.88	0.04	no data	-
LKSD-4	0.56	0.04	0.41	0.05	0.39	0.03	no data	-
STSD-1	1.33	0.07	0.82	0.08	0.80	0.04	no data	-
STSD-2	1.88	0.10	1.42	0.12	1.35	0.05	no data	-
STSD-3	1.33	0.06	0.87	0.09	0.83	0.03	no data	-
STSD-4	1.28	0.05	0.75	0.07	0.73	0.04	no data	-
<b>Mn (<math>\mu\text{g g}^{-1}</math>)</b>								
LKSD-1	700	30	460	60	410	35	no data	-
LKSD-2	2020	100	1840	180	1840	65	no data	-
LKSD-3	1440	80	1220	230	1300	55	no data	-
LKSD-4	500	30	430	30	420	9	no data	-
STSD-1	3950	270	3740	430	3760	300	no data	-
STSD-2	1060	60	720	120	670	80	no data	-
STSD-3	2730	210	2630	140	2520	220	no data	-
STSD-4	1520	90	1200	130	1160	80	no data	-
<b>Mo (<math>\mu\text{g g}^{-1}</math>)</b>								
LKSD-1	10	2	12	2	10	2	11	4
LKSD-2	< 5	-	2	0.7	1	1	2	1
LKSD-3	< 5	-	2	0.9	1	0	1	0
LKSD-4	< 5	-	2	0.6	2	1	2	0
STSD-1	< 5	-	2	0.5	1	0	2	0
STSD-2	13	2	13	2	12	1	11	2
STSD-3	6	2	7	2	6	1	7	1
STSD-4	< 5	-	2	0.6	1	0	1	1
<b>Ni (<math>\mu\text{g g}^{-1}</math>)</b>								
LKSD-1	16	3	11	1	12	2	12	3
LKSD-2	26	4	23	3	23	3	23	4
LKSD-3	47	5	44	4	46	4	45	4
LKSD-4	31	5	32	5	31	4	31	5
STSD-1	24	5	18	3	20	1	21	2
STSD-2	53	6	47	4	49	3	50	4
STSD-3	30	6	25	3	28	2	29	4
STSD-4	30	5	23	2	25	2	24	4
<b>P (<math>\mu\text{g g}^{-1}</math>)</b>								
LKSD-1	700	40	731	47	575	28	no data	-
LKSD-2	1220	130	1290	99	1010	38	no data	-
LKSD-3	1090	40	1110	160	803	32	no data	-
LKSD-4	1440	130	1440	180	1080	51	no data	-
STSD-1	1660	90	1690	220	1260	64	no data	-
STSD-2	1400	90	1450	130	1040	47	no data	-
STSD-3	1570	40	1610	220	1150	42	no data	-
STSD-4	960	40	979	90	718	4	no data	-
<b>Pb (<math>\mu\text{g g}^{-1}</math>)</b>								
LKSD-1	82	5	84	10	83	7	82	5
LKSD-2	44	4	40	7	34	4	38	2
LKSD-3	29	3	26	5	21	3	24	2
LKSD-4	91	6	93	8	91	6	89	4

**Table 6 (continued).**  
 Compilation of data by the different extraction procedures for the LKSD and STSD series reference materials

Sample number	Total		Conc. HCl-conc. HNO <sub>3</sub>		Dilute HCl-dilute HNO <sub>3</sub>		Conc. HNO <sub>3</sub> - HClO <sub>4</sub>	
	$\bar{x}$	<i>s</i>	$\bar{x}$	<i>s</i>	$\bar{x}$	<i>s</i>	$\bar{x}$	<i>s</i>
<b>Pb (<math>\mu\text{g g}^{-1}</math>) (cont.)</b>								
STSD-1	35	3	34	4	35	3	35	3
STSD-2	66	4	66	7	67	4	65	2
STSD-3	40	3	39	5	42	4	40	1
STSD-4	16	3	13	4	14	4	12	2
<b>Sb (<math>\mu\text{g g}^{-1}</math>)</b>								
LKSD-1	1.2	0.1	1.2	0.4	no data	-	no data	-
LKSD-2	1.1	0.1	1.2	0.5	no data	-	no data	-
LKSD-3	1.3	0.1	1.4	0.5	no data	-	no data	-
LKSD-4	1.7	0.1	1.5	0.6	no data	-	no data	-
STSD-1	3.3	0.3	2.0	1.0	no data	-	no data	-
STSD-2	4.8	0.4	2.6	1.5	no data	-	no data	-
STSD-3	4.0	0.4	2.4	1.2	no data	-	no data	-
STSD-4	7.3	0.7	3.6	2.5	no data	-	no data	-
<b>Sr (<math>\mu\text{g g}^{-1}</math>)</b>								
LKSD-1	250	53	62	4	62	2	no data	-
LKSD-2	220	41	32	6	29	1	no data	-
LKSD-3	240	42	36	8	28	1	no data	-
LKSD-4	110	38	41	5	40	1	no data	-
STSD-1	170	42	28	6	29	1	no data	-
STSD-2	400	65	144	7	133	4	no data	-
STSD-3	230	52	67	5	65	2	no data	-
STSD-4	350	60	71	10	62	2	no data	-
<b>Ti (<math>\mu\text{g g}^{-1}</math>)</b>								
LKSD-1	3010	260	619	126	no data	-	no data	-
LKSD-2	3460	450	1010	166	no data	-	no data	-
LKSD-3	3330	330	1410	236	no data	-	no data	-
LKSD-4	2270	470	660	86	no data	-	no data	-
STSD-1	4600	370	539	130	no data	-	no data	-
STSD-2	4870	400	1720	319	no data	-	no data	-
STSD-3	4400	390	614	130	no data	-	no data	-
STSD-4	4530	270	1070	202	no data	-	no data	-
<b>V (<math>\mu\text{g g}^{-1}</math>)</b>								
LKSD-1	50	5	27	3	no data	-	no data	-
LKSD-2	77	8	48	10	no data	-	no data	-
LKSD-3	82	8	55	13	no data	-	no data	-
LKSD-4	49	8	32	10	no data	-	no data	-
STSD-1	98	15	47	11	no data	-	no data	-
STSD-2	101	10	58	14	no data	-	no data	-
STSD-3	134	18	61	22	no data	-	no data	-
STSD-4	106	10	51	19	no data	-	no data	-
<b>Zn (<math>\mu\text{g g}^{-1}</math>)</b>								
LKSD-1	331	22	337	11	335	18	311	10
LKSD-2	209	18	200	6	205	10	188	7
LKSD-3	152	14	139	10	151	5	132	6
LKSD-4	194	19	189	10	195	16	174	7
STSD-1	178	16	165	8	165	9	161	8
STSD-2	246	21	216	15	220	10	222	18
STSD-3	204	16	192	11	195	10	184	10
STSD-4	107	12	82	8	84	6	85	5



### Concentrated HNO<sub>3</sub> - concentrated HCl (Table 3)

Four laboratories reported using 3:1 mixtures of concentrated HCl - concentrated HNO<sub>3</sub>; sample weights were either 0.5 g or 1.0 g. Three laboratories used the reverse ratio of acids with 1 g samples.

### Concentrated HNO<sub>3</sub> - concentrated HClO<sub>4</sub> (Table 4)

Sample weights varied from 0.2 g to 1.0 g. Various mixtures of HNO<sub>3</sub> - HClO<sub>4</sub> were added to the samples and heated to HClO<sub>4</sub> fumes. Fuming was maintained for periods of 1 to 4 hours. The ratios of the two acids varied from 75% HNO<sub>3</sub> - 25% HClO<sub>4</sub> to 15% HNO<sub>3</sub> - 85% HClO<sub>4</sub>. Atomic absorption spectroscopy was used by seven laboratories. One laboratory used ICP-AES and one laboratory determined just arsenic using a colorimetric method.

### Total extraction elements (Table 5)

The methods of extraction for Cd, Ga, Gd, Ho and Tm involved the use of the following acid mixtures:

1. HNO<sub>3</sub> - HClO<sub>4</sub> - HCl - HF
2. HNO<sub>3</sub> - HClO<sub>4</sub> - HF
3. HNO<sub>3</sub> - HCl - HF
4. HNO<sub>3</sub> - HF
5. HNO<sub>3</sub> - HClO<sub>4</sub> - HF + LiBO<sub>2</sub> fusion of insoluble residue.

One laboratory employed a pyrohydrolysis method for the dissolution of Cl. The methods of determination were AAS, DCP-AES, ICP-AES, ICP-MS, INAA, XRF and ion chromatography.

## Results and compiled data

The provisional values reported in Tables 1 to 5 were calculated in the manner described previously by Lynch (1990). The contents of each table are as follows:

Table 1. Elements derived from the analysis of dilute HNO<sub>3</sub> - dilute HCl acid extractions of STSD-1, -2, -3 and -4, the LKSD data having been published previously.

Table 2. Elements derived from the analysis of dilute HNO<sub>3</sub> - dilute HCl extractions of LKSD-1, -2, -3 and -4 and STSD-1, -2, -3 and -4.

Table 3. Elements derived from the analysis of concentrated HNO<sub>3</sub> - concentrated HCl extractions of LKSD-1, -2, -3 and -4 and STSD-1, -2, -3, and -4.

Table 4. Elements derived from the analysis of concentrated HNO<sub>3</sub> - concentrated HClO<sub>4</sub> acid extraction of LKSD-1, -2, -3 and -4 and STSD-1, -2, -3 and -4.

Table 5. Total elements for LKSD-1, -2, -3 and -4 and STSD-1, -2, -3 and -4.

Table 6. Compilation of all data by multiple extractions for the LKSD and STSD series. This table contains previously published as well as new data.

In these tables:

N = number of laboratories after outliers have been removed.

n = number of determinations after outliers have been removed.

$\bar{x}$  = mean.

s = standard deviation.

Qualified = number of values reported below the detection limit. If more than 50% of the values from a laboratory were below the detection limit for a given element in a particular sample, all of the values for that element in that sample were deleted and did not appear in either of the last columns. If the qualified values were not discarded, they were set arbitrarily to one half of the detection limit.

Outliers = values which were deleted by the two step trimming method.

Given the significant differences in the exact conditions used for the four categories of digestion (Tables 2-5), the overall precision of these results is surprisingly good.

## Acknowledgement

Thanks are due to G.E.M. Hall for critically reading the manuscript and providing encouragement for this project. Thanks also are due to Maureen Leaver and William Bowman of CCRMP, CANMET. This paper is Geological Survey of Canada Contribution Number 1996380.

† With regret, John Lynch passed away on 16th July 1998. Revisions to his manuscript were prepared by Gwendy Hall and Phil Potts.

## Reference

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**Lynch J. (1990)**

Provisional elemental values for eight new geochemical lake sediment and stream sediment reference materials LKSD-1, LKSD-2, LKSD-3, LKSD-4, STSD-1, STSD-2, STSD-3 and STSD-4. *Geostandards Newsletter*, 14, 153-167.

## Appendix 1. Participating laboratories

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Acme Analytical Laboratories, Vancouver, British Columbia, Canada.

Analytical Service Laboratories Ltd., Vancouver, British Columbia, Canada.

Atlantic Industrial Research Institute, Halifax, Nova Scotia, Canada.

Atomic Energy Canada Ltd., Kanata, Ontario, Canada.

Barringer Laboratories Ltd., Mississauga, Ontario, Canada.

CanTech Laboratories, Calgary, Alberta, Canada.

Bondar-Clegg and Company Ltd., Ottawa, Ontario, Canada.

Bondar-Clegg and Company Ltd., North Vancouver, British Columbia, Canada.

Cdn. Resource Laboratories Ltd., Delta, British Columbia, Canada.

Chemex Labs Ltd., North Vancouver, British Columbia, Canada.

Chimitec Ltee, Ste-Foy, Quebec, Canada.

Government of Canada, Environment Canada, EPS/DFO Laboratory Services, West Vancouver, British Columbia, Canada.

Government of Canada, Geological Survey of Canada, Analytical Chemistry Section, Ottawa, Ontario, Canada.

Government of Canada, Geological Survey of Canada, Applied Geochemistry Section, Ottawa, Ontario, Canada.

Government of Newfoundland and Labrador, Department of Mines, St. Johns, Newfoundland, Canada.

Government of Ontario, Ministry of Natural Resources, Toronto, Ontario, Canada.

Government of Switzerland, Swiss Federal Laboratories for Materials Testing and Research, Bern 30520, Switzerland.

Min En Laboratories Ltd., North Vancouver, British Columbia, Canada.

The Mineral Lab, Lakewood, CO, USA.

Monenco Analytical Laboratories, Calgary, Alberta, Canada.

Vangeochem Lab Limited, North Vancouver, British Columbia, Canada.