



Industry, Energy and Technology

Mines

TILL-GEOCHEMISTRY OF THE PUDDLE POND, STAR LAKE, RAINY LAKE (NTS MAP AREAS 12A/05, 11 AND 14), AND ADJACENT AREAS

J.S. Organ

Open File 012A/1843

**St. John's, Newfoundland
September, 2020**

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SUMMARY

Analytical results for 405, <63 μ till samples and 30 field duplicates from west-central Newfoundland, (NTS map areas 12A/05 and parts of NTS 12A/04, 06 and 11–14) are released. These samples have been analyzed by ICP-OES for aluminum, arsenic, barium, beryllium, cadmium, calcium, chromium, cobalt, copper, dysprosium, iron, lanthanum, lead, lithium, magnesium, manganese, molybdenum, nickel, niobium, phosphorus, potassium, scandium, silver, sodium, strontium, sulphur, titanium vanadium, yttrium, zinc, and zirconium; and by INAA for antimony, arsenic, barium, bromine, cerium, cesium, chromium, cobalt, europium, gold, iron, hafnium, lanthanum, lutetium, molybdenum, rubidium, scandium, samarium, selenium, sodium, strontium, tantalum, terbium, thorium, tungsten, uranium, ytterbium and zirconium. Loss-on-ignition (LOI) was determined gravimetrically, while fluoride was analyzed by ion-selective electrode after alkaline fusion. The analyses have been quality checked for acceptable accuracy and precision. Interpretations of the geochemical data along with an interpretation of the surficial geology will be released later as two separate Open File reports.

INTRODUCTION

This report provides the results of a till-geochemistry survey conducted in 2016 and 2018 in the following NTS map areas: Puddle Pond (12A/05; road networks only), the western half of Star Lake (12A/11) and the southern part of Rainy Lake (12A/14), as well as along the borders of neighbouring NTS map areas 12A/04, 06, 12 and 13 (Figure 1). The samples were collected as part of an ongoing till-geochemistry and surficial-mapping program across the island of Newfoundland. The primary objective is to assist the mineral exploration industry by delineating prospective areas using both till geochemical anomalies and regional ice-flow history. Organ and Dyke (2019) have summarized the surficial mapping work and ice-flow history for the Puddle Pond, Star Lake and Rainy Lake NTS map areas (12A/05, 11 and 14). The field survey work was conducted using truck and ATV traverses, as well as helicopter support in remote areas.

Using aerial photography and ground-truth data, a map of the surficial geology and landforms for the surveyed areas will be released at a later date, at a scale of 1:50 000, along with an interpretation of the geochemical data.

This report comprises notes on location data contained in the database, followed by description of methods of sampling, sample preparation, and analytical processes, including quality assurance.

LOCATION DATA

The location for each sample is given in Appendix A, as Universal Transverse Mercator (UTM) easting and northings (Zone 21; NAD 27). A short description of each sample and site is also included.

SAMPLING METHODS

Till samples collected from NTS map area 12A/11 and 14, and from the forest-access roads of map area 12A/05 and the boundaries of 12A/04, 06, 12 and 13 are shown on Figure 1. The num-

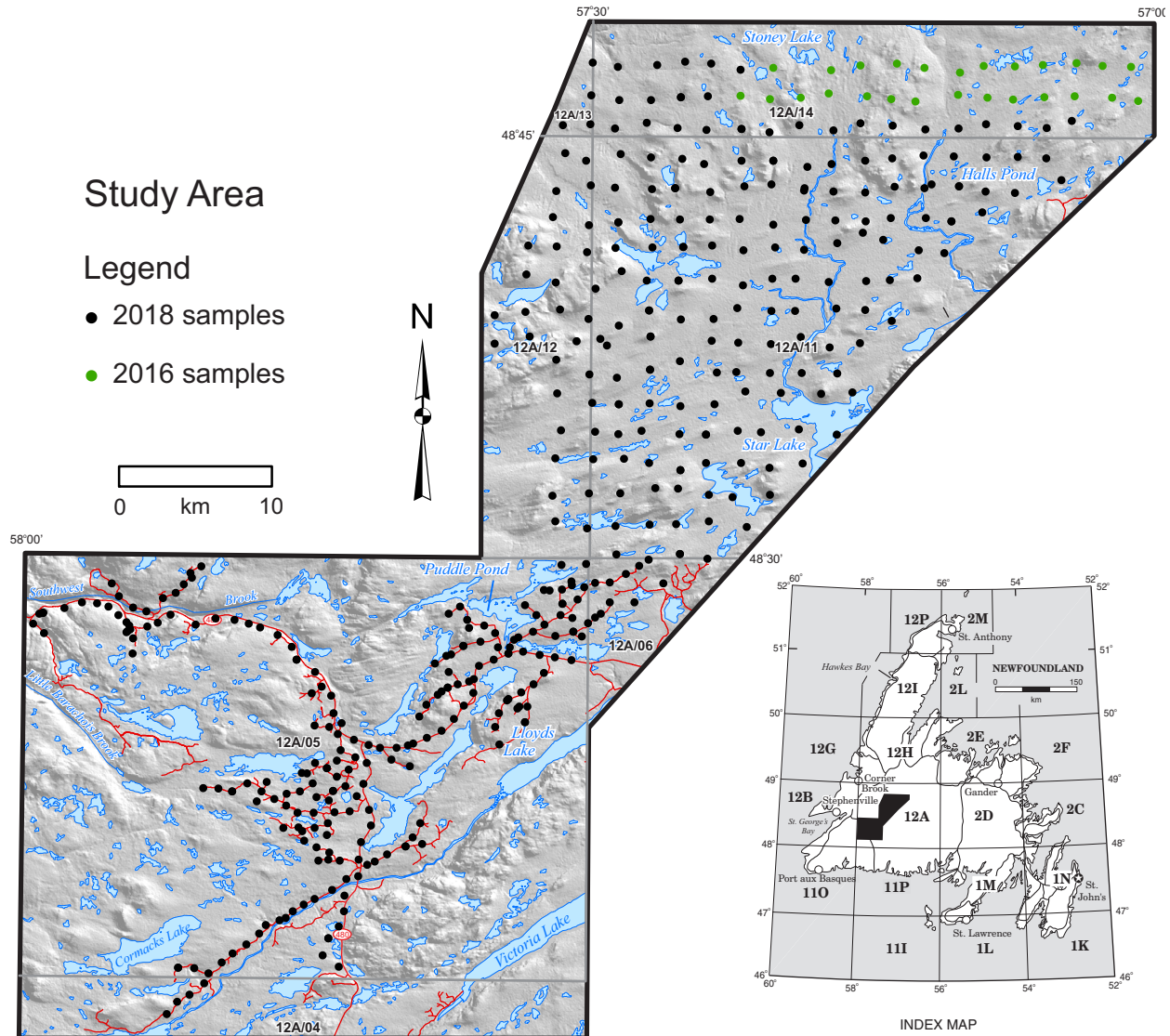


Figure 1. Maps showing location of study area, and of till geochemical samples released with current open file. Green dots: 2016 samples; black dots: 2018 samples.

number of samples collected in each NTS map area is given in Table 1.

Approximately 1 kg of till was collected, and placed in Kraft paper bags, from the C- or BC- soil horizons exposed in hand-dug pits, mudboils, roadcuts or ditches. Sample spacing was determined by access along existing roadways and the availability of appropriate sample material. Along forest-resource and other roads, the sample density was one sample every 1 linear kilometre. In remote areas,

Table 1. Samples by NTS map area

NTS Map Area	Map Sheet Name	Number of Routine Till Sample Sites
12A/04	King George IV Lake	4
12A/05	Puddle Pond	176
12A/06	Victoria Lake	9
12A/11	Star Lake	135
12A/12	Little Grand Lake	26
12A/13	Corner Brook	3
12A/14	Rainy Lake	52

only accessible *via* helicopter, sample density averaged one sample per 4 km². Field duplicates were collected at 30 sites, at an overall frequency of 1 in 13, to estimate the natural inhomogeneity of the sample medium. The results of the field-duplicate analyses are summarized in a later section.

SAMPLE PREPARATION METHODS AND ANALYSIS

Samples were processed and analyzed in the geochemical laboratory of the Geological Survey of Newfoundland and Labrador (GSNL) in St. John's. The samples were air-dried at 60°C, and dry-sieved through 63 µm (230 mesh) stainless-steel sieves to recover the silt and clay fraction for analysis.

The geochemical analysis of 61 elements from the silt and clay fraction of 435 C- or BC- soil horizon samples, collected in 2016 and 2018, make up the database in Appendix A. The GSNL laboratory carried out inductively-coupled plasma optical emission spectrometry (ICP-OES) following a multi-acid (HF/HCL/HNO₃/HClO₄) digestion for Al, As, Ba, Be, Ca, Cd, Ce, Co, Cr, Cu, Dy, Fe, K, La, Li, Mg, Mn, Mo, Na, Nb, Ni, P, Pb, S, Sc, Sr, Ti, V, Y, Zn and Zr. Note that sulphur analysis was only performed on samples collected in 2018.

Instrumental neutron activation analysis (INAA) was carried out by Maxxam Laboratories (now named Bureau Veritas) in Mississauga, Ontario, for the following elements: As, Au, Ba, Br, Ce, Co, Cr, Cs, Eu, Fe, Hf, La, Lu, Mo, Na, Nd, Rb, Sb, Sc, Se, Sm, Sr, Ta, Tb, Th, U, W, Yb and Zr.

Of the 61 elements determined, 12 were determined by both ICP-OES and INAA: As, Ba, Ce, Co, Cr, Fe, La, Mo, Na, Rb, Sc and Zr.

Analyses for silver, fluoride, and loss-on ignition (LOI) were also completed at the GSNL laboratory. Silver was analyzed by ICP-OES after nitric acid digestion. Fluoride was analyzed by ion-selective electrode (ISE) after an alkaline fusion, and LOI was determined gravimetrically.

Analytical variables are labelled in this report and in the database with a combination of element symbol name and a numeric suffix (1 – INAA with no digestion; 2 – ICP-OES after multi-acid (HF/HCl/ HNO₃/HClO₄) digestion; 6 – ICP-OES after nitric acid digestion; 9 – ISE after alkaline fusion) indicating analytical method; the unit of measurement is also given. Detection limits in the database are replaced by a value that is ½ of the detection limit. A complete list of analytical variables is given in Table 2, and the analytical methods are described, in detail, in Finch *et al.* (2018).

QUALITY ASSURANCE

Quality assurance in the lab consisted of insertion of one certified reference standard (TILL-1, TILL-2, TILL-3, TILL4), Lynch, 1996; and OREAS-46 and OREAS-47 (www.ore.com.au), and one analytical duplicate, in every sequence of 20 samples. Standard analyses for both analytical methods in 2016 and 2018 and duplicate analyses for INAA and ICP-OES, were mostly satisfactory and re-analyses were not requested. Control charts are included as Appendix B.

Table 2. Geochemical variables with analytical method, units, detection limit (D.L.), number of analyses below the detection limit (<D.L.) and range of data values. Eight elements had multiple detection limits and are listed separately in the detection limit column. The suffix “1” denotes INAA; “2” denotes ICP-OES after multi-acid digestion; “6” denotes ICP-OES after nitric acid digestion; and “9” denotes ISE after alkaline fusion

Element	Method	Units	D.L.	<D.L.	Max	Min	Element	Method	Units	D.L.	<D.L.	Max	Min
Ag6	ICP-OES	ppm	0.1	435	<0.1	<0.1	Mg2	ICP-OES	%	0.01	0	9.55	0.14
Al2	ICP-OES	%	0.01	0	11.4	3.8	Mn2	ICP-OES	ppm	1	0	1297	158.9
As1	INAA	ppm	0.5	0	148.0	0.6	Mo1	INAA	ppm	1	377	26	<1
As2	ICP-OES	ppm	1, 2	14	137.8	<1	Mo2	ICP-OES	ppm	1	303	24.3	<1
Au1	INAA	ppb	1, 2,5	305	24	<1	Na1	INAA	%	0.1	0	3.1	0.3
Ba1	INAA	ppm	50	0	1000	110	Na2	ICP-OES	%	0.05	0	2.97	0.27
Ba2	ICP-OES	ppm	1	0	1025.6	89.9	Nb2	ICP-OES	ppm	1	0	56.1	1.7
Be2	ICP-OES	ppm	0.1	0	9.2	0.6	Ni2	ICP-OES	ppm	1	0	401.2	5.1
Br1	INAA	ppm	1	0	281	2	P2	ICP-OES	ppm	1	0	3305.8	79.2
Ca2	ICP-OES	%	0.01	0	5.00	0.31	Pb2	ICP-OES	ppm	1	1	235.6	<1
Cd2	ICP-OES	ppm	0.1	35	1.7	<0.1	Rb1	INAA	ppm	5	6	120	<5
Ce1	INAA	ppm	3	0	260	15	Rb2	ICP-OES	ppm	1, 5	0	114.5	5.8
Ce2	ICP-OES	ppm	1, 5	0	214.1	16.9	S2	ICP-OES	ppm	5	0	1105.9	29.5
Co1	INAA	ppm	2, 4 5	25	79	<2	Sb1	INAA	ppm	0.1	10	1.4	<0.1
Co2	ICP-OES	ppm	1	0	85.8	1.8	Sc1	INAA	ppm	0.1	0	35.8	3.4
Cr1	INAA	ppm	10	4	990.0	<10	Sc2	ICP-OES	ppm	0.1	0	37.8	3.1
Cr2	ICP-OES	ppm	1	0	775.4	9.4	Se1	INAA	ppm	1, 2, 3, 4	434	3	<1
Cs1	INAA	ppm	0.5	52	3.9	<0.5	Sm1	INAA	ppm	0.1	0	12.9	2.3
Cu2	ICP-OES	ppm	1	0	201.8	2.7	Sr2	ICP-OES	ppm	1	0	661.7	46
Dy2	ICP-OES	ppm	0.1, 0.5	0	12.7	1.8	Ta1	INAA	ppm	0.2	3	4.3	<0.2
Eu1	INAA	ppm	0.5	77	2.9	<0.5	Tb1	INAA	ppm	0.5	8	2.5	<0.5
F9	ISE	ppm	5	0	916.0	22.0	Th1	INAA	ppm	0.1	1	40.1	2.5
Fe1	INAA	%	0.1	0	9.3	1.1	Ti2	ICP-OES	ppm	1, 5	0	15626.5	1859
Fe2	ICP-OES	%	0.01	0	10.1	1.0	U1	INAA	ppm	0.1	0	10	0.7
Hf1	INAA	ppm	1	1	35	<1	V2	ICP-OES	ppm	1	0	289.1	16.8
K2	ICP-OES	%	0.01	0	2.63	0.15	W1	INAA	ppm	1	261	5	<1
La1	INAA	ppm	1	0	74	6.0	Y2	ICP-OES	ppm	1	0	67.8	10.5
La2	ICP-OES	ppm	1	0	71.1	7.2	Yb1	INAA	ppm	0.5	1	12	<0.5
Li2	ICP-OES	ppm	0.1	0	31.1	1.4	Zn2	ICP-OES	ppm	1	0	512.5	14.3
LOI	Gravimetric	%	0.1	0	55.1	1.3	Zr1	INAA	ppm	100, 270	86	900	<100
Lu1	INAA	ppm	0.05	1	1.8	<0.05	Zr2	ICP-OES	ppm	1	0	354.2	15.9

ACCURACY

Comparison of the standard analyses with ‘recommended values’ for ICP-OES (based on the arithmetic means and standard deviations of multiple re-analyses) indicate that the multi-acid digestion is near total (>95% recovery) for Ba, Ca, Co, Cu, Fe, Li, Mn, Na, Ni, P, Pb, Rb, Sc, Sr and V, but only partial (<75%) for Y and Zr (Table 3). The greatest underestimations are for Zr (19% of the recommended values). For several elements, the recovery is greater than 100%, indicating that the element is being overestimated; the greatest overestimation is for Sr (average 119% of the recommended values). Overall, only nine elements (Ba, Cu, Fe, Li, Mn, Na, P, Pb and Rb) out of 30 show recoveries within $\pm 5\%$ of 100%. Standards OREAS-46 and OREAS-47 were each used only once in the ICP-OES dataset; therefore, it is not possible to create control charts. Analyses of these standards are shown in Table 4.

Not surprisingly, near-total analyses by INAA are more numerous because they are not dependent on mineral solubility in a digestion reagent. Recoveries of 95% or less were only

Table 3. Accuracy of ICP-OES analyses; calculated as the arithmetic mean of multiple analyses of each certified reference standard, divided by the recommended value for the standard

	TILL-1	TILL-2	TILL-3	TILL-4	Arithmetic Mean
Al	0.92	0.93	0.93	0.93	93%
As	0.89	0.95	0.93	0.95	93%
Ba	1.03	1.00	1.02	1.02	102%
Be	0.65	0.90	0.66	0.88	77%
Ca	0.94	0.98	0.97	0.99	97%
Ce	0.95	0.87	0.95	0.84	90%
Co	1.12	1.16	1.10	1.13	113%
Cr	0.94	0.93	0.90	0.84	90%
Cu	1.02	1.06	1.03	1.07	105%
Fe	1.00	1.01	1.01	1.01	101%
K	1.00	0.90	0.91	0.90	93%
La	0.94	0.90	0.94	0.89	92%
Li	1.01	0.95	1.01	0.94	98%
Mg	0.94	0.96	0.95	0.93	94%
Mn	0.99	1.00	0.97	1.02	100%
Mo		0.93		0.91	92%
Na	0.98	0.97	0.98	0.97	97%
Nb	0.84	0.80	0.84	0.85	83%
Ni	1.10	1.06	0.99	1.21	109%
P	0.99	0.96	0.98	1.00	98%
Pb	0.91	0.99	0.91	1.08	97%
Rb	1.05	1.02	1.02	1.01	102%
Sc	1.16	1.13	1.13	1.20	115%
Sr	1.15	1.20	1.16	1.25	119%
Ti	0.85	0.90	1.00	0.96	93%
V	1.04	1.08	1.08	1.07	107%
Y	0.70	0.44	0.75	0.48	59%
Zn	0.92	0.93	0.90	0.96	93%
Zr	0.14	0.19	0.27	0.17	19%

reported for Br, Cr, Hf, Lu, Ta, Tb and U (Table 5). Seventeen elements out of 26 (As, Ba, Ce, Co, Cs, Eu, Fe, La, Na, Rb, Sb, Sc, Sm, Th, W, Yb and Zr) show average recoveries within $\pm 5\%$ of 100%, although W is only detectable in one standard (TILL-2).

PRECISION

The overall precision of the field and analytical duplicates is shown in Table 6. This single parameter does not take into account the variability of precision with concentration levels.

Table 4. Recoveries for standards OREAS-46 and OREAS-47 used in ICP-OES analyses. Those elements whose analyses were below the detection limit are shown as <DL. Analyses outside the two-standard deviation limit are denoted with a plus sign for overestimations, and a minus sign for underestimations

Element	OREAS 46				OREAS 47			
	m-2s	m	m+2s	Lab Number 7834460	m-2s	m	m+2s	Lab Number 7834480
Al2	5.94	6.26	6.58	6.01	5.88	6.25	6.62	5.94
As2	0.5	1.0	1.5	<DL	8.70	9.57	10.44	9
Ba2	449	473	496	483	457.45	485.28	513.10	495
Be2	0.76	0.91	1.07	1.0	0.81	1.04	1.26	1.1
Ca2	2.32	2.40	2.49	2.39	2.20	2.31	2.42	2.28
Cd2	0.03	0.06	0.09	<DL	0.43	0.50	0.57	
Ce2	33	36	40	38	50	55	59	56
Co2	9	10	11	12 (+)	49	53	57	64 (+)
Cr2	29	46	63	64 (+)	59	82	106	86
Cu2	22	23	24	24	152	159	166	158
Dy2	1.8	2.0	2.3	2.0	1.9	2.1	2.4	2.1
Fe2	2.48	2.61	2.75	2.66	2.64	2.78	2.91	2.78
K2	1.13	1.19	1.25	1.14	1.12	1.18	1.24	1.13
La2	17	19	21	20	28	30	32	32
Li2	9.5	10.4	11.3	10.3	37.8	42.5	47.3	39
Mg2	0.89	0.94	1.00	0.94	0.93	0.98	1.02	0.96
Mn2	452	489	525	490	476	509	542	502
Mo2	0.6	0.8	0.9	<DL	12.0	12.9	13.8	12
Na2	2.44	2.61	2.78	2.68	2.50	2.61	2.72	2.49 (-)
Nb2	3.7	4.6	5.4	4	15.3	17.0	18.7	15 (-)
Ni2	25	27	29	25	84	90	94	74 (-)
P2	502	543	583	535	530	564	597	535
Pb2	6.6	7.0	7.4	4 (-)	263	284	304	317 (+)
Rb2	30.4	33.5	36.7	34	34	38	42	39
S2		<0.005		41	333	437	540	369
Sc2	8.0	8.8	9.5	10.2 (+)	8.5	9.1	9.8	10.4 (+)
Sr2	382	408	433	455 (+)	374	408	442	449 (+)
Ti2	1880	2075	2270	2255	1962	2128	2294	2262
V2	52	56	61	61	54	58	62	61
Y2	9.8	10.5	11.2	11	9.9	10.7	11.5	11
Zn2	33	36	38	36	198	226	254	207
Zr2	52	61	71	30 (-)	50	633	76	33 (-)

Table 5. Accuracy of INAA analyses; calculated as the arithmetic mean of multiple analyses of each certified reference standard, divided by the recommended value for the standard

	TILL-1	TILL-2	TILL-3	Arithmetic Mean
As	0.98	0.97	1.02	99%
Au	1.76	0.75	0.90	114%
Ba	1.00	0.95	1.00	98%
Br	0.57	0.95	0.92	81%
Ce	0.98	1.07	1.00	102%
Co	0.92	1.12	0.92	99%
Cr	0.78	0.63	1.01	81%
Cs	0.94	0.98	1.12	101%
Eu	1.05	1.04		105%
Fe	1.00	1.00	1.03	101%
Hf	1.03	0.94	0.75	91%
La	0.94	1.04	0.90	96%
Lu	0.78	0.78	1.09	88%
Na	0.97	1.03	1.02	101%
Rb	0.98	0.99	0.99	99%
Sb	0.99	0.99	0.89	96%
Sc	1.03	1.04	1.00	102%
Sm	1.04	1.03	1.07	105%
Ta	1.00	1.02	0.67	90%
Tb	0.92	0.94	0.20	69%
Th	0.99	0.96	1.04	100%
U	0.93	0.94	0.93	93%
W		0.98		98%
Yb	0.94	0.97	1.02	98%
Zr	0.96	0.90	0.98	95%

Therefore, results of analytical and field duplicates for all elements are displayed graphically in Appendix C (Thompson-Howarth plots, *see* Thompson and Howarth, 1978). Figures 2 and 3 show examples where the elements' repeatability in field duplicates varies significantly from the repeatability in analytical duplicates (Mn by ICP-OES), and where it does not (Ce by INAA). Figure 4 summarizes the precision of all the analytical parameters, in field and analytical parameters in bar-chart form.

Table 6. Overall analytical and field precision

Element	Precision (95% C.L.)		Element	Precision (95% C.L.)	
	Analytical	Field		Analytical	Field
Al2	2.0	17.8	Mn2	3.1	29.2
As1	55.2	52.5	Mo1		66.7
As2	21.2	46.0	Mo2	54.3	74.5
Au1	155.6	168.3	Na1	6.1	29.8
Ba1	14.7	22.0	Na2	5.1	31.6
Ba2	3.6	21.8	Nb2	9.6	22.1
Be2	2.7	18.9	Ni2	4.7	33.6
Br1	10.3	89.3	P2	4.5	45.1
Ca2	3.4	29.7	Pb2	7.1	35.8
Cd2	22.5	71.8	Rb1	23.9	36.8
Ce1	15.2	39.8	Rb2	9.0	20.4
Ce2	6.4	43.4	S2	4.2	62.8
Co1	33.0	67.0	Sb1	27.3	40.0
Co2	5.0	43.2	Sc1	10.3	15.8
Cr1	17.6	65.1	Sc2	2.4	15.5
Cr2	5.4	25.0	Sm1	7.9	28.6
Cs1	117.5	89.2	Sr2	3.5	27.4
Cu2	11.1	74.1	Ta1	44.9	39.4
Dy2	4.6	16.7	Tb1	12.8	22.8
Eu1	136.5	99.2	Th1	8.1	40.2
Fe1	6.5	25.7	Ti2	4.0	15.6
Fe2	3.9	24.3	U1	13.9	26.6
Hf1	17.6	57.5	V2	2.9	19.0
K2	3.0	28.3	W1	109.3	66.7
La1	15.1	39.1	Y2	5.4	20.2
La2	8.1	34.5	Yb1	19.0	33.3
Li2	4.9	27.0	Zn2	4.1	24.5
LOI	3.8	70.2	Zr1	146.7	148.2
Lu1	20.0	31.7	Zr2	8.7	53.4
Mg2	2.6	36.1			

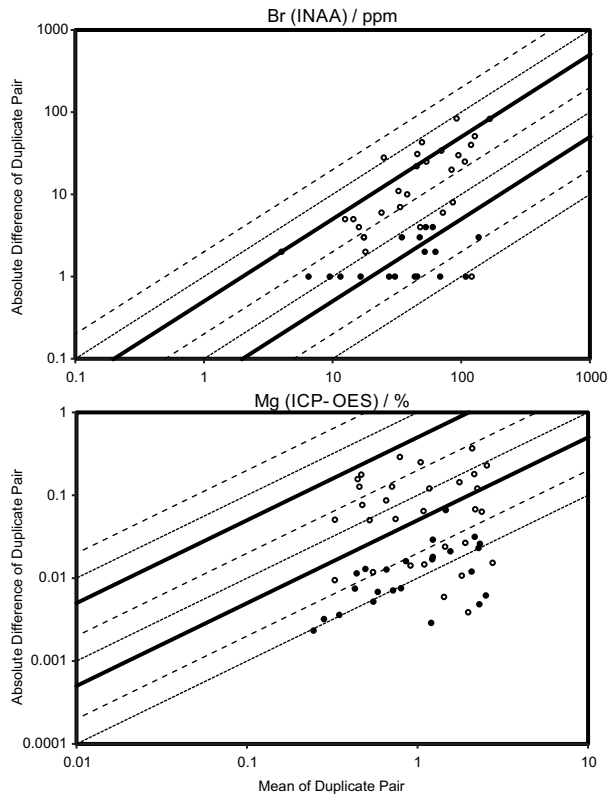


Figure 2. Thompson-Howarth precision plots for field and analytical duplicates of Br (INAA analyses) and Mg (ICP-OES analyses): examples of elements whose field variability significantly exceeds its analytical variability. In these precision plots, the mean of each pair of duplicates is plotted against their absolute difference; both axes are scaled logarithmically. A series of parallel lines indicates precision of gradually increasing absolute value, from $\pm 1\%$ to $\pm 200\%$. Field duplicates are denoted by open circles, and analytical duplicates by closed circles; the absolute value of the precision for the former is invariably greater (i.e., the repeatability is worse).

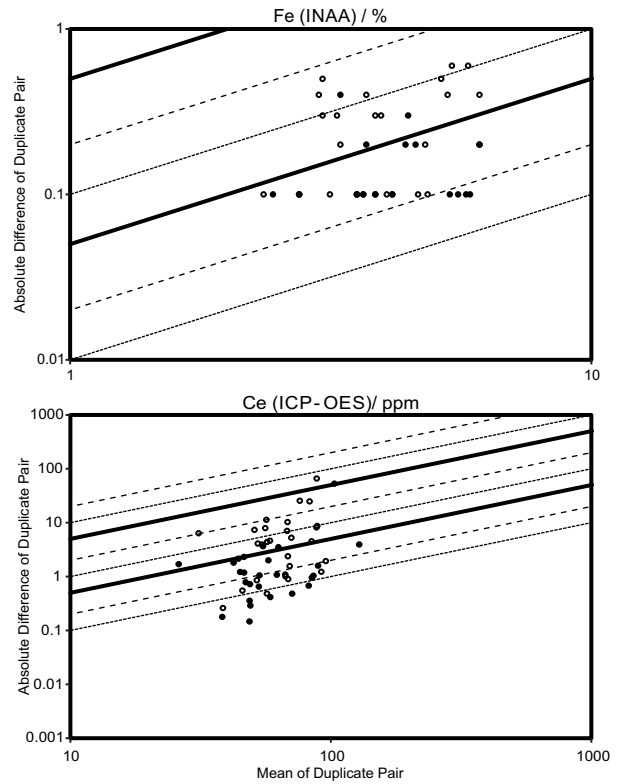


Figure 3. Thompson-Howarth precision plots for field and analytical duplicates of Fe (INAA analyses) and Ce (INAA analyses): examples of elements whose field variability does not significantly exceed its analytical variability.

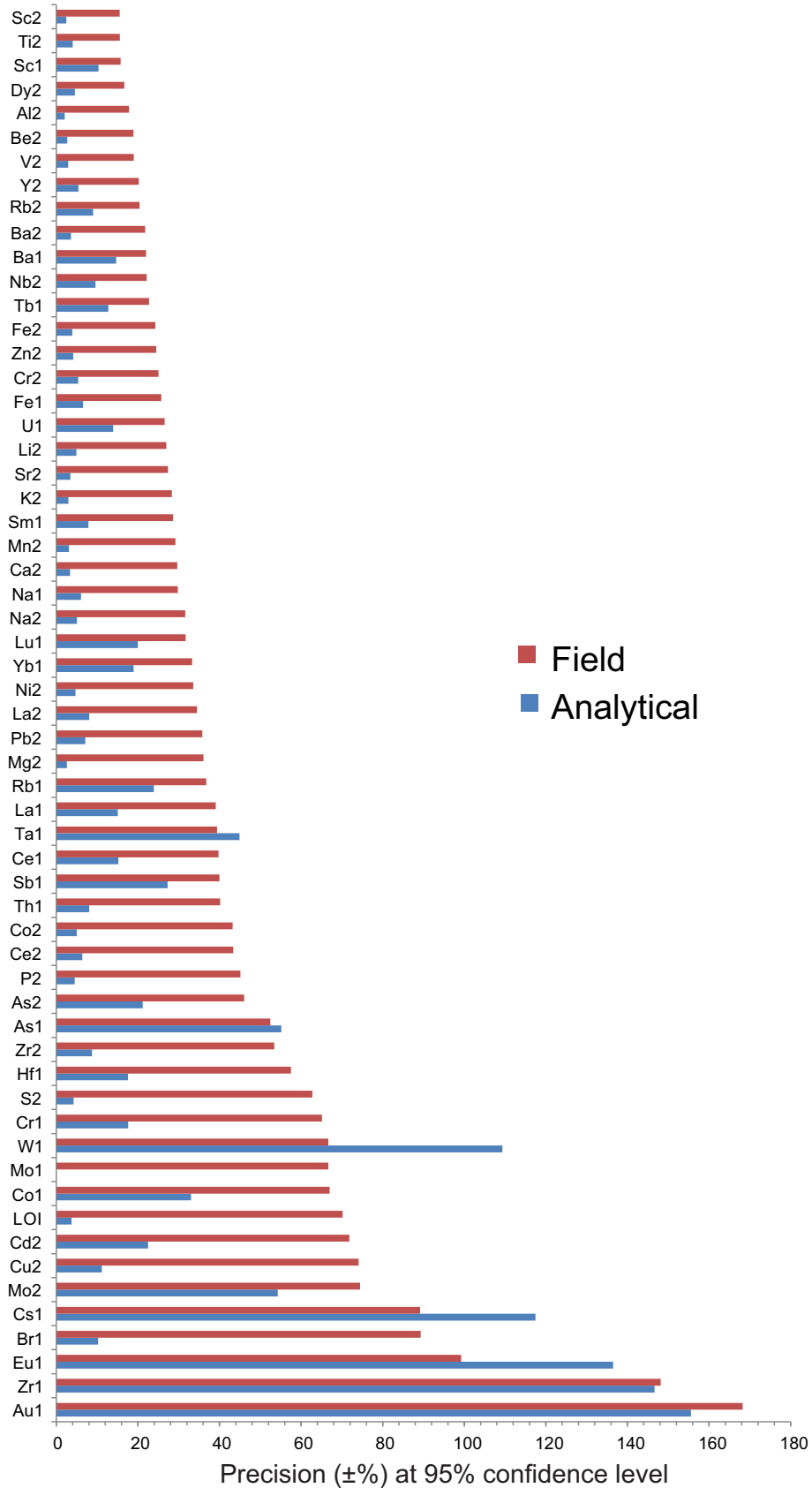


Figure 4. Bar chart summarizing precision of field and analytical duplicates.

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APPENDICES A–C

Appendix A is available as a digital comma-separated file (.csv) and Appendices B and C are available as pdf files through [this link](#).

Appendix A: Till Geochemistry 2016–2018

Suffixes

1. INAA
2. ICP-OES after multi-acid (HF/HCl/HNO₃/HClO₄) digestion
6. AAS after nitric acid digestion
9. ISE after alkaline fusion

Detection limits in the database are replaced by a value that is ½ of the detection limit.

Appendix B: Control Charts

In each chart, a dashed black line represents the expected value (the mean of multiple analyses, carried out at several labs and reported by Lynch (1996) and two continuous black lines represent the upper and lower ‘limits of acceptability’, established by adding and subtracting two standard deviations (also reported by Lynch, *op. cit.*). Charts for certain elements are omitted because the latter were undetectable in the establishment of recommended values.

Suffixes

1. INAA
2. ICP-OES after multi-acid (HF/HCl/HNO₃/HClO₄) digestion

Units

- Al₂, Ca₂, Fe₁, Fe₂, K₂, Mg₂, Na₁, Na₂ in weight percent.
- As₁, As₂, Ba₁, Ba₂, Be₂, Br₁, Ce₁, Ce₂, Co₁, Co₂, Cr₁, Cr₂, Cs₁, Cu₂, Eu₁, Hf₁, La₁, La₂, Lu₁, Mn₂, Li₂, Mo₁, Mo₂, Nb₂, Ni₂, P₂, Pb₂, Rb₁, Rb₂, S₂, Sc₁, Sc₂, Sm₁, Sr₂, Ta₁, Tb₁, Th₁, Ti₂, U₁, V₂, Zn₂ and Zr₂ in parts per million (ppm).
- Au₁ in parts per billion (ppb).

Appendix C: In these precision plots, the mean of each pair of duplicates is plotted against their absolute difference; both axes are scaled logarithmically. A series of parallel lines indicates precision of gradually increasing absolute value, from ± 1% to ± 200%. Field duplicates are denoted by open circles, and analytical duplicates by closed circles; the absolute value of the precision for the former is invariably greater (*i.e.*, the repeatability is worse).