

GEOLOGICAL SURVEY OF CANADA OPEN FILE 8010

Regional Lake Sediment Geochemical Data, Nonacho Basin – East Arm of Great Slave Lake Region, Northwest Territories (NTS 75-C, NTS 75-F and NTS 75-K)

M.W. McCurdy, R.J. McNeil, J.A. Percival and R.G. Garrett

2016





GEOLOGICAL SURVEY OF CANADA OPEN FILE 8010

Regional Lake Sediment Geochemical Data, Nonacho Basin – East Arm of Great Slave Lake Region, Northwest Territories (NTS 75-C, NTS 75-F and NTS 75-K)

M.W. McCurdy, R.J. McNeil, J.A. Percival and R.G. Garrett

2016

 $\hbox{@}$ Her Majesty the Queen in Right of Canada, as represented by the Minister of Natural Resources, 2016

doi:10.4095/297797

This publication is available for free download through GEOSCAN (http://geoscan.nrcan.gc.ca/).

Recommended citation

McCurdy, M.W., McNeil, R.J., Percival, J.A., and Garrett, R.G., 2016. Regional lake sediment geochemical data, Nonacho Basin – East Arm of Great Slave Lake Region, Northwest Territories (NTS 75-C, NTS 75-F and NTS 75-K); Geological Survey of Canada, Open File 8010, 1.zip file. doi:10.4095/297797

Publications in this series have not been edited; they are released as submitted by the author.

Table of Contents

Introduction
Description of Surveys and Sample Management
Sample Collection and Preparation
Analytical Procedures (2012)
Aqua Regia/ICP-AES/MS
Instrumental Neutron Activation Analysis (INAA)
Analytical Procedures (1975)
Atomic Absorption Spectroscopy (AAS) and Other Analyses
Data Files
Quality Control for Geochemical Results
Accuracy
Precision
Analysis of Variance (ANOVA)
Contoured Element Maps
Acknowledgements
References
Figure 1 Map showing locations of lake sediment samples collected in the Nonacho Basin – East Arm of
Great Slave Lake area.
Figure 2 Field card used to capture site-specific field observation data in 1975.
Tables
Table 1 Elements determined by ICP-AES/MS reanalysis of archive lake sediment samples
Table 2 Variables determined by INA reanalysis of archive lake sediment samples.
Table 3 Summary of original elements determined and methods used in 1975
Table 4 Worksheets in Appendix 1 (lake sediment geochemical data) with a brief description of the contents of each worksheet.
Table 5 Worksheets in Appendix 2 (quality control) with a brief description of contents.
Table 6 Elements with RSD >20%, suggesting poor analytical repeatability, are shown for each CRM used for the Nonacho-East Arm reanalyses.

Regional Lake Sediment Geochemical Data, Nonacho Basin – East Arm of Great Slave Lake Region, Northwest Territories (NTS 75-C, NTS 75-F and NTS 75-K)

Introduction

New analytical data for 60 elements from the reanalysis of lake sediment samples collected from 2,817 sites in the Nonacho Basin – East Arm of Great Slave Lake area, Northwest Territories, in 1975 are presented in this Geological Survey of Canada (GSC) open file release. Field observations and analytical data originally reported in GSC Open Files 324 (1976), 325 (1976) and 326 (1976) are included with this report. New data for NTS map sheets 75-C and 75-F were released in 2012 (McCurdy et al, 2012).

The area outlined in Figure 1 was sampled in 1975 under a Federal Uranium Reconnaissance Program. The Uranium Reconnaissance Program was designed to provide industry with high quality reconnaissance exploration data and to provide the Federal Government with nationally systematic data for undertaking uranium resource appraisals. Funds for the reanalysis of archive samples were made available under the Geo-Mapping for Energy and Minerals (GEM) Program at Natural Resources Canada (NRCan).

The GEM Program is a 5-year investment by the Government of Canada in geoscience information to better define the potential for new energy and mineral resources in Canada. GEM is delivered at the federal level by the Earth Science Sector (ESS) of NRCan and the Polar Continental Shelf Project (PCSP). The major focus is on large areas of Canada's North where insufficient public geoscience information exists to attract and guide effective private sector investment.

In the final phase of GEM, a project called Geomapping Frontiers was initiated to improve geoscience knowledge in the least understood parts of Canada's north. In the spirit of the Intergovernmental Geoscience Accord, co-planning was conducted with our territorial counterparts to identify areas which are perceived to have high potential based on reconnaissance information and current deposit models. In light of the consultation process, geographic areas were prioritized for investigation from the Minerals perspective, and topics were selected for Energy research.

The South Rae Domain Project Area was part of the Geomapping Frontiers Project. The goal of this multidisciplinary activity was to establish if major metal-bearing faults and rock types of northern Saskatchewan extend into southern Northwest Territories in order to stimulate exploration activity for nickel-copper Platinum Group elements (PGEs), rare earth elements (REEs) and uranium. Reconnaissance bedrock mapping and compilation of existing maps combined with integration of data from newly acquired geophysical and geochemical surveys and remote predictive mapping were applied to provide knowledge of an area almost devoid of previous mineral exploration.

Reanalysis of existing samples provides data for additional elements and takes advantage of lower detection limits for many elements, at approximately 5% of the cost of collecting new samples. Analytical results and field observations from this project form part of a national geochemical database used for resource assessment, mineral exploration, geological mapping, and environmental studies. Sample collection, preparation procedures and analytical methods are strictly specified and carefully monitored to ensure consistent and reliable results regardless of the area, the year of collection or the analytical laboratory undertaking the analyses.

1

¹ Sample collection predates the January 24, 1978 crash of COSMOS 954 (Gummer et al., 1980).

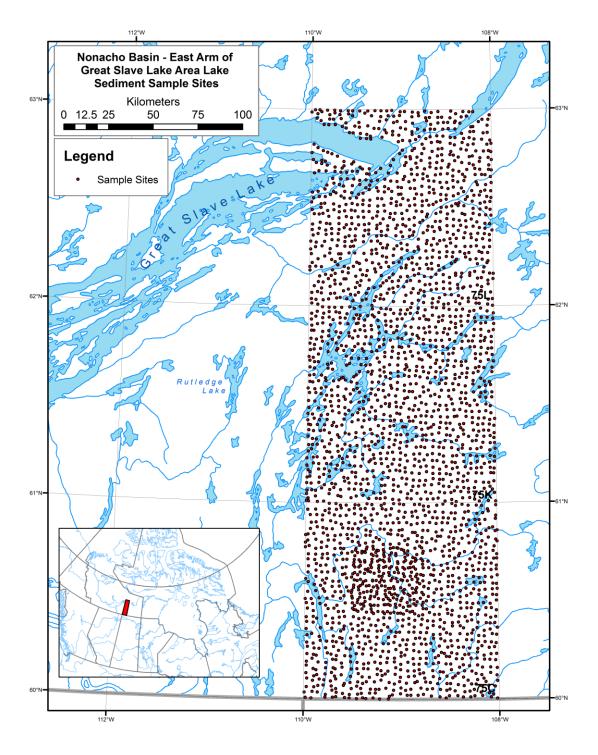


Figure 1 Map showing locations of lake sediment samples collected in the Nonacho Basin – East Arm of Great Slave Lake area.

Description of Surveys and Sample Management

Sample Collection and Preparation

The original lake sediment samples were collected during the summer of 1975. Data for 12 elements in sediments were released in 1976 in three GSC open files, 324 (Geological Survey of Canada, 1976a), 325 Geological Survey of Canada, 1976b) and 326 (Geological Survey of Canada, 1976c). The 2,817 sample sites from which sediments were reanalyzed for this report, shown in Figure 1, are distributed throughout the 35,606 km² area at an average density of one sample per 12.6 km².

A bottom-valved, hollow-pipe sampler was used to collect approximately one kilogram of wet lake sediment. Field observations for each site were recorded on standard forms used by the GSC (Garrett, 1974; see Figure 2). At GSC laboratories in Ottawa, field-dried samples were air-dried, crushed, ballmilled in ceramic mills and sieved through an 80 mesh (177 µm) screen. Typically, one kilogram of gyttia, the preferred collection material, yielded about 50 g of material for analysis. Control reference and blind duplicate samples were inserted into each block of twenty sediment samples. For quality control purposes, the original samples were arranged in groups (consecutively-numbered blocks) of twenty. Each group of twenty contained site duplicate samples; that is, two samples from a single site. The group also contained an analytical duplicate sample pair (a single site sample split and assigned two non-consecutive sample numbers). Finally, each set included a control reference sample. Before publication, thorough inspections of the field and analytical data were made to check for any missing information and/or analytical errors. A more detailed description of collection and quality control methods used by the GSC for lake sediment samples can be found in Cook and McConnell (2001). Samples selected for reanalysis were retrieved from the GSC archive facility in Ottawa and shipped to commercial laboratories for reanalysis. Within these reanalysis suites, the above-described pattern of distribution of quality control samples was maintained, with the exception that new control reference standards replaced the original ones inserted in 1975.

P		ECT		5	1 6	7	0	9	A	REA 11	12	12	14	15	116	112	P 18	HOT	0:	121	22	122	24		OLL		OR:	20	30	21	32	22		ATE 35		3 37	120	3 39	2 1
	N 7	5	S H	EE	T I		N	UM	STREET, STREET	10056568		3.900	NE	Black	U T	HISTORY	ONESDICATE	AS			U T				RT		28	RC	OCK (PE	31	L	AKE	AR		D	EP	ple de-core	100	10
	RELI	EF HIH			SITI		CON	TAM	INAT	ION	TAN	YEL		OUR	BRN		K HV				Y	À	a de	N.		14	15				and a		s	AM	G P L	E A	NO		1000
41	42	43	44	45	46	47	48	49	50	51	52	53	54	100	1000		7 58		60	61	62	63	64	65	66	67	68	69	17373	71	72	73	74	-	76	3 77	78		_
(GEC	CHI	EMI	CA	L L	AKI	E SE	EDII	MEI	TV:	SAI	MPL							S.E.		10	31		70			9149					3330		6200)F (387.	
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	7 18	19	20	21	22	23	24	25	26	27	28	29	30	31	PONE			35 GT5	1000	37	38	3	9
LOV	W ME	нін	SANG	FINES	ORG	GEL	WORK	CAMP	FUEL	GOSN	TAN	YEL	GRN	GRY	BRN	BL	K HV	LGH	,	-	1	P.		75		100					13								
4	1 42	43	44	45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60	61	62	63	64	65	66	67	68	69	70	71	72	73	74	75	76	77	78	79	9
F	REMA	RKS	:																																				
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	7 18	19	20	21	22	23	24	25	26	27	28	29	30	31	32 PONE	100	1000	35 6T5	10000	37	38	3 39	9
LOY	W ME	нін	SAND	FINES	ORG	GEL	WORK	CAMP	FUEL	GOSN	TAN	YEL	GRN	GRY	BRN	BLE	KHV	LGH	1	.5		18	W.	8		1	3	1		19		173							
4	1 42	43	44	45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60	61	62	63	64	65	66	67	68	69	70	71	72	73	74	75	76	77	78	3 79	9
F	REM	ARKS	3:																																				
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	3 3	9
		1			7															8	13	M		3		100					PONE	14.1	1 - 5	GT5				1	
LO	W ME	нін	SANE	FINES	ORG	GEL	WORK	CAMP	FUEL	GOSN	TAN	YEL	GRN	GRY	BRN	BLE	HV	LGH	T	18		18	100	100	3	100	378		100	1		1							I
4	4:	43	44	45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60	61	62	63	64	65	66	67	68	69	70	71	72	73	74	75	7	5 77	78	3 7	9
F	REM	ARKS	3:																																				
1	1 2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	7 18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	3 37	38	3 3	9
100				A	0																			18	1.8						PONG	% - 1	1 - 5	GT5					
LO	W ME	нін	SANG	FINES	ORG	GEL	WORK	CAMP	FUEL	GOSN	TAN	YEL	GRN	GRY	BRN	BLI	K HV	LGH	т		16	13	1	78		1	13		3	100	1	3			To the second	1			
4	1 4:	43	44	45	46	47	48	49	50	51	52	53	54	55	56	57	7 58	59	60	61	62	63	64	65	66	67	68	69	70	71	72	73	74	75	76	77	71	3 7	9

Figure 2 Field card used to capture site-specific field observation data in 1975.

Analytical Procedures (2012)

Aqua Regia/ICP-AES/MS

For the determination of 53 elements listed in Table 1, a one-gram sample was leached with a modified *aqua regia* solution consisting of 6 ml of a mixture of concentrated ACS grade HCl and HNO₃, and demineralized water (1:1:1 volume to volume ratio) at 95° C for one hour. The sample solution was diluted to 20 ml and analysed by inductively coupled plasma - atomic emission spectroscopy (ICP-AES) on a Jarell-Ash instrument or inductively coupled plasma mass spectroscopy on a Perkin-Elmer Elan instrument. Analyses were carried out at Acme Analytical Laboratories, Limited, Vancouver, British Columbia.

Element	Detection Limit	Units of Measurement	Analytical Method	Element	Detection Limit	Units of Measurement	Analytical Method
Ag	2	ppb ¹	ICP-MS	Na	0.001	pct	ICP-MS
Al	0.01	pct ²	ICP-MS	Nb	0.02	ppm	ICP-MS
As	0.1	ppm ³	ICP-MS	Ni	0.1	ppm	ICP-MS
Au	0.2	ppb	ICP-MS	P	0.001	pct	ICP-MS
В	20	ppm	ICP-MS	Pb	0.01	ppm	ICP-MS
Ba	0.5	ppm	ICP-MS	Pd	10	ppb	ICP-MS
Be	0.1	ppm	ICP-MS	Pt	2	ppb	ICP-MS
Bi	0.02	ppm	ICP-MS	Rb	0.1	ppm	ICP-MS
Ca	0.01	pct	ICP-AES	Re	1	ppb	ICP-MS
Cd	0.01	ppm	ICP-MS	S	0.01	pct	ICP-MS
Ce	0.1	ppm	ICP-MS	Sb	0.02	ppm	ICP-MS
Co	0.1	ppm	ICP-MS	Sc	0.1	ppm	ICP-MS
Cr	0.5	ppm	ICP-MS	Se	0.1	ppm	ICP-MS
Cs	0.02	ppm	ICP-MS	Sn	0.1	ppm	ICP-MS
Cu	0.01	ppm	ICP-MS	Sr	0.5	ppm	ICP-MS
Fe	0.01	pct	ICP-AES	Ta	0.05	ppm	ICP-MS
Ga	0.2	ppm	ICP-MS	Te	0.02	ppm	ICP-MS
Ge	0.1	ppm	ICP-MS	Th	0.1	ppm	ICP-MS
Hf	0.02	ppm	ICP-MS	Ti	0.001	pct	ICP-MS
Hg	5	ppb	ICP-MS	Tl	0.02	ppm	ICP-MS
In	0.02	ppm	ICP-MS	U	0.1	ppm	ICP-MS
K	0.01	pct	ICP-AES	V	2	ppm	ICP-MS
La	0.5	ppm	ICP-MS	W	0.1	ppm	ICP-MS
Li	0.1	ppm	ICP-MS	Y	0.01	ppm	ICP-MS
Mg	0.01	pct	ICP-AES	Zn	0.1	ppm	ICP-MS
Mn	1	ppm	ICP-AES	Zr	0.1	ppm	ICP-MS
Mo	0.01	ppm	ICP-MS				

parts per billion, μg/kg

Table 1 Elements determined by ICP-AES/MS reanalysis of archive lake sediment samples

² percent

parts per million, mg/kg

Instrumental Neutron Activation Analysis (INAA)

Weighed and encapsulated samples were packaged for irradiation along with internal standards and international reference materials. Samples and standards were irradiated together with neutron flux monitors in a two-megawatt pool-type reactor. After a seven day decay period, samples were measured for the elements shown in Table 2 on a high resolution germanium detector. Typical counting times were 500 seconds. The sample weights are reported in **Appendix 1 GSC OF 8010 DATA.xlsx**. Analyses were carried out at Becquerel Labs (now Maxxam Analytics), Mississauga, Ontario.

Variable	Detection Limit	Units of Measurement	Variable	Detection Limit	Units of Measurement
As	0.5	ppm ¹	Ni	10	ppm
Au	2	ppb ²	Rb	5	ppm
Ba	40	ppm	Sb	0.1	ppm
Br	0.5	ppm	Sc	0.2	ppm
Cd	5	ppm	Sm	0.1	ppm
Ce	5	ppm	Sn	100	ppm
Co	5	ppm	Ta	0.5	ppm
Cr	20	ppm	Tb	0.5	ppm
Cs	0.5	ppm	Te	10	ppm
Eu	1	ppm	Th	0.2	ppm
Fe	0.2	pct ³	Ti	500	ppm
Hf	1	ppm	U	0.2	ppm
Ir	50	ppb	W	1	ppm
La	2	ppm	Wt	0.1	g^4
Lu	0.2	ppm	Yb	2	ppm
Mo	1	ppm	Zn	100	ppm
Na	0.02	pct	Zr	200	ppm

- parts per million, mg/kg
- ² parts per billion, μg/kg
- 3 percent
- 4 grams

Table 2 Variables determined by INA reanalysis of archive lake sediment samples

Analytical Procedures (1975)

Atomic Absorption Spectroscopy (AAS) and Other Analyses

For the determination of Zn, Cu, Pb, Ni, Co, Ag, Mn, Fe and Cd, a 1 g sample was reacted with 3 ml concentrated HNO_3 in a test tube overnight at room temperature. After digestion, the test tube was immersed in a hot water bath at room temperature and heated to 90° C and held at this temperature for 30 minutes with periodic shaking. One ml of concentrated HCl was added and heating continued for another 90 minutes. The sample solution was then diluted to 20 ml with metal-free water and mixed. Zn, Cu, Pb, Ni, Co, Ag, Mn, Fe and Cd were determined by AAS using an air-acetylene flame. Background corrections were made for Pb, Ni, Co, Ag and Cd.

Molybdenum was determined by AAS using a nitrous oxide acetylene flame. A 0.5 g sample was reacted with 1.5 ml concentrated HNO $_3$ in a test tube overnight at room temperature. After digestion, the test tube was immersed in a hot water bath at room temperature and brought up to 90° C and held at this temperature for 30 minutes with periodic shaking. At this point, 0.5 ml concentrated HCl was added and the digestion

continued at 90° C for an additional 90 minutes. After cooling, 8 ml of 1250 ppm Al solution was added and the sample solution diluted to 10 ml before aspiration.

Mercury was determined by the Hatch and Ott procedure with some modifications. The method is described by Jonasson et al. (1973). A 0.5 g sample was reacted with 20 ml concentrated HNO $_3$ and 1 ml concentrated HCl in a test tube for 10 minutes at room temperature prior to two hours of digestion with mixing at 90° C in a hot water bath. After digestion, the sample solutions were cooled and diluted to 100 ml with metal-free water. The Hg present was reduced to the elemental state by the addition of 10 ml 10% weight per volume (w/v) SnSO $_4$ in 1 M H $_2$ SO $_4$. The Hg vapour was then flushed by a stream of air into an absorption cell mounted in the light path of an atomic absorption spectrophotometer. Absorption measurements were made at 253.7 nm.

Loss-on-ignition was determined using a 500 mg sample. The sample, weighed into a 30 ml beaker, was placed in a cold muffle furnace and brought up to 500° C over a period of two to three hours. The sample was held at this temperature for four hours, before cooling to room temperature for weighing. Loss-on-ignition was determined gravimetrically as the percentage difference in weights.

Arsenic was determined by a colorimetric method using silver diethyldithiocarbamate. Sample material was digested by heating a 1 g sample with 20 ml of 6M HCl at 90°C for 1.5 hours. Arsenic in the reaction solution was converted to arsine gas, which was evolved and then complexed with silver diethyldithiocarbamate. The intensity of the colour of the complex was determined with a spectrophotometer. Colorimetric measurements were made at 520 nm.

Analyses for LOI and the 12 elements described above and listed in Table 3 were carried out at Chemex Laboratories, Vancouver, British Columbia.

Uranium was determined using a neutron activation method with delayed neutron counting. A detailed description of this method is provided by Boulanger et al. (1975). In brief, a 1 g sample was weighed into a 7-dram polyethylene vial, capped and sealed. The samples were pneumatically transferred from an automatic loader to a 'Slowpoke' reactor, where each sample was irradiated for 60 seconds in an operating flux of 1012 neutrons.cm⁻².sec⁻¹. After irradiation, the samples were transferred to a counting facility where, after a ten second delay, each sample was counted for 60 seconds with six BF₃ detector tubes embedded in paraffin wax. Following counting, samples were ejected into a shielded storage container. Analysis of uranium in lake sediments was carried out at Atomic Energy of Canada, Limited (AECL), Ottawa, Ontario.

Element	Detection Limit	Units of Measurement	Analytical Method
Ag	0.2	ppm	AAS ¹
As	1	ppm	COL^2
Co	2	ppm	AAS
Cu	2	ppm	AAS
Fe	0.02	pct	AAS
Hg	10	ppb	CV-AAS ³
LOI	1.0	pct	$GRAV^4$
Mn	5	ppm	AAS
Mo	2	ppm	AAS
Ni	2	ppm	AAS
Pb	2	ppm	AAS
U	0.5	ppm	NADNC ⁵
Zn	2	ppm	AAS

Atomic Absorption Spectrophotometry Colorimetric

6

- Cold vapour Atomic Absorption Spectrometry
- 4 Gravimetric methods
- Neutron activation delayed neutron counting

Table 3 Summary of original elements determined and methods used in 1975.

Data Files

Analytical results are presented in an Excel® workbook included with this report: **Appendix 1 GSC OF 8010 FIELD OBSERVATIONS & ANALYTICAL DATA.xlsx**. The two worksheets in this file are described below in Table 4.

Worksheet	Contents
Table A1-1: Field Data	Site-specific field observations including geographic coordinates
Table A1-2: Original 1975 Data	AAS and specific methods analytical data for silt samples
Table A1-3: Reanalysis ICP Data	ICP-MS/ES analytical data for silt samples
Table A1-4: Reanalysis INAA Data	INAA analytical data for silt samples

Table 4 Worksheets in Appendix 1 (lake sediment geochemical data) with a brief description of the contents of each worksheet.

Quality Control for Geochemical Results

Tables A2-1 through A2-4 in **Appendix 2 GSC OF 8010 QUALITY CONTROL.xlsx** (Table 5) can be used to estimate the quality of analysis for elements listed in Tables 1 and 2 above. Reliability (accuracy and precision) of analytical data returned from commercial laboratories was determined by incorporating field duplicates (FD pairs) within the sampling protocol, and including analytical ('blind') duplicates (AD), and control reference materials (CRMs) in the sample suite submitted to the labs. Analytical data for CRMs, analytical and field duplicates are included with this report in Tables A2-5, A2-6 and A2-7. Elements are grouped based on their position in the Periodic Table.

Worksheet	Contents
Table A2-1: Accuracy (LKSD-1)	Compares accepted values for certified reference material (CRM) LKSD-1 with results from analysis of CRM LKSD-1 included with Nonacho Basin – East Arm area samples
Table A2-2: Accuracy (LKSD-4)	Compares accepted values for certified reference material (CRM) LKSD-4 with results from analysis of CRM LKSD-4 included with Nonacho Basin – East Arm area samples
Table A2-3: Precision	Provides an estimate of precision using analytical duplicate pairs
Table A2-4: ANOVA	Simple pair ANOVA estimates derived from field duplicate pairs of the proportion of total variability due to each of sampling and analysis
Table A2-5: CRM Data	Certified reference material data used to estimate accuracy
Table A2-6: Analytical Duplicate Data	Analytical duplicate data used to estimate precision
Table A2-7: Field Duplicate Data	Field duplicate data used for ANOVA estimates

 Table 5 Worksheets in Appendix 2 (quality control) with a brief description of contents.

Accuracy

Accuracy of analytical data was evaluated by inserting Canadian Certified Reference Lake Sediments LKSD-1 and LKSD-4 at random locations throughout the analytical suite. These two standards were incorporated into Nonacho Basin – East Arm of Great Slave Lake area samples. LKSD-1 is a combination of lake sediments from two lakes located in central Ontario (Brady Lake, NTS 31-M and Joe Lake, NTS 31-F). Sediment from three lakes, Big Gull Lake (NTS 31-C) in Ontario and Key Lake and Seahorse Lake (NTS 74-H) in Saskatchewan, were combined to make up LKSD-4 (Lynch, 1990).

In Appendix 2 (Quality Control), Worksheet 'Accuracy', the means and standard deviations (MEAN \pm SD) for control reference standards LKSD-1 and LKSD-4 for which provisional values have been published by Lynch (1990, 1999), Hechler (2013) and Burnham and Schweyer (2004) are compared with the means and standard deviations for these elements determined by total and partial methods in Nonacho-East Arm area samples (Tables A2-1 and A2-2). Accepted values in square brackets are derived from published and unpublished data (n > 30) collected from recent projects at the GSC. The lower detection limits (LDL) for each element estimated by the commercial laboratories are also listed. A per cent Relative Standard Deviation (RSD %) is calculated for each element with values above detection limits.

Several elements have concentrations below detection in both LKSD-1 and LKSD-4, including Zr (INA), Pd (AR), Ag (INA), Cd (INA), Ta (AR), Ta (INA), Ir (INA), Pt (AR), Sn (INA), B (AR), Ge (AR), Te (INA) and Se (INA) and no statistics are calculated. For several of these elements, the lower detection limits are relatively high. The lower detection limit for Zr (INAA) for example, is 200 ppm.

		L	KSD-1			LKSD-4								
Element (Method)	Unit	LDL	Mean	SD	RSD%	Element	Unit	LDL	Mean	SD	RSD%			
Au (AR)	ppb	0.2	4.3	4.1	95.9	Au (AR)	ppb	0.2	2.9	1.6	57.2			
Au (INA)	ppb	2	4	1	31.4	Be (AR)	ppm	0.1	0.4	0.1	26.1			
Be (AR)	ppm	0.1	0.1	0.1	57.6	Cr (INA)	ppm	20	36	8	23.6			
Cr (INA)	ppm	20	29	7	25.7	Eu (INA)	ppm	1	1	<1	38.9			
Eu (INA)	ppm	1	1	<1	44.9	Hf (AR)	ppm	0.02	0.03	0.01	37.8			
Hf (AR)	ppm	0.02	0.03	0.01	41.2	In (AR)	ppm	0.02	0.05	0.01	21.4			
Lu (INA)	ppm	0.2	0.3	0.1	33.2	Lu (INA)	ppm	0.2	0.4	0.2	60.6			
Mo (INA)	ppm	1	10	3	29.1	Mo (INA)	ppm	1	4	2	47.8			
Ni (INA)	ppm	10	15	4	30.3	Re (AR)	ppb	1	3	2	52.8			
Te (AR)	ppm	0.02	0.02	0.01	62.3	Te (AR)	ppm	0.02	0.14	0.03	25.5			
W (AR)	ppm	0.05	0.60	0.23	37.9	W (INA)	ppm	1	1	<1	24.5			
W (INA)	ppm	1	1	1	58.8	Yb (INA)	ppm	2	2	1	35.3			
Yb (INA)	ppm	2	2	1	29.1									
Zr (AR)	ppm	0.1	1.1	0.3	25.9									

Table 6 Elements with RSD >20%, suggesting poor analytical repeatability, are shown for each CRM used for the Nonacho-East Arm reanalyses.

A relatively high RSD, suggesting poor analytical repeatability, can result when concentrations in a CRM are close to the detection limit for that element (Thompson, 1983). Low detectable concentrations and subsequent relatively high RSD values (>20%) in some CRMs can be caused by elements being present within discrete, often refractory, minerals, including spinels, beryl, tourmalines, chromite, zircon, monazite, niobates, tungstates, topaz, tantalite and cassiterite (Crock and Lamothe, 2011). Such elements may include Cr, Hf, W, Be, Eu, Yb, Lu and Te. For Au (AR) and Au (INAA), RSD % will be relatively high (>20%) due to low concentrations and the difficulty of creating homogeneous standard materials (Harris, 1982). Elements with possible analytical problems, as indicated by a relatively high (>20%) RSD, are shown in bold type in Table A2-1 and A2-2 and listed above in Table 6.

Precision

Precision is considered in terms of the closeness of agreement between analytical duplicate samples analyzed by the same method, i.e. independent test results obtained using the same equipment within short intervals of time on duplicate project samples. The estimation of the analytical precision follows the procedure of Youden (1951) for the up to 168 duplicates where both results were above the respective detection limits. The resulting numerical estimate of precision for variables is listed in Table A2-3 in Appendix 2 (Precision) as a per cent Relative Standard Deviation (the Standard Deviation was divided by the overall mean of the samples and multiplied by 100 to obtain a percentage) (Reimann et al., 2008). Elements (or analytes) are grouped based on their position in the Periodic Table. Included with the element and method of analysis are the Lower Detection Limit (LDL), the percentage of duplicate sample pairs below the Lower Detection Limit (% Below LDL), the number of duplicate pairs removed from the calculations because one or both values are below detection ('Duplicate Pairs Removed') the Range of the remaining sample pairs and the Mean of the data used for each calculation of precision. This information provides context for the estimates of Precision in Table A4.

Elements with precisions poorer than ±10% and with more than 18% (30 or more) of pairs having both values above detection in Table A3, Appendix 2, tend towards concentrations at or just above detection in samples, as indicated by the Range, the Mean and the percentage of data below the detection limit. Such is the case for the elements Zr (AR), Hf (AR), W (AR), Re (AR), Sn (AR), Bi (AR), Be (AR), Eu (INA), Lu (INA), and Sb (AR). Results for Au (AR) and Au (INA) are affected by the difficulty of homogenizing this element and should be considered accordingly. Although a precision was estimated for the elements Zr (INA), W (INA), Pt (AR), Au (INA) In (AR), Yb (INA), Ge (AR), Sb (INA) and Te (AR), less than 30 sample pairs were available for the calculation and the user should consider these statistics less reliable than those using 30 or more sample pairs in the calculation (Walpole, 1982). For the elements Pd (AR), Ag (INA), Cd (INA), Ta (AR), Ir (INA), Sn (INA), B (AR), Te (INA), and Se (INA), 100% of one or both of the duplicate sample pairs were below detection and no precision was estimated. Possible problems with precision may exist with Hg (AR), particularly at concentrations less than approximately 75 ppb, and As (AR) and As (INA).

Analysis of Variance (ANOVA)

Field duplicate data were used to test the hypothesis that the combined sampling and analytical variability, s_{sa}^2 , was equal to the 'regional' variability, s_{r}^2 , across the areal extent of the field duplicates, i.e. H_0 : $s_{sa}^2 = s_{r}^2$, using a one-way Analysis of Variance (ANOVA) (Garrett, 1983). It is desirable that this test fails and the sampling and analytical variability not be equal to the regional variability, but smaller. Otherwise there is as much average variability at the sample sites as there is across the survey area, and if that is the case spatial variation across the survey area cannot be reliably identified. The ANOVA procedure allows the variance components to be estimated, and thus the percentage of the variability in the field duplicate pair data that can be ascribed to sampling and analytical variability and regional variability; ideally the latter percentage should be greater than the former, and statistical significance of the underlying F-test can be used as annotation in an abbreviated table of ANOVA results, that focuses on the key issues.

Using the 'anova2' function found in the 'rgr' package running under the R system, a random effects model Analysis of Variance (ANOVA) estimates the combined sampling and analytical variability between sets of duplicate field samples (Garrett, 2016). Table A2-4 in **Appendix 2 GSC OF 8010 QUALITY**

CONTROL.xlsx shows results from an ANOVA undertaken on up to 164 field duplicate pairs collected for the original surveys. Duplicate pairs of which one or both values of an element are below detection were removed from the calculations. Calculations were only carried out if the number of duplicate pairs with both values above detection exceeds 1. Data were logarithmically transformed (base 10) to meet homogeneity of variance considerations (i.e. severe heteroscedasticity) and to account for ranges of observations exceeding 1.5 orders of magnitude (Garrett, 2016).

The Analysis of Variance (ANOVA) of field duplicates partitions variability into two components, 'Between Sites' and 'Within Sites' in Table A2-4. The variance ratio, F, is calculated in 'anova2' to gauge whether the variance 'within' is significantly smaller than the variation 'between'. As a 'rule of thumb' this ratio should exceed 4.0 for sampling and analytical errors to be significantly smaller at the 95% confidence level. The p-value is a measure of whether the observed F-ratio could have occurred by chance alone. Generally an acceptable p-value is less than 0.05 (>95th percentile), i.e. there is a <5% probability the observed F ratio could have occurred due to chance alone. It should be noted that in cases where an element is evenly distributed throughout all samples, 'F' and 'p-values' may fall below levels of confidence.

The ANOVA indicates that the sampling and analytical variability is significantly lower than the field survey variability, at the p < 0.05 level (>95% confidence level) for all but Zn (INA), Re (AR) and Au (AR) in Table A2-4. From this it is inferred that maps of the distribution for all but the elements listed above will display the true spatial variability of those elements. For the elements Pd (AR), Ag (INA), Cd (INA), Ta (AR), Ir (INA), Pt (AR), Sn (INA), B (AR), Te (INA) and Se (INA), 100% of one or both of the field duplicate sample pairs were below detection and no ANOVA results were reported. Although ANOVA was calculated for the elements Zr (INA), Ta (INA), W (INA), Au (INA) In (AR), Yb (INA), Ge (AR) and Te (AR), less than 30 sample pairs with both values above detection were reported and the user should consider these results less reliable than those using 30 or more sample pairs in the calculation.

Contoured Element Maps

Single-element grid maps (PDF®) are located in **Appendix 3 GSC OF 8010** for the following elements: Ag, As, Au, Cr, Cu, Hg, La, Li, Mo, Ni, Pb, Pt, Re, Sb, Sc, U, Y, Yb and Zn. Summary statistics are included with a related histogram, Empirical Cumulative Distribution Frequency plot, and box plots for all data and each map sheet. The grid maps were generated in Vertical Mapper© within MapInfo® using the following parameters:

Interpolation: Inverse Distance Weighting

Cell Size: 100 m Search Radius: 7.5 km Display Radius: 7.5 km

Exponent: 2 Minimum Points: 3

The summary statistics, histogram, empirical cumulative distribution frequency and box plots were generated using REFLEX ioGAS® data analysis software. A numbered sample location map (scale 1:233,400) is included with the grid maps.

Acknowledgements

E.H. Hornbrook directed the original surveys carried out in 1975, coordinating the activities of contract and Geological Survey of Canada staff. Contracts for sample preparation and analysis were managed by J.J. Lynch, GSC.

Paul Gammon of the GSC reviewed this open file and provided useful comments and suggestions.

References

- Boulanger, A., Evans, D.J.R. and Raby, B.F., 1975. Uranium analysis by neutron activation delayed neutron counting; Proceedings of the 7th Annual Symposium of Canadian Mineral Analysts, Thunder Bay, Ontario, September 22-23, 1975.
- Burnham, O.M. and Schweyer, J., 2004. Trace element analysis of geological samples by inductively coupled plasma mass spectrometry at the Geoscience Laboratories: revised capabilities due to improvements to instrumentation *in* Summary of Field Work and Other Activities 2004; Ontario Geological Survey, Open File Report 6145, p. 54-1 to 54-20.
- Cook, S.J. and McConnell, J.W., 2001. Lake sediment geochemical methods in the Canadian Shield, Cordillera and Appalachia. *in* McClenaghan,, M.B., Bobrowsky, P.T., Hall, G.E.M. & Cook, S.J. (ed.); Drift Exploration in Glaciated Terrain, Geological Society, London, Special Publications, **185**, 125-149.
- Crock, J.G. and Lamothe, P.J. 2011. Inorganic chemical analysis of environmental materials A lecture series: U.S Geological Survey Open-File Report 2011-1193, 117 p.
- Garrett, R.G., 1974. Field data acquisition methods for applied geochemical surveys at the Geological Survey of Canada; Geological Survey of Canada, Paper 74-52.
- Garrett, R.G., 2016. The GSC Applied Geochemistry EDA Package, http://cran.r-project.org/web/packages/rgr/index.html, 21 January 2016.
- Garrett, R.G., 1983. Sampling methodology. Chapter 4 *of* Handbook of Exploration Geochemistry, Vol. 2, Statistics and Data Analysis in Geochemical Prospecting (Ed. R.J. Howarth), Elsevier, pp. 83-110.
- Geological Survey of Canada, 1976a. Regional lake sediment geochemical reconnaissance data, Nonacho Belt, east of Great Slave Lake, N.W.T.; Geological Survey of Canada, Open File 324, 31 p.
- Geological Survey of Canada, 1976b. Regional lake sediment geochemical reconnaissance data, Nonacho Belt, east of Great Slave Lake, N.W.T.; Geological Survey of Canada, Open File 325, 30 p.
- Geological Survey of Canada, 1976c. Regional lake sediment geochemical reconnaissance data, Nonacho Belt, east of Great Slave Lake, N.W.T.; Geological Survey of Canada, Open File 326, 28 p.
- Gummer, W.K., Campbell, F.R., Knight, G.B., and Ricard, J.L., 1980. COSMOS 954, the occurrence and nature of recovered debris; Atomic Energy of Canada Limited, Info 6. (available from http://geoscan.nrcan.gc.ca/starweb/geoscan/servlet.starweb?path=geoscan/fulle.web&search1=R=289316)
- Harris, J.F., 1982. Sampling and analytical requirements for effective use of geochemistry in exploration for gold; In: A.A. Levinson (Ed.), Precious Metals in the Northern Cordillera, proceedings of a symposium sponsored by the Association of Exploration Geochemists and the Cordilleran Section of the Geological Association of Canada, pp. 53-67.
- Hechler, J.H., 2013. QA/QC: Summary of 2012-2013 quality-control data at the Geoscience Laboratories; *in* Summary of Field Work and Other Activities 2013; Ontario Geological Survey, Open File Report 6290, p. 48-1 to 48-6.

- Jonasson, I.R., Lynch, J.J. and Trip, L.J., 1973. Field and laboratory methods used by the Geological Survey of Canada in geochemical surveys; No. 12, Mercury in Ores, Rocks, Soils, Sediments and Water, Geological Survey of Canada, Paper 73-21.
- Lynch, J.J., 1990. Provisional elemental values for eight new geochemical lake sediment and stream sediment reference materials, LKSD-1, LKSD-2, LKSD-3, LKSD-4, STSD-1, STSD-2, STSD-3 and STSD-4; Geostandards Newsletter, v. 14, no. 1, p. 153-167.
- Lynch, J.J., 1999. Additional provisional elemental values for LKSD-1, LKSD-2, LKSD-3, LKSD-4, STSD-1, STSD-2, STSD-3 and STSD-4; Geostandards Newsletter, Vol. 23 (2), p. 251-260.
- McCurdy, M.W., McNeil, R.J., Abramovitch, Y.E. and Day, S.J.A., 2012. Regional lake sediment geochemical data, Nonacho Basin East Arm of Great Slave Lake Region, Northwest Territories (NTS 75-C and 75-F); Geological Survey of Canada, Open File 7232, 12 p.
- Reimann, C., Filzmoser, P., Garrett, R.G., Dutter, R., 2008. Statistical Data Analysis Explained. John Wiley & Sons, Ltd., 343 p.
- Thompson, M., 1983. Control procedures in geochemical analysis. In: Howarth, R.J. (Ed.), Handbook of Exploration Geochemistry, Vol. 2; Statistics and Data Analysis in Geochemical Prospecting. Elsevier, Amsterdam, 437 p.
- Walpole, R.E., 1982. Introduction to Statistics, 3rd Edition. Macmillan Publishing Company, New York, 521 p.
- Youden, W.J., 1951. Statistical methods for chemists. John Wiley & Sons Ltd., New York, 126 p.