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# TILL GEOCHEMISTRY OF SHEFFIELD LAKE, SPRINGDALE, DAWES POND AND THE TOPSAILS (NTS MAP AREAS 12H/07, 08, 01, 02) 


J.S. Organ and S.D. Amor

Open File 012H/2212
St. John's
Newfoundland and Labrador
October, 2017

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Cover: Gold in till (-180 micron fraction) east of Sheffield Lake. Striation symbols indicate that ice movement was generally to the northeast. Superimposed on total-field aeromagnetic data from Assessment File NFLD/3083.

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#### Abstract

Analytical results for 1028 routine $<180 \mu$ till samples from north-central Newfoundland are presented. These samples have been analyzed by ICP-OES and INAA and checked for acceptable accuracy and precision. Older data from the same area, some of which have been levelled to compensate for different size fractions and digestion methods, are re-released with this report.


## INTRODUCTION

This report complements the release of analytical data for $1028<180 \mu$ ( -80 mesh) till samples collected from the Sheffield Lake, Springdale, Dawes Pond and the Topsails (NTS map areas $12 H / 07,08,01$ and 02 , respectively). Sampling statistics are shown in Table 1. The field program also included the determination of the paleo ice flow to aid in the subsequent interpretation of geochemical anomalies, and the understanding of the regional ice-flow history. An interpretation of the surficial and geochemical data will be released at a later date.

Table 1. Samples by NTS map area

| NTS Map <br> Area | Map Sheet <br> Name | Number of <br> Routine Till <br> Sample Sites |
| :--- | :--- | :--- |
| $12 \mathrm{H} / 01$ | Dawes Pond | 189 |
| 12H/02 | The Topails | 40 |
| $12 \mathrm{H} / 07$ | Sheffield Lake | 448 |
| $12 \mathrm{H} / 08$ | Springdale | 350 |
| $12 \mathrm{H} / 10$ | Hampden | 1 |

The samples were collected during the 2012 and 2013 field seasons, as part of an ongoing till-geochemistry and surficial-mapping program carried out across the island. The study area extends from Millertown Junction in the south, to Springdale in the north and from the community of South Brook in the east to Route 420 in the west. The Trans Canada highway (TCH), along with routes 380 (Beothuk Trail-Robert's Arm), 390 (Springdale), 410 (Dorset TrailBaie Verte), 420 (White Bay South HighwaySop's Arm) and 421 (Hampton) provide good access across the study area (Figure 1). An extensive network of forestry-resource roads south of the TCH provides ATV access. The work was conducted using truck and ATV traverses; in remote areas, access was provided by helicopter.

Parts of NTS map areas 12H/02 and 07 were sampled by the Geological Survey of Canada in the early 1990s (Klassen, 1994a; 276 samples) and subsequently by the Geological Survey of Newfoundland and Labrador (GSNL; Davenport et al., 1996, 193 samples; Liverman et al., 2000; 63 samples). In-fill sampling was necessary to provide complete coverage because the previous sampling was conducted at a wider spacing than that currently applied by the GSNL for regional till sampling.

## BEDROCK GEOLOGY AND MINERAL RESOURCES

The study area lies within the Dunnage and Humber zones of the Newfoundland Appalachians (Figure 2A, B, C). Polydeformed schists, gneisses and granitoid intrusions of the Humber Zone record the evolution and destruction of the continental margin of eastern North America (Hibbard, 1983). Ophiolite suites, along with volcanic and intrusive complexes within the Dunnage Zone, are the remnants of the early Paleozoic Iapetus Ocean (Hibbard, 1983). The Baie Verte Line, a complex fault zone, separates the Dunnage and Humber zones. It strikes northeastward from Birchy Lake towards Baie Verte and separates the schists of the Fleur De Lys Supergroup in the west from the ophiolites of the Advocate Complex in the east. Hibbard (1983) and O'Brien (2003, 2009) give detailed descriptions of the bedrock geology within the study area.

Figure 1. Location of study area. Red lines represent provincial roads; green lines represent forestry-resource roads.

Figure 2A. Bedrock geology, mineral occurrences and till sample locations in NTS map areas 12H/01, 02. Red and black dots show locations of samples collected in 2012 and 2013 respectively. Coloured squares represent samples collected prior to this study by Klassen (1994; green), by Liverman (Davenport et al., 1996; blue), and Taylor (Liverman et al., 2000; yellow).

Figure 2B. Bedrock geology, mineral occurrences and till sample locations in NTS map area 12H/07. Red and black dots show locations of samples collected in 2012 and 2013 respectively. Coloured squares represent samples collected prior to this study by Klassen (1994; green), by Liverman (Davenport et al., 1996; blue), and Taylor (Liverman et al., 2000; yellow).
 Figure 2C. Bedrock geology, mineral occurrences and till sample locations in NTS map area 12H/08. Red and black dots show locations of samples collected in 2012 and 2013 respectively. Coloured squares represent samples collected prior to this study by Klassen (1994; green), by Liverman (Davenport et al., 1996; blue), and Taylor (Liverman et al., 2000; yellow).

Legend for Figure 2.

## MINERAL OCCURRENCES

Eighty-one mineral occurrences including those of asbestos and pyrite, as well as minerals of gold, chromium, copper, nickel, lead and zinc, are located within the study area; these include the worked-out Gullbridge Cu mine (Table 2; Geological Survey of Newfoundland and Labrador, 2016). More than $60 \%$ of the occurrences are associated with marine mafic volcanic rocks of the Roberts Arm Group (Geological Survey of Newfoundland and Labrador, 2013).

In the Dawes Pond area (NTS map area 12H/01), 47 metallic-mineral occurrences comprising 18 showings, 22 indications, 5 prospects, one past producer (Gullbridge) and one developed prospect (Lake Bond) are associated with three rock groups. Of these, the Roberts Arm Group hosts occurrences of Au (2), Cu (17), Ni (1), Pb (1), Zn (2) and pyrite (16), while single occurrences of Ni and pyrite are hosted within the argillaceous Shoal Arm Formation and the Dawes Pond Granite, respectively.

In the Sheffield Lake area (NTS map area $12 \mathrm{H} / 07$ ) 14 occurrences, comprising 9 showings and 5 indications, are associated with 4 rock units (Table 2). Two Zn occurrences are associated with the non-marine felsic volcanic rocks of the Sheffield Lake Complex. One Cu occurrence is

Table 2. Mineral occurrences in the study area

| Map Sheet No. | MODS <br> No. | Latitude ( ${ }^{\circ} \mathbf{N}$ ) <br> (NAD 1927) | $\begin{aligned} & \text { Longitude }\left({ }^{\circ}\right. \text { W) } \\ & \text { (NAD 1927) } \end{aligned}$ | Occurrence Name | Status | Commodity |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 12H/01 | Cu 001 | 49.1412 | 56.2221 | Starkes Pond North Copper | Showing | Copper |
| $12 \mathrm{H} / 01$ | Cu 002 | 49.0599 | 56.2061 | Bayly Showing | Showing | Copper |
| $12 \mathrm{H} / 01$ | Cu 003 | 49.1578 | 56.2085 | North Road Showing | Indication | Copper |
| $12 \mathrm{H} / 01$ | Cu 004 | 49.1793 | 56.1692 | Southwest Shaft | Prospect | Copper |
| $12 \mathrm{H} / 01$ | Cu 005 | 49.1673 | 56.1650 | Baker Brook No. 1 | Indication | Copper |
| $12 \mathrm{H} / 01$ | Cu 006 | 49.1633 | 56.1688 | Baker Brook No. 2 | Indication | Copper |
| 12H/01 | Cu 007 | 49.1984 | 56.1550 | Gullbridge Mine | Past Producer (Exhausted) | Copper |
| 12H/01 | Cu 008 | 49.2019 | 56.1537 | Mineral Point North No. 1 | Indication | Copper |
| $12 \mathrm{H} / 01$ | Cu 009 | 49.2060 | 56.1520 | Mineral Point North No. 2 | Indication | Copper |
| $12 \mathrm{H} / 01$ | Cu 010 | 49.1184 | 56.2507 | Dawes Pond West Copper | Showing | Copper |
| $12 \mathrm{H} / 01$ | Cu 011 | 49.1269 | 56.2286 | Starkes Pond South Copper | Showing | Copper |
| $12 \mathrm{H} / 01$ | Cu 012 | 49.1393 | 56.2405 | Starkes Pond West Copper \#1 | Prospect | Copper |
| $12 \mathrm{H} / 01$ | Cu 013 | 49.1468 | 56.2341 | Starkes Pond West Copper \#2 | Showing | Copper |
| $12 \mathrm{H} / 01$ | Cu 014 | 49.1660 | 56.1921 | Sam Occurrence | Showing | Copper |
| $12 \mathrm{H} / 01$ | Cu 015 | 49.1588 | 56.2121 | Diamond Pond South Copper | Showing | Copper |
| $12 \mathrm{H} / 01$ | Cu 016 | 49.1341 | 56.2231 | Starkes Pond South Road | Showing | Copper |
| $12 \mathrm{H} / 01$ | Cu 017 | 49.0034 | 56.1862 | Skull Hill Brook | Showing | Copper |
| $12 \mathrm{H} / 01$ | Au 001 | 49.0635 | 56.0842 | Rocky Brook East | Showing | Gold |
| $12 \mathrm{H} / 01$ | Au 002 | 49.0307 | 56.2101 | Lake Bond West Gold | Showing | Gold |
| $12 \mathrm{H} / 01$ | Ni 001 | 49.1000 | 56.1064 | Powderhorn Lake - Road Showing | Showing | Nickel |
| $12 \mathrm{H} / 01$ | Ni 002 | 49.0974 | 56.1007 | Powderhorn Lake - Main Showing | Prospect | Nickel |
| 12H/01 | Pb 001 | 49.1383 | 56.2236 | Starkes Pond West Lead | Indication | Lead |
| 12H/01 | Zn 001 | 49.0274 | 56.1865 | Lake Bond | Developed <br> Prospect | Zinc |
| $12 \mathrm{H} / 01$ | Zn 002 | 49.2162 | 56.1344 | Great Gull Pond North | Indication | Zinc |
| $12 \mathrm{H} / 01$ | Zn 003 | 49.1100 | 56.1083 | Powderhorn Lake | Prospect | Zinc |
| $12 \mathrm{H} / 01$ | Zn 004 | 49.1235 | 56.2448 | Dawes Pond West Zinc | Showing | Zinc |
| $12 \mathrm{H} / 01$ | Zn 005 | 49.1462 | 56.2365 | Starkes Pond West Zinc | Showing | Zinc |

Table 2. Continued

| Map Sheet No. | MODS No. | $\begin{aligned} & \text { Latitude }\left({ }^{\circ} \mathrm{N}\right) \\ & \text { (NAD 1927) } \end{aligned}$ | $\begin{aligned} & \text { Longitude }\left({ }^{\circ} \mathrm{W}\right. \text { ) } \\ & \text { (NAD 1927) } \end{aligned}$ | Occurrence Name | Status | Commodity |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 12H/01 | Zn 006 | 49.2480 | 56.0973 | Handcamp - MAC Zone | Showing | Zinc |
| 12H/01 | Zn 007 | 49.1134 | 56.1047 | Powderhorn Lake-Dead Tree Zone | Showing | Zinc |
| $12 \mathrm{H} / 01$ | Zn 008 | 49.1083 | 56.1016 | Powderhorn Lake-South Zone | Showing | Zinc |
| $12 \mathrm{H} / 01$ | Pyr001 | 49.1711 | 56.1756 | Great Gull Pond Southwest | Indication | Pyrite |
| $12 \mathrm{H} / 01$ | Pyr002 | 49.0148 | 56.1808 | Lake Bond Southwest | Indication | Pyrite |
| $12 \mathrm{H} / 01$ | Pyr003 | 49.0211 | 56.1806 | Lake Bond West No. 1 | Indication | Pyrite |
| 12H/01 | Pyr004 | 49.0191 | 56.1855 | Lake Bond West No. 2 | Indication | Pyrite |
| 12H/01 | Pyr005 | 49.0304 | 56.1977 | Lake Bond Northwest No. 1 | Indication | Pyrite |
| $12 \mathrm{H} / 01$ | Pyr006 | 49.0332 | 56.1782 | Lake Bond North No. 2 | Indication | Pyrite |
| 12H/01 | Pyr007 | 49.0280 | 56.1782 | Lake Bond North No. 1 | Indication | Pyrite |
| $12 \mathrm{H} / 01$ | Pyr008 | 49.0351 | 56.1921 | Lake Bond Northwest No. 2 | Indication | Pyrite |
| 12H/01 | Pyr009 | 49.0413 | 56.1939 | Two Bit Pond East | Indication | Pyrite |
| $12 \mathrm{H} / 01$ | Pyr010 | 49.0458 | 56.2099 | Two Bit Pond Northwest | Indication | Pyrite |
| $12 \mathrm{H} / 01$ | Pyr011 | 49.1435 | 56.1699 | Baker Lake | Indication | Pyrite |
| 12H/01 | Pyr012 | 49.1399 | 56.1855 | Baker Lake West | Indication | Pyrite |
| $12 \mathrm{H} / 01$ | Pyr013 | 49.1359 | 56.2106 | Starkes Pond East | Indication | Pyrite |
| 12H/01 | Pyr014 | 49.1403 | 56.2396 | Starkes Pond West | Prospect | Pyrite |
| 12H/01 | Pyr015 | 49.0763 | 56.2339 | Dawes Pond Southwest | Indication | Pyrite |
| $12 \mathrm{H} / 01$ | Pyr016 | 49.1374 | 56.2317 | Starkes Pond Road | Indication | Pyrite |
| 12H/01 | Pyr017 | 49.0346 | 56.2232 | Skull Pond West | Showing | Pyrite |
| $12 \mathrm{H} / 07$ | Cu 001 | 49.3794 | 56.9715 | Sandy Lake | Indication | Copper |
| 12H/07 | Cr 001 | 49.3943 | 56.6223 | Gillards Lake Chromium | Indication | Chromium |
| 12H/07 | Ni 001 | 49.3818 | 56.6470 | Gillards Lake South Nickel | Showing | Nickel |
| $12 \mathrm{H} / 07$ | Ni 002 | 49.4068 | 56.6186 | Gillards Lake Nickel | Indication | Nickel |
| 12H/07 | Ni 003 | 49.4536 | 56.5571 | Balloon Pond Nickel \#1 | Showing | Nickel |
| 12H/07 | Ni 004 | 49.4555 | 56.5567 | Balloon Pond Nickel \#2 | Showing | Nickel |
| 12H/07 | Zn 001 | 49.3352 | 56.6308 | Sheffield Brook Bridge Zinc | Showing | Zinc |
| 12H/07 | Zn 002 | 49.3295 | 56.6658 | Nichols Brook Zinc | Indication | Zinc |
| $12 \mathrm{H} / 07$ | Asb001 | 49.4402 | 56.5826 | South North Brook | Showing | Asbestos |
| 12H/07 | Asb002 | 49.3819 | 56.6765 | Indian Brook | Showing | Asbestos |
| 12H/07 | Asb003 | 49.3913 | 56.6249 | Gillards Lake | Showing | Asbestos |
| 12H/07 | Asb 004 | 49.4632 | 56.5567 | North Pond | Showing | Asbestos |
| 12H/07 | Asb 005 | 49.4556 | 56.5656 | North Brook | Showing | Asbestos |
| 12H/07 | Asb006 | 49.4039 | 56.6420 | West Gillards Lake | Indication | Asbestos |
| $12 \mathrm{H} / 08$ | Cu 001 | 49.4443 | 56.0697 | South Brook | Indication | Copper |
| $12 \mathrm{H} / 08$ | Cu 002 | 49.3382 | 56.0393 | Tommy's Arm River | Showing | Copper |
| 12H/08 | Cu 003 | 49.3247 | 56.0457 | Moose Brook | Showing | Copper |
| $12 \mathrm{H} / 08$ | Cu 004 | 49.3189 | 56.0315 | Rocky Pond | Indication | Copper |
| $12 \mathrm{H} / 08$ | Cu 005 | 49.4419 | 56.3947 | Whitehorn Brook | Showing | Copper |
| 12H/08 | Cu 006 | 49.4589 | 56.3582 | East Indian Pond | Indication | Copper |
| $12 \mathrm{H} / 08$ | Cu 007 | 49.4081 | 56.4723 | Jawbone Pond | Showing | Copper |
| $12 \mathrm{H} / 08$ | Cu 008 | 49.3873 | 56.0094 | Knife Pond | Indication | Copper |
| $12 \mathrm{H} / 08$ | Cu 009 | 49.3013 | 56.0735 | Bushy Bog | Showing | Copper |
| $12 \mathrm{H} / 08$ | Cu 010 | 49.2759 | 56.0982 | Angle Zone | Indication | Copper |
| 12H/08 | Cu 011 | 49.2624 | 56.0943 | Handcamp-Central Zone | Showing | Copper |
| $12 \mathrm{H} / 08$ | Cu 012 | 49.4996 | 56.2998 | Ursa Major | Prospect | Copper |
| 12H/08 | Cu 013 | 49.4913 | 56.3084 | Southern Cross | Prospect | Copper |
| $12 \mathrm{H} / 08$ | Au 001 | 49.2808 | 56.0736 | Handcamp | Prospect | Gold |
| $12 \mathrm{H} / 08$ | Zn 001 | 49.4260 | 56.0309 | Loon Pond West | Showing | Zinc |
| $12 \mathrm{H} / 08$ | Pyr001 | 49.4061 | 56.0146 | Loon Pond South | Showing | Pyrite |
| $12 \mathrm{H} / 08$ | Pyr002 | 49.4192 | 56.4701 | West Silver Brook | Indication | Pyrite |
| $12 \mathrm{H} / 08$ | Pyr003 | 49.3795 | 56.4835 | Cecil's Showing | Indication | Pyrite |
| 12H/08 | Pyr004 | 49.4118 | 56.0361 | Loon Pond | Indication | Pyrite |
| $12 \mathrm{H} / 08$ | Pyr005 | 49.4793 | 56.0275 | Halls Bay | Indication | Pyrite |

associated with the schists of the Fleur De Lys Supergroup. The ophiolitic rocks of the Advocate Complex host one Cr occurrence along with 4 of Ni and 6 of asbestos.

The Springdale (NTS map area 12H/08) contains 20 mineral occurrences, consisting of 8 indications, 6 showings and one prospect. Occurrences of $\mathrm{Au}(1), \mathrm{Cu}(9), \mathrm{Zn}(1)$ and pyrite (1) are hosted by the volcanosedimentary Roberts Arm Group or at its contact with granite. Copper (4) and pyrite (2) occurrences are also associated with the marine volcanic Catchers Pond Group (4) or at its contact with granite; one pyrite occurrence is also reported from within the Halls Bay granite.

The only two mineral occurrences in NTS map area $12 \mathrm{H} / 02$ are the exhausted Quarry Station and Summit Quarry granite deposits in the southeast of the map area.

## QUATERNARY HISTORY

During the late Wisconsinan glacial maximum, the Newfoundland Ice Cap extended out to the continental shelf (Grant, 1989; Shaw et al., 2006). Approximately 13000 years before present (BP), deglaciation became terrestrially based and the deglacial configuration was irregular and time-transgressive, due to both ice thickness and topography (Shaw et al., 2006). As can be seen from Figure 3A, the Springdale and South Brook areas were deglaciated by 12000 BP, with ice still covering much of the area of the Topsails and Sheffield Lake (Shaw et al., 2006). As ice continued to retreat, it disintegrated into a number of small isolated ice caps (Grant, 1974). Three of these were located within the study area: in the north part of NTS map area $12 \mathrm{H} / 07$, extending onto the Baie Verte Peninsula; in the south of NTS $12 \mathrm{H} / 02$ on the Topsails; and south and southeast of Dawes Pond in NTS 12H/01 (Figure 3B).

Coastal areas were ice free between $12470 \pm 300$ BP and $11000 \pm 190$ BP (Tucker, 1974, sample GSC-2085; Scott et al., 1991, sample TO-2305). These dates represent the time of formation of glaciomarine deltas on the northeast coast of Newfoundland (Grant, 1974). A marine limit of approximately 75 m above sea level (asl) is indicated by the contact between the topset and foreset beds of a delta in Halls Bay (Scott et al., 1991).

The southwestern part of the study area was also undergoing deglaciation between 12600 and 12300 BP in the Grand Lake Basin (Batterson and Catto, 2001; Batterson, 2003; Bell et al., 2003). Radiocarbon dates from the Stephenville area indicate that the Grand Lake Basin was deglaciated sometime after 12600 BP , allowing for drainage from inland melting glaciers to reach St. George's Bay. Glacial Lake Howley developed in front of a rapidly retreating ice margin in the Grand Lake Basin. Impounded water formed a long, narrow glacial lake, up to 135 km long and 10 km wide, with a surface area of $650 \mathrm{~km}^{2}$ (Figure 4). The water level was controlled by the elevation of an outlet at the southwestern end of Grand Lake. Subsequent lowering of the lake was controlled by the opening of topographically lower outlets, as the ice retreated to the northeast. The northeastern extent is inferred from the presence of rhythmically bedded silt and clay, providing evidence for standing water in the Gillards Lake area and in the canal (watershed divide) between Indian Brook and Birchy Lake (Lundqvist, 1965; Batterson, 2003). Batterson (op. cit.) concluded that the ice dam controlling the extent of the lake was east of Birchy Lake. Final

Figure 3. Pattern of glaciation on the Island of Newfoundland (after Shaw et al., 2006). A) Retreat of ice onto land and the location of ice divides at 12 000BP (~14 000 Calendar years); B) Map of Newfoundland showing the approximate location of remnant ice caps as the Newfoundland Ice Cap disintegrated (modified after Grant, 1974).


Figure 4. Paleogeography of glacial Lake Howley (modified after Batterson, 2003).
drainage of the lake was through a spillway currently occupied by Junction Brook, northeast of Howley (Figure 4). The lake emptied about 12300 BP, based on the elevation of the deltas at the head of Deer Lake. Continued ice retreat resulted in shifting ice centres, and formed isolated ice caps.

## ICE-FLOW HISTORY

Three ice-flow directions were mapped by Tucker (1974); Alley and Slatt (1975); Liverman et al. (1991); Taylor and Vatcher (1993) and Klassen (1994b) for NTS map areas 12H/01, 07 and 08 (Figure 5). From these, two major ice-flow phases were recognized, although the relative age of the remaining ice-flow direction was not determined. Phase 1, the oldest and dominant ice-flow, was from the northeast and originated from an ice centre connecting the Long Range Mountains and the Topsails. The younger Phase 2 was a northward to northwestward flow in NTS map area $12 \mathrm{H} / 07$ and in the western part of NTS $12 \mathrm{H} / 08$. This flow was the result of further ice retreat, and isolation of the ice cap situated over the Topsails. The northwestward flow identified in Phase 2, in the eastern part of NTS $12 \mathrm{H} / 08$, was from a minor ice cap in the Twin Lakes area (Taylor and Liverman, 2000). Evidence for an eastward flow was identified on the northwestern side of Halls

Figure 5. Location of striations (red) measured in 2013. Black symbols represent striations measured prior to 2013 (Geological Survey of Newfoundland and Labrador, 2013). Large arrows indicate the generalized multiphase ice-flow history for the area.

Bay, and is attributed to late-stage local ice flow towards Halls Bay; however, its relative age has not been determined (Liverman et al., 1991).

## SURFICIAL GEOLOGY

The diamicton (till) deposits of the Sheffield Lake-Indian Pond area have been described by Alley and Slatt (1975) as having a two-component stratigraphy: a lower red till, overlain by an upper grey till. The lower red till is gravelly, oxidized, fissile, and contains clasts from local sources, whereas the upper grey till is sandy, slightly oxidized, massive and contains clasts from more distant sources.

Liverman et al. (1991) mapped the Springdale NTS map area (12H/08) and reported the sediment cover over the area to be variable, with the valleys containing a thick surficial fill of eroded diamicton, as well as glaciofluvial and glaciomarine sediments related to marine incursion. Upland areas are mainly composed of diamicton, bogs and outcrops of bedrock. In addition to veneers and blanket deposits, the diamicton also forms hummocks and ridges.

Relief is high between the southeastern shore of Sandy Lake, in the southwestern corner of NTS map area $12 \mathrm{H} / 07$, and Black Brook in the northwest corner of NTS $12 \mathrm{H} / 08$; this area includes Sheffield Lake. It is low between the northeastern shore of Sandy Lake (NTS 12H/07) and Indian Pond (12H/07), between Pauls Lake (NTS 12H/01) and Sheppardville (NTS 12H/08) and east of the Topsails in the southeastern corner of NTS 12H/02. Relief is moderate to high elsewhere.

## REGIONAL SURFICIAL SEDIMENT SAMPLING

## SAMPLING AND SAMPLE PREPARATION METHOD

During the survey, till samples were collected at 1028 sites from the B ( 30 sites), C ( 877 sites) and BC (121 sites) soil horizons. Samples were collected from test pits (495 sites; median depth 49.5 cm ), ditches ( 335 sites; median depth 74 cm ), roadcuts ( 129 sites; median depth 87 cm ), mudboils ( 17 sites; median depth 57 cm ), pits/quarries ( 25 sites; median depth 83 cm ) and a riverbank section ( 1 site; depth 150 cm ), while the source of 26 samples was not recorded. Marine and fluvial or glaciofluvial sediments were avoided because of the probability of reworking and the difficulty in defining distances and directions of transport. Sample spacing was controlled by access and presence of till, but was generally one sample every 1 km along all primary and secondary roads for vehicle-based sampling, and one sample every $4 \mathrm{~km}^{2}$ for helicopter-supported sampling. Field duplicate samples were collected at 55 sites: an overall frequency of one in 19. The results of the field-duplicate analyses are presented in a later section.

In the field, samples were placed in kraft-paper sample bags, and sent to the Geological Survey's geochemical laboratory in St. John's, where they were air-dried in ovens at $60^{\circ} \mathrm{C}$ and dry-sieved to $<180 \mu$ (minus 80 mesh) in stainless steel sieves.

## ANALYSIS

The $<180 \mu$ fraction was subjected to inductively-coupled plasma optical emission spectrometry (ICP-OES) analysis after multi-acid ( $\mathrm{HF} / \mathrm{HCl} / \mathrm{HNO}_{3} / \mathrm{HClO}_{4}$ ) digestion at the Geological Survey's geochemical laboratory, and instrumental neutron activation analysis (INAA) at Becquerel Laboratories (now known as Maxxam Analytics) in Mississauga, Ontario. Of the 47 elements determined, 12 were determined by both ICP-OES and INAA (As, $\mathrm{Ba}, \mathrm{Ce}, \mathrm{Co}, \mathrm{Cr}, \mathrm{Fe}$, $\mathrm{La}, \mathrm{Mo}, \mathrm{Na}, \mathrm{Rb}, \mathrm{Sc}$ and Zr ). Loss-on-ignition (LOI) was determined gravimetrically at the Geological Survey's geochemical laboratory. A complete list of analytical variables is given in Table 3, and the analytical methods are described in detail in Appendix A.

Table 3. Geochemical variables with analytical method, units, detection limit and number of analyses below the detection limit; one sample was only analyzed for INAA elements and LOI and one sample was only analyzed for LOI

| Element | Method | Units | D.L. | <D.L. | Element | Method | Units | D.L. | <D.L. |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| Ag | AAS | ppm | 0.1 | 707 | Mg | ICP-OES | \% | 0.01 | 0 |
| Al | ICP-OES | \% | 0.01 | 0 | Mn | ICP-OES | ppm | 1 | 0 |
| As | INAA | ppm | 0.5 | 20 | Mo | INAA | ppm | 1 | 938 |
| Au | INAA | ppb | 1 | 876 | Na | INAA | \% | 0.1 | 0 |
| Ba | INAA | ppm | 50 | 0 | Na | ICP-OES | \% | 0.01 | 0 |
| Ba | ICP-OES | ppm | 1 | 0 | Nb | ICP-OES | ppm | 1 | 0 |
| Be | ICP-OES | ppm | 0.1 | 0 | Ni | ICP-OES | ppm | 1 | 0 |
| Br | INAA | ppm | 1 | 42 | P | ICP-OES | ppm | 1 | 0 |
| Ca | ICP-OES | \% | 0.01 | 0 | Pb | ICP-OES | ppm | 1 | 0 |
| Cd | ICP-OES | ppm | 0.1 | 590 | Rb | INAA | ppm | 5 | 1 |
| Ce | INAA | ppm | 3 | 0 | Rb | ICP-OES | ppm | 1 | 0 |
| Ce | ICP-OES | ppm | 1 | 0 | Sb | INAA | ppm | 0.1 | 35 |
| Co | INAA | ppm | 2 | 144 | Sc | INAA | ppm | 0.1 | 0 |
| Co | ICP-OES | ppm | 1 | 57 | Sc | ICP-OES | ppm | 0.1 | 0 |
| Cr | INAA | ppm | 10 | 75 | Se | INAA | ppm | 1 | 1026 |
| Cr | ICP-OES | ppm | 1 | 0 | Sm | INAA | ppm | 0.1 | 0 |
| Cs | INAA | ppm | 0.5 | 46 | Sr | ICP-OES | ppm | 1 | 0 |
| Cu | ICP-OES | ppm | 1 | 0 | Ta | INAA | ppm | 0.2 | 0 |
| Dy | ICP-OES | ppm | 0.1 | 0 | Tb | INAA | ppm | 0.5 | 9 |
| Eu | INAA | ppm | 0.5 | 110 | Th | INAA | ppm | 0.5 | 0 |
| Fe | INAA | \% | 0.1 | 0 | Ti | ICP-OES | ppm | 1 |  |
| Fe | ICP-OES | \% | 0.01 | 0 | U | INAA | ppm | 0.1 | 0 |
| Hf | INAA | ppm | 1 | 0 | V | ICP-OES | ppm | 1 | 0 |
| K | ICP-OES | \% | 0.01 | 0 | W | INAA | ppm | 1 | 785 |
| La | INAA | ppm | 1 | 0 | Y | ICP-OES | ppm | 1 | 0 |
| La | ICP-OES | ppm | 1 | 0 | Yb | INAA | ppm | 0.5 | 2 |
| Li | ICP-OES | ppm | 0.1 | 0 | Zn | ICP-OES | ppm | 1 | 0 |
| LOI | Gravimetric | \% | 0.1 | 0 | Zr | ICP-OES | ppm | 1 | 0 |
| Lu | INAA | ppm | 0.05 | 4 |  |  |  |  |  |

Analyses of 1028 till samples, excluding field duplicates, are presented as part of this report in a comma-delimited digital attachment in Appendix B, where the analytical variables are labelled with a combination of the element name, a numeric code denoting the analytical method, and the unit of measurement.

## INCORPORATION OF EARLIER DATA

Analyses of 276 samples collected by R. Klassen (1994b), D. Liverman (Davenport et al., 1996) and D. Taylor (Liverman et al., 2000) are also included as comma-delimited files in Appendices C, D and E, respectively. These analyses are included so that complete sampling coverage of the study areas can be achieved with the recent data presented with this report. The Liverman and Taylor data are the same as those currently incorporated into the Geoscience Atlas of Newfoundland and Labrador, and are based on the same sample-preparation and analytical methods as those applied in the current study, although analyses for several elements are missing from some of the Liverman samples from 1990. The Klassen samples were subjected to a preparation procedure which followed GSC protocols, and differed from those of the GSNL, viz. sieving to $<63 \mu$ ( 230 mesh ), and digestion with aqua regia. They were subjected to ICP-OES analysis for $\mathrm{Ag}, \mathrm{Al}, \mathrm{As}, \mathrm{Ba}, \mathrm{Be}, \mathrm{Ca}, \mathrm{Cd}, \mathrm{Co}, \mathrm{Cr}, \mathrm{Cu}, \mathrm{Fe}, \mathrm{K}, \mathrm{La}, \mathrm{Mg}, \mathrm{Mn}, \mathrm{Mo}, \mathrm{Na}, \mathrm{Ni}, \mathrm{Pb}, \mathrm{Sc}, \mathrm{Sr}, \mathrm{Ti}, \mathrm{V}$ and Zn , as well as $\mathrm{Bi}, \mathrm{Ga}, \mathrm{Hg}, \mathrm{Sb}, \mathrm{Tl}, \mathrm{U}$, and W which are not in the GSNL's ICP-OES package. Analyses by INAA were for $\mathrm{Au}, \mathrm{As}, \mathrm{Ba}, \mathrm{Br}, \mathrm{Co}, \mathrm{Cr}, \mathrm{Cs}, \mathrm{Fe}, \mathrm{Hf}, \mathrm{Mo}, \mathrm{Na}, \mathrm{Rb}, \mathrm{Sb}, \mathrm{Sc}, \mathrm{Se}, \mathrm{Ta}, \mathrm{Th}$, $\mathrm{U}, \mathrm{W}, \mathrm{Zn}, \mathrm{La}, \mathrm{Ce}, \mathrm{Sm}, \mathrm{Eu}, \mathrm{Tb}, \mathrm{Yb}$ and Lu , as well as $\mathrm{Ag}, \mathrm{Ca}, \mathrm{Hg}, \mathrm{Ir}, \mathrm{Ni}, \mathrm{Sn}, \mathrm{Sr}, \mathrm{Nd}$ which are, similarly, not analyzed by Becquerel for the GSNL. An attempt has been made to establish a relationship between the contents of the $<63 \mu$ and $<180 \mu$ that can be used for levelling purposes; this is described later.

It should be noted that an adjustment has already been applied to the Klassen (1994b) analyses currently in the Geoscience Atlas. The procedure is described in a Help file within the Atlas. Levelling equations were derived to relate analyses of the same samples, subjected on the one hand to aqua regia digestion (to simulate the GSC procedure) and on the other, to multi-acid digestion (as followed by the GSNL). However, the results of this exercise are considered to be unsuitable for the current study because the samples were sieved to $<63 \mu$, whereas the most recent samples were sieved to $<180 \mu$; furthermore, what were probably strongly positively skewed data were not subjected to log-transformation prior to the regression and the latter's results are likely to reflect this skew.

For the current study, paired analyses of the same sample material ( $<63 \mu$ /aqua regia on the one hand, and $<180 \mu /$ multi-acid) were not available. Consequently, pairs of samples, each comprising one from the Klassen (1994b) program and one from the current program and separated by a distance of less than 2 km , were selected for comparison and for the establishment of relationships. First, for each element the two populations were compared for equality of means using a paired ttest (R-Project, 2016) after log-transformation; second, the log-transformed analyses from the current program were regressed, as dependent variables, against their independent counterparts in the Klassen (op. cit.) dataset.

The results of the statistical tests are shown in Table 4, which lists the following:

1. The probability that the means of the two populations for that element were the same, as estimated from the paired t-test, with a cut-off of 0.05 (R.G. Garrett, personal communication, 2016);
2. The statistical measure of how close the data are to the fitted regression line, also known as the coefficient of determination or ' $\mathrm{R}^{2}$ ' (while the $\mathrm{R}^{2}$ statistic is more frequently quoted as a measure of regression quality, its significance is dependent on the number of pairs of values from which it is calculated; this is variable, because pairs of 'undetectable' analyses were omitted from the regression; therefore $\mathrm{R}^{2}$ is an approximation, albeit a useful one);
3. The ' F ratio' of the variance of the dependent variable's raw values to that of its residuals;
4. The significance of the F ratio, viewed alternately as the probability that the regression relationship between the earlier and later samples could be achieved with random numbers, with a cut-off of 0.001 , and
5. The regression coefficients and constants for the elements where the relationship between the two variables was shown to be significant.

The regression coefficients and constants can be used to construct levelling equations if the harmonization of the earlier and later samples is desired. However, it is important to log-transform the input (Klassen, 1994b) values prior to calculation of levelled values, and to antilog-transform the latter prior to their incorporation in a merged database.

The statistical significance of the two tests, for all elements, is summarized graphically in Figure 6. In this diagram, the elements in the upper left-hand section (Sc1, Th1, U1, Cr1, Sb1, Fe1, $\mathrm{Co} 2, \mathrm{~Tb} 1$ and Sm 1 ) are those for which regression relationships are significant, although conversion is not necessary because the means (between the Klassen (1994b) samples and their neighbouring samples from the current study) are not significantly different. The upper right-hand section represents an element ( Brl only) for which regression relationship is not significant, but whose means are not significantly different. Application of a regression equation to harmonize the earlier and later analyses in both these categories is not necessary. Significantly, all except Co2 are elements analyzed by INAA, and not subject to the differing digestion reagents that preceded the ICP-OES analyses.

The large lower left-hand section includes the elements of which the means are not the same in the two populations, but whose regression relationships are significant; therefore, the regression equations linking the two populations can be used to harmonize the Klassen (1994b) analyses for $\mathrm{Al} 2, \mathrm{As} 1, \mathrm{Ba} 1, \mathrm{Ba} 2, \mathrm{Ca} 2, \mathrm{Ce} 1, \mathrm{Co} 1, \mathrm{Cr} 2, \mathrm{Cu} 2, \mathrm{Fe} 2, \mathrm{Hf1}, \mathrm{Lu} 1, \mathrm{Mg} 2, \mathrm{Mn} 2, \mathrm{Na} 1, \mathrm{Ni} 2, \mathrm{Ta} 1$, Rb1, Sc2, Sr2, V2 and Yb1. The lower right-hand section of the diagram shows elements whose concentration levels are significantly different between the two sets of analyses, but whose regression fit was not good enough to apply regression equations.
Table 4. Results of statistical tests of nearest neighbour regression

| Element | $\mathrm{t}_{\text {Sig }}$ | $\mathbf{R}^{2}$ | F ratio | $\mathbf{F}_{\text {sig }}$ | Constant | Coefficient | Element | $\mathrm{t}_{\text {Sig }}$ | $\mathbf{R}^{2}$ | F ratio | $\mathbf{F}_{\text {sig }}$ | Constant | Coefficient |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| Al2 | $2.20 \mathrm{E}-16$ | 0.20 | 43.6 | $5.03 \mathrm{E}-10$ | 0.8039 | 0.0743 | La2 | $2.20 \mathrm{E}-16$ | 0.00 | 0.03 | 0.86887 |  |  |
| As1 | $6.86 \mathrm{E}-05$ | 0.28 | 64.9 | $1.65 \mathrm{E}-13$ | 0.4192 | 0.4007 | Lu1 | $2.20 \mathrm{E}-16$ | 0.45 | 138.5 | $1.06 \mathrm{E}-23$ | -0.2129 | 0.5764 |
| As2 | $2.20 \mathrm{E}-16$ | 0.02 | 4.7 | 0.032296 |  |  | Mg2 | $2.20 \mathrm{E}-16$ | 0.44 | 130.9 | $9.59 \mathrm{E}-23$ | 0.1754 | 0.6359 |
| Ba1 | $4.15 \mathrm{E}-05$ | 0.10 | 20.5 | 0.000011 | 1.7924 | 0.3169 | Mn2 | $2.20 \mathrm{E}-16$ | 0.28 | 67.0 | $6.47 \mathrm{E}-14$ | 1.9700 | 0.3294 |
| Ba2 | $2.20 \mathrm{E}-16$ | 0.08 | 15.9 | 0.000101 | 2.4954 | 0.0662 | Na1 | $2.20 \mathrm{E}-16$ | 0.19 | 40.0 | 2.18E-09 | 0.2046 | 0.5998 |
| Be2 | $2.20 \mathrm{E}-16$ | 0.00 | 1.5 | 0.228643 |  |  | Na 2 | $2.20 \mathrm{E}-16$ | 0.02 | 3.7 | 0.055999 |  |  |
| Br1 | 0.5518 | 0.00 | 1.3 | 0.261637 |  |  | Ni2 | $2.20 \mathrm{E}-16$ | 0.31 | 73.8 | $6.77 \mathrm{E}-15$ | 0.8338 | 0.2894 |
| Ca2 | $2.20 \mathrm{E}-16$ | 0.60 | 256.6 | $1.01 \mathrm{E}-35$ | 0.4915 | 1.0060 | Pb2 | $2.11 \mathrm{E}-08$ | 0.03 | 6.2 | 0.013968 |  |  |
| Ce1 | 0.02172 | 0.16 | 32.9 | $4.46 \mathrm{E}-08$ | 0.8819 | 0.4789 | Rb1 | $2.06 \mathrm{E}-13$ | 0.54 | 168.2 | $7.23 \mathrm{E}-26$ | 0.6283 | 0.6614 |
| Co1 | 0.01096 | 0.32 | 81.9 | $1.22 \mathrm{E}-15$ | 0.3298 | 0.6893 | Sb1 | 0.1883 | 0.38 | 103.8 | $3.02 \mathrm{E}-19$ |  |  |
| Co 2 | 0.1179 | 0.31 | 78.1 | $1.22 \mathrm{E}-15$ |  |  | Sc1 | 0.3089 | 0.67 | 348.0 | $1.08 \mathrm{E}-42$ |  |  |
| Cr1 | 0.1443 | 0.42 | 121.8 | $1.13 \mathrm{E}-21$ |  |  | Sc2 | $2.20 \mathrm{E}-16$ | 0.35 | 94.1 | $5.79 \mathrm{E}-18$ | 0.8198 | 0.4846 |
| Cr2 | $2.20 \mathrm{E}-16$ | 0.50 | 172.3 | $1.40 \mathrm{E}-27$ | 0.5208 | 0.7484 | Sm1 | 0.4612 | 0.12 | 24.5 | 0.000002 |  |  |
| Cs1 | 0.000301 | 0.00 | 1.4 | 0.243013 |  |  | Sr2 | $2.20 \mathrm{E}-16$ | 0.56 | 220.7 | $1.79 \mathrm{E}-32$ | 1.1697 | 0.7993 |
| Cu 2 | $2.20 \mathrm{E}-16$ | 0.43 | 128.3 | $1.74 \mathrm{E}-22$ | 0.1442 | 0.7244 | Ta1 | $1.30 \mathrm{E}-05$ | 0.39 | 43.0 | $1.06 \mathrm{E}-08$ | -0.0720 | 0.6450 |
| Eu1 | $2.28 \mathrm{E}-11$ | 0.00 | 0.8 | 0.37267 |  |  | Tb1 | 0.1615 | 0.30 | 64.4 | $2.71 \mathrm{E}-13$ |  |  |
| Fe1 | 0.9059 | 0.37 | 97.2 | $3.27 \mathrm{E}-18$ |  |  | Th1 | 0.694 | 0.63 | 292.8 | $9.93 \mathrm{E}-39$ |  |  |
| Fe 2 | $2.20 \mathrm{E}-16$ | 0.26 | 59.0 | $1.28 \mathrm{E}-12$ | 0.2948 | 0.5760 | Ti2 | $2.20 \mathrm{E}-16$ | 0.43 | 129.0 | $1.44 \mathrm{E}-22$ | 0.1916 | 0.6785 |
| Hf1 | $2.60 \mathrm{E}-09$ | 0.65 | 317.4 | $1.22 \mathrm{E}-40$ | 0.2893 | 0.6860 | U1 | 0.4791 | 0.54 | 190.1 | 4.15E-29 |  |  |
| K2 | $2.20 \mathrm{E}-16$ | 0.08 | 15.8 | 0.000105 | -0.1151 | -0.1502 | V2 | $2.20 \mathrm{E}-16$ | 0.75 | 510.7 | $1.14 \mathrm{E}-52$ | -0.0459 | 1.0608 |
| La1 | $2.20 \mathrm{E}-16$ | 0.01 | 3.0 | 0.086016 |  |  | Yb1 | $2.20 \mathrm{E}-16$ | 0.59 | 247.0 | $6.99 \mathrm{E}-35$ | 0.1185 | 0.6497 |
| $\mathbf{t}_{\text {sig }}$ is the probability that the means of the earlier (Klassen) samples and samples from the current study are the same $\mathbf{R}^{2}$ is a measure of how close the data are to the fitted regression line (minimum 0.0 , maximum 1.0) |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| The Constant and Coefficient can used to level the Klassen samples to harmonize them with the later samples, where the means are dissimilar and the regr relationship is sufficiently strong. Input values should be $\log _{10}$-transformed, and output values antilog-transformed, in the application of these equations |  |  |  |  |  |  |  |  |  |  |  |  |  |



Figure 6. Graphical representation of significance of statistical tests. See text for details.
The value of $\mathrm{R}^{2}$ exceeds 0.5 for $\mathrm{Ca} 2, \mathrm{Cr} 2$, Hf1, Rb1, Sc1, Sr2, Th1, U1, V2 and Yb1. This value was used by Davenport et al. (1996) as a cut-off for judging the strength of the correlation between analyses of the same sample material, which provides a measure of the success of the current exercise.

## QUALITY ASSURANCE

Quality assurance in the laboratory consisted of insertion of one certified reference standard (TILL-1, TILL-2, TILL-3 or TILL-4; Lynch, 1996), and one analytical duplicate in every sequence of 20 samples. Standard analyses for both analytical methods, in 2016 and 2017, and duplicate analyses for INAA and ICP-OES were satisfactory and no re-analyses were necessary.

## ACCURACY

The relationship of the reported analysis of a standard to its recommended value (normally, the arithmetic mean of a large number of similar analyses of the standard, with outliers excluded) depends on a number of factors. For analyses that follow some kind of digestion (in this case, $\mathrm{HF} / \mathrm{HCl} / \mathrm{HNO}_{3} / \mathrm{HClO}_{4}$ ) the refractory nature of certain minerals may lead to the recovery of certain elements hosted in those minerals (i.e., the analyses of the standard expressed as a percentage of the recommended value) being less than $100 \%$; in some cases, considerably so (Table 5).

Table 5. Accuracy of ICP-OES analyses

|  |  |  |  |  |  |
| :--- | :---: | :---: | :---: | :---: | :---: |
|  | Till-1 | Till-2 | Till-3 | Till-4 | Average |
| $\mathbf{A l}$ | 0.92 | 0.93 | 0.93 | 0.95 | $93 \%$ |
| $\mathbf{A s}$ | 0.87 | 0.93 | 0.93 | 0.94 | $92 \%$ |
| $\mathbf{B a}$ | 0.96 | 0.98 | 0.98 | 0.97 | $97 \%$ |
| $\mathbf{B e}$ | 0.61 | 0.80 | 0.61 | 0.78 | $70 \%$ |
| $\mathbf{C a}$ | 0.91 | 0.98 | 0.96 | 0.98 | $96 \%$ |
| $\mathbf{C e}$ | 0.98 | 0.91 | 1.02 | 0.90 | $95 \%$ |
| $\mathbf{C o}$ | 0.73 | 0.79 | 0.73 | 0.73 | $75 \%$ |
| $\mathbf{C r}$ | 0.72 | 0.75 | 0.71 | 0.66 | $71 \%$ |
| $\mathbf{C u}$ | 0.85 | 0.90 | 0.88 | 0.92 | $89 \%$ |
| $\mathbf{F e}$ | 0.96 | 0.98 | 0.99 | 0.99 | $98 \%$ |
| $\mathbf{K}$ | 0.92 | 0.92 | 0.91 | 0.91 | $92 \%$ |
| $\mathbf{L a}$ | 0.92 | 0.95 | 0.90 | 0.91 | $92 \%$ |
| $\mathbf{L i}$ | 1.01 | 0.92 | 1.00 | 0.94 | $97 \%$ |
| $\mathbf{M g}$ | 0.90 | 0.95 | 0.94 | 0.92 | $93 \%$ |
| $\mathbf{M n}$ | 0.95 | 0.99 | 0.96 | 1.01 | $98 \%$ |
| $\mathbf{M o}$ |  | 0.94 |  | 0.92 | $93 \%$ |
| $\mathbf{N a}$ | 0.96 | 0.99 | 0.97 | 0.96 | $97 \%$ |
| $\mathbf{N b}$ | 0.84 | 0.78 | 0.80 | 0.87 | $82 \%$ |
| $\mathbf{N i}$ | 1.04 | 1.09 | 1.01 | 1.11 | $106 \%$ |
| $\mathbf{P}$ | 0.93 | 0.91 | 0.95 | 0.96 | $94 \%$ |
| $\mathbf{P b}$ | 0.72 | 0.82 | 0.81 | 0.92 | $82 \%$ |
| $\mathbf{R b}$ | 1.04 | 0.93 | 0.86 | 0.84 | $92 \%$ |
| $\mathbf{S c}$ | 1.12 | 1.11 | 1.09 | 1.16 | $112 \%$ |
| $\mathbf{S r}$ | 1.08 | 1.17 | 1.12 | 1.20 | $114 \%$ |
| $\mathbf{T i}$ | 0.80 | 0.87 | 0.93 | 0.91 | $88 \%$ |
| $\mathbf{V}$ | 0.93 | 1.03 | 0.97 | 0.97 | $97 \%$ |
| $\mathbf{Y}$ | 0.58 | 0.45 | 0.63 | 0.41 | $52 \%$ |
| $\mathbf{Z n}$ | 0.89 | 0.91 | 0.90 | 0.95 | $91 \%$ |
| $\mathbf{Z r}$ | 0.08 | 0.23 | 0.31 | 0.20 | $21 \%$ |
|  |  |  |  |  |  |

Calculated as the arithmetic mean of multiple analyses of each certified reference standard, divided by the recommended value for the standard.

Elements analyzed by ICP displaying mean values within $\pm 5 \%$ of $100 \%$, indicating that the multi-acid digestion is near total, consist of $\mathrm{Ba}, \mathrm{Ca}, \mathrm{Fe}, \mathrm{Li}, \mathrm{Mn}, \mathrm{Na}$, and V ; analyses of $\mathrm{Al}, \mathrm{As}, \mathrm{Ce}, \mathrm{Cu}, \mathrm{K}, \mathrm{La}$, $\mathrm{Mg}, \mathrm{Mo}, \mathrm{P}, \mathrm{Rb}, \mathrm{Ti}$ and Zn lie between $80 \%$ and $95 \%$ of the recommended values; Co and Cr show recoveries of $75 \%$ and $71 \%$, respectively; and $\mathrm{Be}, \mathrm{Y}$ and Zr show recoveries of $70 \%$ or less. At $21 \%$, the recovery of Zr is probably a consequence of the element's residence in zircon, known to be resistant to HF digestion (but whose recommended values were derived after fusion, by which zircon is susceptible to digestion). The poor recoveries of Be and Cr may also be explainable by the refractory nature of their host minerals, most probably beryl and chromite, respectively; those of Y and Co are more difficult to explain, because the recoveries of the rare-earth elements, on the one hand, and chalcophile or siderophile elements on the other, are generally much better. The elements $\mathrm{Ni}, \mathrm{Sc}$ and Sr show recoveries in excess of $105 \%$; this may be due to calibration issues, or the possible oxidation of minerals hosting these elements in the standards, resulting in more of those elements being available for digestion, although it is not known what those minerals are.

Recoveries of INAA analyses (Table 6) would be expected to be more accurate because they do not depend on the dissolution of the sample. Elements analyzed by ICP displaying mean values within $\pm 5 \%$ of $100 \%$ are As, $\mathrm{Ba}, \mathrm{Ce}, \mathrm{Co}, \mathrm{Cr}, \mathrm{Fe}$, $\mathrm{Hf}, \mathrm{La}, \mathrm{Na}, \mathrm{Rb}$ and Th ; analyses of $\mathrm{Br}, \mathrm{Eu}, \mathrm{Lu}, \mathrm{Mo}, \mathrm{Sb}, \mathrm{Sm}, \mathrm{Ta}, \mathrm{U}, \mathrm{W}, \mathrm{Yb}$ and Zr lie between $80 \%$ and $95 \%$ of the recommended values; while Tb shows a recovery of $77 \%$. The elements $\mathrm{Au}, \mathrm{Cs}$, and Sc are overestimated by more than $5 \%$. These recoveries, particularly those of the multi-acid/ICP-OES elements, are very similar to those reported by Brushett and Amor (2016).

Table 6. Accuracy of INAA analyses

|  | Till-1 | Till-2 | Till-3 | Till-4 | Average |
| :--- | :---: | :---: | :---: | :---: | :---: |
| $\mathbf{A s}$ | 0.97 | 1.01 | 1.00 | 0.99 | $99 \%$ |
| $\mathbf{A u}$ | 1.90 | 0.37 | 1.32 | 0.91 | $113 \%$ |
| $\mathbf{B a}$ | 1.00 | 0.99 | 0.99 | 0.94 | $98 \%$ |
| $\mathbf{B r}$ | 0.95 | 1.02 | 0.89 | 0.86 | $93 \%$ |
| $\mathbf{C e}$ | 1.07 | 1.04 | 1.01 | 1.07 | $105 \%$ |
| $\mathbf{C o}$ | 1.00 | 1.21 | 0.96 | 0.90 | $101 \%$ |
| $\mathbf{C r}$ | 0.99 | 1.02 | 1.00 | 0.87 | $97 \%$ |
| $\mathbf{C s}$ | 1.04 | 1.00 | 1.14 | 1.07 | $107 \%$ |
| $\mathbf{E u}$ | 1.18 | 1.62 |  | 0.01 | $93 \%$ |
| $\mathbf{F e}$ | 1.02 | 1.03 | 1.00 | 0.98 | $101 \%$ |
| $\mathbf{H f}$ | 1.05 | 0.97 | 0.77 | 1.16 | $99 \%$ |
| $\mathbf{L a}$ | 1.01 | 1.08 | 0.94 | 1.02 | $101 \%$ |
| $\mathbf{L u}$ | 0.91 | 0.91 | 1.00 | 0.94 | $94 \%$ |
| $\mathbf{M o}$ |  | 0.74 |  | 1.01 | $87 \%$ |
| $\mathbf{N a}$ | 1.02 | 1.03 | 1.00 | 0.98 | $100 \%$ |
| $\mathbf{R b}$ | 1.01 | 1.04 | 1.02 | 1.03 | $103 \%$ |
| $\mathbf{S b}$ | 0.98 | 1.03 | 0.88 | 0.93 | $95 \%$ |
| $\mathbf{S c}$ | 1.13 | 1.11 | 1.03 | 1.10 | $109 \%$ |
| $\mathbf{S m}$ | 1.07 | 1.10 | 0.11 | 1.08 | $84 \%$ |
| $\mathbf{T a}$ | 1.06 | 1.04 | 0.70 | 0.98 | $95 \%$ |
| $\mathbf{T b}$ | 0.91 | 1.04 | 0.24 | 0.89 | $77 \%$ |
| $\mathbf{T h}$ | 1.02 | 1.00 | 1.04 | 0.97 | $101 \%$ |
| $\mathbf{U}$ | 0.91 | 0.99 | 0.94 | 0.94 | $94 \%$ |
| $\mathbf{W}$ |  | 0.92 |  | 0.95 | $94 \%$ |
| $\mathbf{Y b}$ | 0.95 | 0.92 | 0.87 | 0.84 | $90 \%$ |
| $\mathbf{Z r}$ | 0.90 | 0.94 | 0.70 | 0.94 | $87 \%$ |

## CONTROL CHARTS

The analyses of the standards TILL-1, TILL-2, TILL-3 and TILL-4 are displayed in control charts in Appendix F. Standard TILL-2 was not analyzed in 2013 due to temporary exhaustion of supplies; charts for certain other elements are omitted because they were undetectable in the establishment of recommended values.

Generally, the random error associated with geochemical analyses is normally distributed; this means that approximately $5 \%$ of the analyses will fall outside the 'limits of acceptability', without indicating a problem with the analysis of the sequence of samples within which that standard was analyzed. However, a succession of analyses, all of which fall outside of the same limit, is cause for concern; as is a single standard analysis if its deviance from the recommended value is extreme, or a series of standard analyses for different elements, all of which are deviant. An example of the last situation can be found in the charts of ICP-OES analysis of standard TILL-1, in sample 7832280, from 2012, which shows extremely low values of almost all elements with the exception of Be and K , which are extremely high. None of the other standards (which are normally inserted in turn) show similar deviance and so the sequence of samples within which the error occurred, and requiring re-analysis, is short ( 39 samples). Reanalysis indicated that lab numbers had been assigned to an even shorter sequence of ten samples, which included the standard, in the wrong order. The new standard analysis was satisfactory in every respect and the new analyses were added to the database to replace the originals.

## FIELD AND ANALYTICAL VARIABILITY

The overall precision of the field and analytical duplicates is shown in Table 7, and displayed graphically in bar-chart form in Figure 7. This single parameter does not take into account the variability of precision with concentration level. Therefore, results of analytical and field duplicates for all elements are displayed graphically as plots devised by Thompson and Howarth (1978) in Appendix G. Figure 8 shows examples where elements' repeatability in field duplicates varies

Table 7. Overall analytical and field precision

| Element | Precision |  | Element | Precision |  |
| :---: | :---: | :---: | :---: | :---: | :---: |
|  | Analytical | Field |  | Analytical | Field |
| Al2 | 4.2 | 9.9 | Mg2 | 4.5 | 36.4 |
| As1 | 34.1 | 66.0 | Mn2 | 5.6 | 28.5 |
| As2 | 14.5 | 127.3 | Mo1 | 71.7 | 115.0 |
| Au1 | 146.6 | 176.8 | Mo2 | 82.0 | 92.1 |
| Ba1 | 10.3 | 21.8 | Na1 | 13.6 | 16.4 |
| Ba2 | 4.9 | 18.1 | Na2 | 4.0 | 13.3 |
| Be2 | 5.4 | 16.6 | Nb2 | 17.0 | 16.1 |
| Br1 | 56.7 | 81.4 | Ni2 | 17.3 | 49.8 |
| Ca2 | 4.0 | 18.3 | P2 | 6.6 | 44.4 |
| Cd2 | 85.2 | 132.7 | Pb2 | 23.1 | 95.2 |
| Ce1 | 22.2 | 33.5 | Rb1 | 18.6 | 19.7 |
| Ce2 | 14.3 | 30.8 | Rb2 | 15.5 | 22.9 |
| Co1 | 65.9 | 76.3 | Sb1 | 32.6 | 48.0 |
| Co2 | 43.1 | 45.9 | Sc1 | 15.5 | 40.4 |
| Cr1 | 82.4 | 89.8 | Sc2 | 4.1 | 32.4 |
| Cr 2 | 15.0 | 47.1 | Sm1 | 7.7 | 33.4 |
| Cs1 | 34.1 | 82.4 | Sr2 | 5.3 | 24.2 |
| Cu 2 | 21.4 | 71.0 | Ta1 | 23.8 | 23.8 |
| Dy2 | 13.5 | 23.4 | Tb1 | 19.2 | 28.6 |
| Eu1 | 136.5 | 123.8 | Th1 | 7.6 | 20.9 |
| Fe1 | 12.5 | 23.3 | Ti2 | 11.1 | 17.9 |
| Fe 2 | 5.1 | 27.1 | U1 | 11.2 | 26.8 |
| Hf1 | 11.8 | 26.8 | V2 | 6.0 | 29.9 |
| K2 | 4.0 | 18.9 | W1 | 66.7 | 120.0 |
| La1 | 14.5 | 33.8 | Y2 | 16.8 | 20.2 |
| La2 | 13.0 | 37.1 | Yb1 | 25.9 | 29.8 |
| Li2 | 5.8 | 28.1 | Zn2 | 4.7 | 25.2 |
| LOI | 8.4 | 27.8 | Zr1 | 116.7 | 134.8 |
| Lu1 | 41.1 | 35.4 | Zr2 | 9.4 | 18.2 |

significantly from the repeatability in analytical duplicates (Cu by ICP-OES and As1 by INAA), and where it does not (Nb2 by ICP-OES and Rb1 by INAA).

## DISPLAY OF GEOCHEMICAL DATA

The purpose of this report is to accompany the timely release of till-geochemical data. No geochemical maps or interpretation are presented.

Figure 7. Bar chart summarizing precision of field and analytical duplicates.

As (INAA): examples of elements for which field variability significantly exceeds analytical variability; and of Nb (ICP-OES) and Rb (INAA): examples of elements whose field variability does not significantly exceed analytical variability.

## SUMMARY

During the 2012 and 2013 surficial mapping field seasons of NTS map areas $12 \mathrm{H} / / 01, / 02, / 07$ and/08, 1028 till samples were collected. These samples have been analyzed by ICP-OES and INAA and checked for acceptable accuracy and precision. Where earlier analyses of the <63 micron fraction of till samples are significantly different from corresponding analyses of the $<180$ micron fraction in their near neighbours, and when relationships are statistically significant, levelling equations have been derived.

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## APPENDIX A: Analytical Methods

## Gravimetric Analysis (LOI)

Organic carbon content was estimated from the weight loss-on-ignition (LOI) during a controlled combustion in which 1 g aliquots of sample were gradually heated to $500^{\circ} \mathrm{C}$ in air over a 3 hour period.

## Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES)

For these analyses, the procedures outlined by Finch (1998) were followed, with minor revisions. One gram of sample was weighed into a 125 ml Teflon beaker, and $15 \mathrm{ml} \mathrm{HF}(\sim 48 \%), 5 \mathrm{ml}$ of concentrated $\mathrm{HCl}, 5 \mathrm{ml}$ of concentrated $\mathrm{HNO}_{3}$ and 2.5 ml of $70 \% \mathrm{HClO}_{4}$ was added to each sample. The samples were placed on a hotplate at $200^{\circ} \mathrm{C}$ and evaporated to dryness, after which 5 ml concentrated HCl and 45 ml deionized water were added and the samples returned to the hotplate at $100^{\circ} \mathrm{C}$. When the residue was completely dissolved the samples were removed, cooled and transferred to 50 ml volumetric flasks. One ml of $50 \mathrm{~g} / \mathrm{l}$ boric acid was added to each sample to remove any residual hydrofluoric acid. The samples were made up to volume and analyzed by ICP-OES (Licthe et al., 1987).

Values for the following elements were determined: $\mathrm{Al}, \mathrm{Ba}, \mathrm{Be}, \mathrm{Ca}, \mathrm{Ce}, \mathrm{Co}, \mathrm{Cr}, \mathrm{Cu}, \mathrm{Dy}, \mathrm{Fe}$, K, La, Li, Mg, Mn, Mo, Na, Nb, Ni, P, Pb, Rb, Sc, Sr, Ti, V, Y, Zn and Zr.

## Instrumental Neutron Activation Analysis (INAA)

These analyses were carried out at Maxxam Analytics (formerly Becquerel Laboratories), Mississauga, Ontario. An average of 24 g of sample was used for analysis and the samples were weighed and encapsulated in the Geochemical Laboratory of the Department of Natural Resources in St. John's. Samples were irradiated with flux wires and an internal standard (1 for 11 samples) at a thermal neutron flux of $7 \times 10^{11} \mathrm{n} / \mathrm{cm}^{2} \mathrm{~s}$. After 7 days (to allow $\mathrm{Na}^{24}$ to decay), samples were counted on a high purity Ge detector with a resolution of better than 1.7 KeV . Using the flux wires, the decay-corrected activities were compared to a calibration developed from multiple certified international reference materials. The standard present is only a check on accuracy of the analysis and is not used for calibration purposes. Ten to 30 percent of the samples were checked by remeasurement.

Total contents of the following elements were determined quantitatively: $\mathrm{As}, \mathrm{Au}, \mathrm{Ba}, \mathrm{Br}, \mathrm{Ce}$, $\mathrm{Co}, \mathrm{Cr}, \mathrm{Cs}, \mathrm{Eu}, \mathrm{Fe}, \mathrm{Hf}, \mathrm{La}, \mathrm{Lu}, \mathrm{Mo}, \mathrm{Na}, \mathrm{Rb}, \mathrm{Sb}, \mathrm{Sc}, \mathrm{Se}, \mathrm{Sm}, \mathrm{Ta}, \mathrm{Tb}, \mathrm{Th}, \mathrm{U}, \mathrm{W}, \mathrm{Yb}$ and Zr .

## Atomic-Absorption Spectrometry (AAS)

0.5 g of sample is weighed into each 10 ml digestion tube. 2 ml of nitric acid are added, and the tubes are capped and left overnight. The next day, the digestion tubes are heated in a digestion block at $90^{\circ} \mathrm{C}$ for two hours. The tubes are shaken every 30 minutes. When digestion is complete, the samples are cooled and made up to a final volume of 10 ml with distilled deionized water and analyzed for silver by ICP-OES (Lichte, 1987). Only Ag was analyzed by this method.

Appendices B-E are available as digital comma-separated files (.csv) and Appendices F and G as .pdf files through this link.

## APPENDIX B:

## Digital Data Listing for Samples Collected in 2012 to 2013

## Suffixes

1: INAA

2: ICP-OES after multiacid $\left(\mathrm{HF} / \mathrm{HCl} / \mathrm{HNO}_{3} / \mathrm{HClO}_{3}\right)$ digestion
6: AAS after nitric acid digestion.

## APPENDIX C:

## Analyses of Samples Collected by R. Klassen (1994a)

Analyses of Al2, As1, Ba1, Ba2, Ca2, Ce1, Co1, Cr2, Cu2, Fe2, Hf1, Lu1, Mg2, Mn2, Na1, Ni2, $\mathrm{Ta} 1, \mathrm{Rb} 1, \mathrm{Sc} 2, \mathrm{Sr} 2, \mathrm{~V} 2$ and Yb 1 have been levelled to harmonize them with analyses from later programs. Analyses of other elements have not been adjusted.

## APPENDIX D:

Analyses of Samples Collected by D.G. Liverman (Davenport et al., 1996)

## APPENDIX E:

Analyses of Samples Collected by D. Taylor (Taylor and Liverman, 2000)

## APPENDIX F:

## 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.). Closed circles represent samples collected in 2012 and analyzed in 2013; open circles represent samples collected in 2013 and analyzed in 2014. Standard TILL-2 was not analyzed in 2013; charts for certain other elements are omitted because the latter were undetectable in the establishment of recommended values.

## Suffixes

1: INAA
2: ICP-OES after multiacid $\left(\mathrm{HF} / \mathrm{HCl} / \mathrm{HNO}_{3} / \mathrm{HClO}_{4}\right)$ digestion

## Units

- Al2, $\mathrm{Ca} 2, \mathrm{Fe} 1, \mathrm{Fe} 2, \mathrm{~K} 2, \mathrm{Mg} 2, \mathrm{Na} 1, \mathrm{Na} 2$ 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, Sb1, Sc1, Sc2, Sm1, Sr2, $\mathrm{Ta} 1, \mathrm{~Tb} 1, \mathrm{Th} 1, \mathrm{Ti} 2, \mathrm{U} 1, \mathrm{~V} 2, \mathrm{Zn} 2$ and Zr 2 in parts per million (ppm).
- Aul in parts per billion (ppb).


## APPENDIX G:

## Thompson-Howarth Precision Plots of Field and Analytical Duplicates

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).

