



Mines

**GEOCHEMICAL DATA RELATED TO URANIUM
OCCURRENCES IN NEWFOUNDLAND
(NTS MAP AREAS 11O/14, 16, 11P/13, 14, 12B/09,
12H/03, 04, 06, 09, 11, 1L/13, 14, 1M/04, 05)**

G.W. Sparkes

Open File NFLD/3373

**St. John's, Newfoundland
September, 2019**

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SUMMARY

This open file release contains geochemical results for 111 rock samples collected in relation to GSNL regional studies of uranium mineralization in Newfoundland (NTS map areas 11O/14, 16, 11P/13, 14, 12B/09, 12H/03, 04, 06, 09, 11, 1L/13, 14, 1M/04, 05; Figure 1). Sample sites focus on known occurrences of uranium mineralization and include material from both outcrop and drillcore collected between 2007 and 2010. This report does not provide an interpretation of the geochemical data; however, for a discussion on some of the related occurrences, *see* Sparkes (2011; and references therein).

NOTES ON THE DATABASE

This database contains geochemical results of major-, trace-, and rare-earth-element-analyses of various rock types, collected from regional studies, across Newfoundland. The database includes sample location data in Universal Transverse Mercator (UTM) eastings and northings, provided in NAD 27 (Zone 21), along with brief sample descriptions (Appendix A). Several different sample types are included within the database: these include grab samples from outcrop, float representing samples from boulders of (inferred) local origin and core samples generated through mineral-exploration activities. The location data provided for core samples represents the collar location of the respective drillhole. The data is available in digital format (*i.e.*, *.csv, comma-separated value files) through the Natural Resources website. A list of abbreviations used in the database are in Table 1.

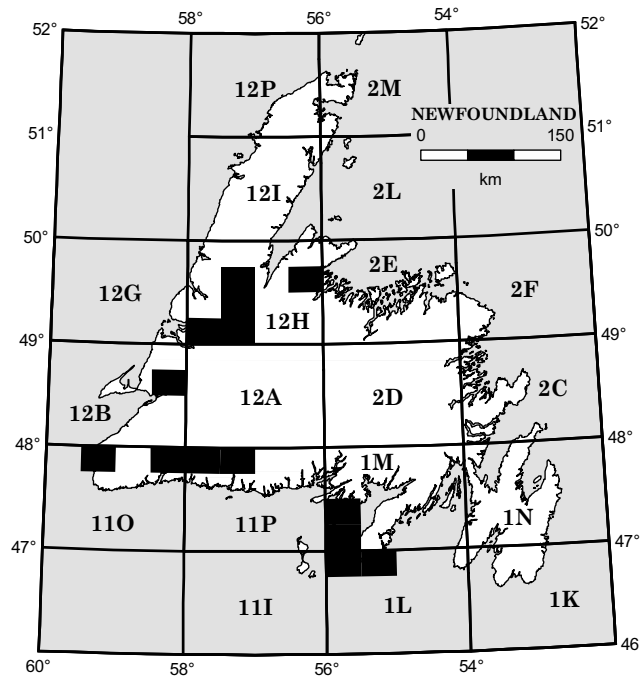


Figure 1. Location map of the study areas in Newfoundland.

This project utilized several different analytical techniques and commercial laboratories in addition to the Geological Survey of Newfoundland and Labrador's (GSNL) Geochemistry Laboratory in St. John's. Select samples were also submitted for external analyses at Actlabs and/or Becquerel Laboratories (now Bureau Veritas Laboratories), located in Ontario. The following represents a brief summary of select analytical procedures utilized in determining the elements included in this release. A summary of the elements included in this release and the method by which they were determined are in Table 2. Standard and duplicate analyses for the various analytical methods are also included; these are provided to enable the reader to access the accuracy and precision of the data provided.

Table 1. List of abbreviated terms used in this release

Abbreviation	Explanation
-99	Sample was not analyzed for that element
4E-Expl.	Actlabs analytical package utilizing INAA and total digestion, lithium metaborate/tetraborate fusion ICP techniques
4 Litho	Actlabs analytical package utilizing a combination of lithium metaborate/tetraborate fusion ICP and trace element ICP-MS techniques
Analysis_Yr	Year sample was analyzed
Au+33	Becquerel Laboratories Ltd. analytical package utilizing INAA
Au+34	Actlabs analytical package utilizing INAA; 1D method
Au+48	Actlabs analytical package utilizing INAA and total digestion ICP techniques; 1H method
Bec.	Becquerel Laboratories Ltd.
cps	Counts per second; measures with handheld, RS-120 scintillometer
DDH	Diamond-drill hole
FUS-ICP	Actlabs ICP Optical Emission Spectrometry; utilizes total digestion, lithium metaborate/tetraborate fusion technique
FUS-MS	Actlabs ICP Mass Spectrometry; utilizes total digestion, lithium metaborate/tetraborate fusion technique
Grav.	Gravimetric
GSNL	Geological Survey of Newfoundland and Labrador
ICP-MS	GSNL ICP Mass Spectrometry; utilizes total digestion, lithium metaborate/tetraborate fusion technique
ICP-OES 4 Acid	GSNL ICP Optical Emission Spectrometry; utilizes multi-acid digestion
ICP-OES-FUS	GSNL ICP Optical Emission Spectrometry; lithium metaborate/tetraborate fusion technique
ICP-OES-HNO ₃	GSNL ICP Optical Emission Spectrometry; utilizes nitric acid digestion for Ag analysis
INAA	Actlabs or Bec. Instrumental Neutron Activation Analysis
ISE	GSNL Ion-selective electrode for F analysis
LOI	Loss-on-ignition
N/A	Not available or not applicable
ppb	Parts per billion
ppm	Parts per million
TD	Total Dissolution
TD-ICP	Actlabs ICP Optical Emission Spectrometry; utilizes multi-acid digestion
TD-MS	Actlabs ICP Mass Spectrometry; utilizing multi-acid digestions, Ultratrace 4 method
Ultratrace 4	Actlabs analytical package utilizing multi-acid digestion and ICP-MS analysis (TD-MS)
wt. %	Weight percent

Table 2. List of elements contained within the database and the corresponding determining analytical method

Element	Analytical Method	Laboratory
SiO ₂ , Al ₂ O ₃ , Fe ₂ O ₃ total, MgO, CaO, Na ₂ O, K ₂ O, TiO ₂ , MnO, P ₂ O ₅ , Ba, Cr, Zr	ICP-OES-FUS	GSNL
As, Ba, Be, Cd, Ce, Co, Cr, Cu, Dy, Fe, La, Li, Mn, Mo, Nb, Ni, P, Pb, Rb, Sc, Sr, Ti, V, Y, Zn	ICP-OES 4 acid	GSNL
SiO ₂ , Al ₂ O ₃ , Fe ₂ O ₃ total, MgO, MnO, CaO, Na ₂ O, K ₂ O, TiO ₂ , P ₂ O ₅ , Ba, Be, Sc, Sr, V, Y, Zr	FUS-ICP; 4 Litho	Actlabs
Ag, As, Bi, Ce, Co, Cr, Cs, Cu, Dy, Er, Eu, Ga, Gd, Ge, Hf, Ho, In, La, Lu, Mo, Nb, Nd, Ni, Pb, Pr, Rb, Sb, Sm, Sn, Ta, Tb, Th, Tl, Tm, U, W, Yb, Zn	FUS-MS; 4 Litho	Actlabs
SiO ₂ , Al ₂ O ₃ , Fe ₂ O ₃ total, MnO, MgO, CaO, Na ₂ O, K ₂ O, TiO ₂ , P ₂ O ₅ , Ba, Be, Sr, V, Y, Zr	FUS-ICP; 4E-Expl.	Actlabs
Ag, Bi, Cd, Cu, Mo, Ni, Pb, S, Zn	TD-ICP; 4E-Expl.	Actlabs
Ag, As, Au, Br, Ce, Co, Cr, Cs, Eu, Hf, Hg, Ir, La, Lu, Nd, Rb, Sb, Sc, Se, Sm, Ta, Tb, Th, U, W, Yb	INAA; 4E-Expl.	Actlabs
Ag, As, Au, Ba, Br, Ca, Ce, Co, Cr, Cs, Eu, Fe, Hf, Hg, Ir, La, Lu, Mo, Na, Nd, Ni, Rb, Sb, Sc, Se, Sm, Sn, Sr, Ta, Tb, Th, U, W, Yb, Zn	INAA; Au+34	Actlabs
Ag, Al, Be, Bi, Ca, Cd, Cu, K, Mg, Mn, Mo, Ni, P, Pb, S, Sr, Ti, V, Y, Zn	TD-ICP; Au+48	Actlabs
Ag, As, Au, Ba, Br, Ce, Co, Cr, Cs, Eu, Fe, Hf, Hg, Ir, La, Lu, Na, Nd, Ni, Rb, Sb, Sc, Se, Sm, Sn, Ta, Tb, Th, U, W, Yb, Zn	INAA; Au+48	Actlabs
Ag, Al, As, Ba, Be, Bi, Ca, Cd, Ce, Co, Cr, Cs, Cu, Dy, Er, Eu, Fe, Ga, Gd, Ge, Hf, Ho, In, K, La, Li, Lu, Mg, Mn, Mo, Na, Nb, Nd, Ni, Pb, Pr, Rb, Re, Sb, Se, Sm, Sn, Sr, Ta, Tb, Te, Th, Tl, Tm, U, V, W, Y, Yb, Zn, Zr	TD-MS; Ultratrace 4	Actlabs
Ag, As, Au, Ba, Br, Cd, Ce, Co, Cr, Cs, Eu, Fe, Hf, Ir, La, Lu, Mo, Na, Ni, Rb, Sb, Sc, Se, Sm, Sn, Ta, Tb, Te, Th, U, W, Yb, Zn, Zr	INAA; Au+33	Bec.
Ag	ICP-OES-HNO ₃	GSNL
F	ISE	GSNL
FeO	Titration	GSNL
Fe ₂ O ₃	Calculation	GSNL
LOI	Gravimetric	GSNL/Actlabs

Major elements plus select trace elements were analyzed by inductively coupled plasma optical emission spectrometry (ICP-OES) following lithium tetraborate and metaborate fusion (Appendix B). Loss-on-ignition (LOI) and ferrous iron (FeO) values are also included with the major elements and are determined using gravimetric and titration methods, respectively (*cf.* Finch *et al.*, 2018). Select trace elements are also provided by ICP-OES following a four acid (HF-HCl-HNO₃-HClO₄) total digestion (Appendix C). Fluoride values are provided by ion-selective electrode analysis following alkaline fusion (Appendix D). Silver analyses are done through ICP-OES following a nitric acid digestion (Appendix E). External analyses conducted at Actlabs were submitted for several different analytical packages which include: 4 Litho (Appendix F), 4E-Exploration (Appendix G), 1D (Au+34; Appendix H), 1H (Au+48; Appendix I) and Ultratrace 4 (Appendix J). External Instrumental Neutron Activation analyses (INAA) conducted at Becquerel Laboratories are in Appendix K.

Note that the negative value, -99, reported for a given sample, indicates a particular element was not analyzed. Negative low numerical values indicate the concentration of the specific element in the sample is below the lower detection limit of the analytical technique (*e.g.*, -0.01), whereas high negative numerical values indicate the concentration of the specific element in the sample was above the upper detection limit (*e.g.*, -1000). Some elements are also reported as negative values for certain samples because these samples contained elevated levels of a particular element that may have resulted in some interelement interference. Samples analyzed by the Actlabs 4E-Exploration, 1D (Au+34) and 1H (Au+48) analytical packages, which report the value “-99” for Ba, Ce, La, Lu, Nd, Pb and Sm, contain such interference due to elevated concentrations of uranium. In addition, some elements in the 4 Litho package have modified upper detection limits due to elevated uranium concentrations.

LABORATORY METHODS

For analyses conducted at the GSNL laboratory, a detailed description of procedures is in Finch *et al.* (2018). Major elements are reported in weight percent (wt. %), and trace elements are reported in parts per million (ppm) or parts per billion (ppb). Volatiles are reported as loss on ignition (LOI), and are determined by gravimetric methods.

Details of the analytical procedures for external commercial analyses are provided below and have been extracted from information provided on the respective company websites (Activation Laboratories, <http://www.actlabs.com>; Becquerel Laboratories [now Bureau Veritas Laboratories], <https://www.bvlabs.com>). These various analytical packages are subdivided by the respective codes used by the companies, which are also used in this report to identify the different analytical procedures used for individual samples.

Actlabs 4 Litho – Lithium Metaborate/Tetraborate Fusion – ICP and ICP-MS (Actlabs, 2017)
(<http://www.actlabs.com/page.aspx?page=516&app=226&cat1=549&tp=12&lk=no&menu=64>)

This analytical package represents a combination of packages Code 4B (lithium metaborate/tetraborate fusion ICP whole rock) and Code 4B2 (trace element ICP-MS).

For the lithium metaborate/tetraborate fusion ICP portion of the analysis, samples are prepared and analyzed in batches. Each batch contains a method reagent blank, certified reference material and 17% replicates. Samples are mixed with a flux of lithium metaborate and lithium tetraborate, and fused in an induction furnace. The molten melt is immediately poured into a solution of 5% nitric acid containing an internal standard, and mixed continuously until completely dissolved (~30 minutes). The samples are run for major oxides and selected trace elements (Code 4B) on a combination simultaneous/sequential Thermo Jarrell-Ash ENVIRO II ICP or a Varian Vista 735 ICP. Calibration is performed using 7 prepared USGS and CANMET certified reference materials. One of the 7 standards is used during the analysis for every group of ten samples.

Totals should be between 98.5 and 101%. If results come out lower, samples are scanned for base metals. Low totals may indicate sulphate being present or other elements like Li that are not normally scanned. Samples with low totals are re-fused and re-analyzed.

For the lithium metaborate/tetraborate fusion – ICP-MS portion of the analysis, samples are fused, diluted and analyzed using a Perkin Elmer SCIEX ELAN 6000, 6100 or 9000 ICP-MS. Three blanks and five controls (three before the sample group and two after) are analyzed per group of samples. Duplicates are fused and analyzed every 15 samples. Instrument is re-calibrated every 40 samples.

Actlabs 4E-Exploration – INAA, Total Digestion – ICP, Lithium Metaborate/Tetraborate Fusion – ICP (Actlabs, 2017)

(<http://www.actlabs.com/page.aspx?page=522&app=226&cat1=549&tp=12&lk=no&menu=64>)

The INAA portion is the same as for the 1D analytical package listed below, except a 1 g aliquot is encapsulated. For the ICP portion of the analytical package, a 0.25 g sample aliquot is digested using a mixture of perchloric, nitric, hydrochloric, and hydrofluoric acids at 200°C to fuming and is then diluted with aqua regia. This digestion is partial for magnetite, chromite, barite and other spinel and potentially massive sulphides. Note when Ag values are <100 ppm the package uses ICP, for values >100 ppm it uses INAA, and when Ba values are <2000 ppm the package uses FUS-ICP, for values >2000 ppm it uses INAA.

Major elements are determined by fusion ICP-OES. Samples are prepared and analyzed in batches. Each batch contains a method reagent blank, certified reference material, and 17% replicates. Samples are mixed with a flux of lithium metaborate and lithium tetraborate and fused in an induction furnace. The molten melt is immediately poured into a solution of 5% nitric acid containing an internal standard, and mixed continuously until completely dissolved (~30 minutes). The samples are then run for major oxides and selected trace elements (Code 4B) on a combination simultaneous/sequential Thermo Jarrell-Ash ENVIRO II ICP.

For the ICP analysis, reagent blanks with and without the lithium borate flux are analyzed, as well as the method reagent blank. Interference correction verification standards are analyzed. Calibration is performed using multiple USGS and CANMET certified reference materials. Two of the standards are used during the analysis for every group of ten samples. This standard brack-

ets the group of samples. The sample solution is also spiked with internal standards and is further diluted and introduced into a Perkin Elmer SCIEX ELAN 6000 ICP-MS using a proprietary sample introduction method. Calibration is performed using USGS and CANMET certified reference materials.

Actlabs 1D – INAA (Au+34) (Hoffman, 1992)

(<http://www.actlabs.com/page.aspx?page=496&app=226&cat1=549&tp=12&lk=no&menu=64>)

The sample is encapsulated in a polyethylene vial and irradiated with flux wires, standards and blanks at a thermal neutron flux of $7 \times 10^{12} \text{ n.cm}^{-2} \text{ s}^{-1}$. The samples are measured the next day for the induced Au-198 photopeak at 411.8 KeV. Samples are compared to a calibration developed from multiple international reference materials.

A 30 g aliquot is encapsulated in a polyethylene vial and irradiated along with flux wires at a thermal neutron flux of $7 \times 10^{12} \text{ n.cm}^{-2} \text{ s}^{-1}$. After a 7-day period to allow Na-24 to decay, the samples are counted on a high purity Ge detector having a resolution of better than 1.7 KeV for the 1332 KeV Co-60 photopeak. Using the flux wires, the decay-corrected activities are compared to a calibration developed from multiple certified international reference materials. The standard present is only a check on accuracy and is not used for calibration purposes. From 10–30% of the samples are re-checked by measurement. For values exceeding the upper limits, assays are recommended. One standard is run for every 11 samples. One blank is analyzed per work order. Duplicates are analyzed when samples are provided.

Actlabs 1H – Total Digestion – ICP, INAA (Au+48) (Hoffman, 1992)

(<http://www.actlabs.com/page.aspx?page=506&app=226&cat1=549&tp=12&lk=no&menu=641>)

The INAA procedure is the same as for the 1D analytical package listed above. For the total digestion ICP portion of the package a 0.25 g sample is digested with four acids beginning with hydrofluoric, followed by a mixture of nitric and perchloric acids. This is then heated using precise programmer-controlled heating in several ramping and holding cycles and takes the samples to incipient dryness. After incipient dryness is attained, samples are brought back into solution using aqua regia.

With this digestion, certain phases may be only partially solubilized. These phases include zircon, monazite, sphene, gahnite, chromite, cassiterite, rutile and barite. Ag greater than 100 ppm and Pb greater than 5000 ppm should be assayed because high levels may not be solubilized. Only sulphide (sulphur) will be solubilized.

The samples are then analyzed using an Agilent 735 ICP. Quality control for the digestion is 14% for each batch, 5 method reagent blanks, 10 in-house controls, 10 sample duplicates, and 8 certified reference materials. An additional 13% quality control is performed as part of the instrumental analysis to ensure quality in the areas of instrumental drift.

Actlabs Ultratrace 4 – “Near Total” Digestion – ICP-MS (Actlabs, 2017)

(<http://www.actlabs.com/page.aspx?page=509&app=226&cat1=549&tp=12&lk=no&menu=64>)

A 0.25 g sample is digested using four acids beginning with hydrofluoric, followed by a mixture of nitric and perchloric acids, heated using precise-programmer controlled heating in several ramping and holding cycles that takes the samples to dryness. After dryness is attained, samples are brought back into solution using hydrochloric and nitric acids. This digestion may not be complete if resistate minerals are present; As, Sb and Cr may be partially volatilized. An in-lab standard (traceable to certified reference materials) or certified reference materials are used for quality control.

Digested samples are diluted and analyzed by Perkin Elmer SEIEX ELAN 6000, 6100 or 9000 ICP-MS. One blank is run for every 40 samples. In-house control is run every 20 samples. Digested standards are run every 80 samples. After every 15 samples, a digestion duplicate is analyzed. Instrument is recalibrated every 80 samples.

Becquerel (now Bureau Veritas Laboratories) – INAA (Au+33) (personal communication Becquerel Laboratories, 2017)

The instrumental neutron activation analysis (INAA) involves the transfer of 10–40 grams of sample to tared, plastic, watertight vials. Each vial is uniquely identified with a bar code and a flux monitor affixed to the base. These vials are stacked into one-foot long bundles for irradiation. The bundles contain randomly selected duplicate samples at the base of the bundle and standards inserted at random positions in the bundle.

The bundles are submitted for exposure to a flux of neutrons at the McMaster Nuclear Reactor, which has flux of 8×10^{12} neutrons/cm²/sec. These bundles are inserted into the core of the reactor for twenty minutes. The bundles are rotated during irradiation so that there is no horizontal flux variation. (The vertical flux variation is monitored with the individual flux monitors.) This irradiation causes the elements in the sample to become radioactive and begin to emit radiation in the form of penetrating gamma rays whose energies (or wavelengths) are characteristic of particular elements.

After the standard decay period of six days, the irradiated samples are loaded onto the counting system. The sample is placed close to a gamma-ray spectrometer having a high resolution, coaxial Ge detector. Gamma rays radiate continuously and the interaction of these with the detector lead to discrete voltage pulses proportional in height to the incident gamma-ray energies. Becquerel’s specially developed multichannel analyzer sorts out the voltage pulses from the detector according to their size, and digitally constructs a spectrum of gamma-ray energies versus intensities. The counting time is twenty to thirty minutes per sample. By comparing spectral peak positions and areas with library standards, the elements comprising the samples are qualitatively and quantitatively identified.

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APPENDICES

Appendices A–K are available as digital comma-separated files (.csv) through [this link](#).

Appendix A: Sample Location and Description Data

Appendix B: GSNL Major-element ICP-OES-FUS Data (standard and duplicate samples)

Appendix C: GSNL Trace-element ICP-OES 4-Acid Data (standard and duplicate samples)

Appendix D: GSNL Fluoride (F-) Ion-selective Electrode Data (standard and duplicate samples)

Appendix E: GSNL Silver (Ag) ICP-OES-HNO₃ Data (standard and duplicate samples)

Appendix F: Actlabs 4 Litho; Lithium Metaborate/Tetraborate Fusion – ICP and ICP-MS (standard and duplicate samples)

Appendix G: Actlabs 4E-Exploration; INAA, Total Digestion – ICP and Lithium Metaborate/Tetraborate Fusion – ICP (standard and duplicate samples)

Appendix H: Actlabs 1D; INAA (Au+34; standard and duplicate samples)

Appendix I: Actlabs 1H; Total Digestion – ICP and INAA (Au+48; standard and duplicate samples)

Appendix J: Actlabs Ultratrace 4; “Near Total” Digestion – ICP-MS (standard and duplicate samples)

Appendix K: Becquerel (now Bureau Veritas Laboratories); INAA (Au+33)