



CCRMP

Canadian Certified Reference Materials Project

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PCMRC

Projet canadien de matériaux de référence certifiés

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Certificate of Analysis

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Provisional Values

TILL-1, TILL-2, TILL-3 and TILL-4

Geochemical Soil and Till Reference Materials

Source

TILL-1, TILL-2, TILL-3 and TILL-4 were collected and characterized in cooperation with the Mineral Resources Division, Minerals and Continental Geoscience Branch, Geological Survey of Canada.

TILL-1 was collected 25 kilometres north-west of Lanark, Ontario; TILL-3 was collected 8 kilometres east of Cobalt, Ontario. These two soil samples were collected from the combined B and C horizons. The two till samples (TILL-2 and TILL-4) were collected near Scission's Brook, New Brunswick. At this location, extensive trenching had been done by the mining company who owned the property in order to expose the till. The company had obtained preliminary analyses of till samples collected at various sites within the trenched areas. These analyses were used as guidelines to sampling. No effort was made to collect any particular horizon. In order to augment the molybdenum levels in TILL-4, a small quantity of a molybdenite-bearing soil was collected near an

old test pit. All sampling was done by shovelling.

Description

These four materials, two soils and two tills, complete a series of reference samples of surficial materials which also includes the lake and stream sediment materials, LKSD-1, LKSD-2, LKSD-3, LKSD-4, STSD-1, STSD-2, STSD-3 and STSD-4, which are already available.

Like the sediment series, the TILL samples are characterised for major element oxides, total elements as well as elements from partial extractions. The partial extractions are concentrated hydrochloride - concentrated nitric acids and dilute hydrochloric-dilute nitric acids. In addition, informational data from a single source are provided for a number of elements derived by EPA digestions 3050 and 3051.



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Intended Use

TILL-1, TILL-2, TILL-3 and TILL-4 are intended for quality control in chemical analysis.

Instructions for Use

TILL samples should be used "as is" without drying. The contents of the bottle should be thoroughly mixed before taking samples.

Method of Preparation

In each case, the collected material was spread to a depth of 7.5 to 10 cm over a polyethylene sheet and allowed to dry for several weeks at room temperature. When dry, each sample was sieved through an 80-mesh (177 µm) screen. The +80 mesh fraction was discarded. The -80-mesh fraction was ball milled and sieved through a 200-mesh screen (74 µm). The oversize material from this sieving was retained, ball milled and sieved a second time through the 200-mesh screen. At this point, any oversize fraction (plus-200 mesh) was discarded. The two minus-200-mesh fractions were combined and tumbled as a single batch in a conical blender for eight hours. Each material was bottled in 100-g units.

State of Homogeneity

A method described by Lynch (1) was employed for homogeneity testing. No evidence of inhomogeneity was found.

Method of Certification

TILL-1, TILL-2, TILL-3 and TILL-4 were characterised by an interlaboratory analysis program involving thirty-one laboratories. The provisional values for these soil and till reference materials were assigned from the average of data after a two-step trimming method described by Lynch (1).

Legal Notice

The Canadian Certified Reference Materials Project has prepared these reference materials and statistically evaluated the analytical data of the interlaboratory certification program to the best of its ability. The purchaser, by receipt hereof, releases and indemnifies the Canadian

Certified Reference Materials Project from and against all liability and costs arising out of the use of these materials and information.

References

(1) J.J. Lynch (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.

The preparation and certification procedures used for TILL-1, TILL-2, TILL-3 and TILL-4, including values obtained by individual laboratories, are to be published in *Geostandards Newsletter*. This report will be available free of charge on application to:

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Material collection locations

Sample	NTS* Designation	Location
TILL-1	31F	Joe Lake, Ontario
TILL-2	21C	5 km West Scisson's Brook, New Brunswick
TILL-3	31M	O'Brien Mine, near Cobalt Ontario
TILL-4	21C 31G	Scisson's Brook, New Brunswick Molybdenite Occurrence near Hull, QuJbec

*National topographic system

Summary of major and minor elements expressed as oxides (%)

	TILL-1	TILL-2	TILL-3	TILL-4
SiO ₂	60.9	60.8	69.1	65.0
Al ₂ O ₃	13.7	16.0	12.2	14.4
Fe ₂ O ₂ (T)	6.82	5.39	3.92	5.63
MgO	2.15	1.83	1.71	1.26
CaO	2.72	1.27	2.63	1.25
Na ₂ O	2.71	2.19	2.64	2.46
K ₂ O	2.22	3.07	2.42	3.25
MnO	0.18	0.10	0.06	0.06
TiO ₂	0.98	0.88	0.49	0.81
P ₂ O ₅	0.22	0.17	0.11	0.20
LOI (1000°C)	7.3	8.1	4.6	5.7
Sum	99.90	99.80	99.88	100.02

Summary of "total" elements in TILL series (in µg/g unless otherwise noted)

	TILL-1	TILL-2	TILL-3	TILL-4
As	18	26	87	111
Au (ppb)	13	2	6	5
Ba	702	540	489	395
Be	2.4	4.0	2.0	3.7
Bi	<5	<5	<5	40
Br	6.4	12.2	4.5	8.6
Ce	71	98	42	78
Co	18	15	15	8
Cr	65	74	123	53
Cs	1.0	12.	1.7	12
Cu	47	150	22	237
Eu	1.3	1.0	<1.0	<1.0
Er	3.6	3.7	1.4	3.2
Fe (%)	4.81	3.84	2.78	3.97
Hf	13	11	8	10
La	28	44	21	41
Li	15	47	21	30
LOI (500°C) %	6.3	6.8	3.6	4.4
Lu	0.6	0.6	0.2	0.5
Mn	1420	780	520	490
Mo	2	14	2	16
Nb	10	20	7	15
Nd	26	36	16	30
Ni	24	32	39	17
P	930	750	490	880
Pb	22	31	26	50
Rb	44	143	55	161
S (%)	<0.05	<0.05	<0.05	0.08
Sb	7.8	0.8	0.9	1.0
Sc	13	12	10	10
Sm	5.9	7.4	3.3	6.1
Sr	291	144	300	109
Ta	0.7	1.9	<0.5	1.6
Tb	1.1	1.2	<0.5	1.1
Th	5.6	18.4	4.6	17.4
Ti	5990	5300	2910	4840
U	2.2	5.7	2.1	5.0
V	99	77	62	67
W	<1	5	<1	204
Y	38	40	17	33
Yb	3.9	3.7	1.5	3.4
Zn	98	130	56	70
Zr	502	390	230	385

Summary of partial extraction elements; concentrated HNO₃ - concentrated HCl (in µg/g unless otherwise noted)

	TILL-1	TILL-2	TILL-3	TILL-4
Ag	0.2	0.2	1.6	<0.2
As	13	22	84	102
Ba	84	95	43	71
Bi	<3	4	<3	44
Cd	<0.2	0.3	<0.2	<0.2
Co	12	13	11	6
Cr	30	40	73	26
Cu	48	149	23	254
Fe (%)	3.1	3.2	2.0	3.3
Hg (ppb)	92	74	107	39
Mn	950	530	310	260
Mo	<2	11	<2	14
Ni	18	31	32	15
Pb	12	21	16	36
V	48	38	33	38
Zn	70	116	43	63

Summary partial extraction elements; dilute HNO₃ - dilute HCl (in µg/g unless otherwise noted)

	TILL-1	TILL-2	TILL-3	TILL-4
Ag	<0.2	<0.2	1.4	<0.2
Co	12	12	10	6
Cu	49	152	23	252
Fe (%)	3.4	3.4	2.2	3.5
Mn	1020	570	310	260
Mo	1	13	1	15
Ni	17	30	32	14
Pb	14	24	17	37
Zn	71	116	43	62

Single source data by EPA 3050 digestion - ICP-AES analysis (all values in µg/g)

Element	TILL -1	TILL - 2	TILL - 3	TILL - 4
Al	18883	32600	10750	25200
Ba	84.3	104.6	46.4	75.1
Be	1.1	2.1	0.8	1.6
Cd	<0.33	<0.33	<0.33	<0.33
Ca	4145	1940	6240	1438
Cr	29.3	39.3	66.7	25.8
Co	11.7	12.5	10.2	5.8
Cu	44.0	162	17.6	266
Fe	29167	33967	19900	32533
Pb	24.0	35.7	24.0	50.0
Mg	6250	7547	6510	5470
Mn	1060	601	294	243
Mo	8.7	18.7	5.0	20.7
Ni	14.0	27.5	28.0	11.0
P	915	856	470	1150
K	1188	4355	1220	3915
Na	530	527	336	313
V	89	107.2	71.6	82.9
Zn	65.2	111.0	42.5	59.0

Single source data by EPA 3051 digestion - ICP-AES analysis (all values in µg/g)

Element	TILL - 1	TILL - 2	TILL - 3	TILL - 4
Al	18050	27550	12000	20400
Ba	77.8	60.8	49.2	102
Be	<0.2	1.7	<0.2	<0.2
Cd	<0.35	<0.3	<0.35	<0.3
Ca	3817	1640	5660	1565
Cr	29.3	34.7	64.7	24.3
Co	12.3	15.5	14.8	8.1
Cu	44.8	176	16.5	332
Fe	37900	38600	21000	40500
Pb	<10.0	31.5	23	42
Mg	6990	8525	7445	5570
Mn	1060	588	317	280
Mo	<2.5	<1.7	6.0	22.5
Ni	18.7	29.5	26.5	11.0
P	834	540	457	1260
K	640	3370	964	3115
Na	575	450	427	485
V	70	111.2	66.1	88.0
Zn	69.8	112.7	42.7	57.6

CCRMP Reference Materials LKSD, STSD, and TILL

Explanation of Extraction Methods

The provisional values for the lake, stream and till reference materials (LKSD, STSD and TILL) were determined from the results of numerous laboratories involved in geochemical exploration using their existing routine methods. Two main types of methods were used.

The first type can be termed total methods wherein the complete amount of a particular element is determined. One subgroup of these total methods involves the sample being analyzed without pretreatment i.e., instrumental neutron activation and powder x-ray diffraction. Another subgroup of total methods includes pretreatments such as a multi acid dissolution with hydrofluoric acid, or fusions followed by, for example, an instrumental finish. This second group of methods may be used to determine the total concentration of numerous elements, but losses of some other elements may occur.

A second type of techniques involves a partial dissolution or extraction of the elements using different combinations of acids at varying strengths. The laboratories that submitted data did not follow established protocols with regards to either the types or amounts of acid utilized. However, two main subgroups of partial extraction or partial dissolution techniques were chosen. The first of these involves the use of both diluted nitric and hydrochloric acids i.e., 2 mL of nitric acid, 1 mL of hydrochloric acid and 5 mL of water; or 3 mL, 2 mL and 1 mL of each component, respectively; or 3 mL, 1 mL and 4 mL of each component, respectively. The second subgroup involves the use of varying amounts of these two acids without the addition of any water i.e., 3 mL of hydrochloric acid plus 1 mL of nitric acid, or 3 mL of nitric acid plus 1 mL of hydrochloric acid. For both of these two subgroups, the sample and acid mixture were heated from 2 to 4 hours at 95°C to 100°C, cooled, diluted to volume and analyzed.