Preliminary Release of Geochemical, Mineralogical and Kimberlite Indicator Mineral Electron Microprobe Data from Silts, Heavy Mineral Concentrates and Waters from a National Geochemical Reconnaissance Stream Sediment and Water Survey in the Northern Buffalo Head Hills Area, Northern Alberta (Parts of NTS 84F, 84G and 84K)

Introduction

A regional stream sediment and water geochemical survey was undertaken in the northern part of the northern Buffalo Head Hills in 2003. The 2003 data are part of an ongoing survey of the Buffalo Head Hills area initiated in 2001. This National Geochemical Reconnaissance (NGR) project is aligned with Alberta's plan for a multi-year, multi-disciplinary study in the northern part of the province.

Analytical and statistical data are published for 48 elements in stream sediments, 50 elements in heavy mineral concentrates, and up to 58 variables in waters from 95 sites collected in 2003. The Geological Survey of Canada, under the Targeted Geoscience Initiative II (TGI II) and Northern Resources Development Program, and the Alberta Energy and Utilities Board/Alberta Geological Survey (EUB/AGS), funded the 2003 survey.

Analytical results and field observations contribute to building a national geochemical database 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 (Friske and Hornbrook, 1991).

The northern Buffalo Head Hills survey area lies south and southeast of La Crete, a community located 800 km north of Edmonton (Fig. 1). Access to sample sites was mainly by helicopter. Seismic cut lines and pipeline right-of-ways provide some access for all-terrain vehicles. Services available in La Crete include lodging, gas stations, grocery and hardware stores, and restaurants. There is a small airport with no services.

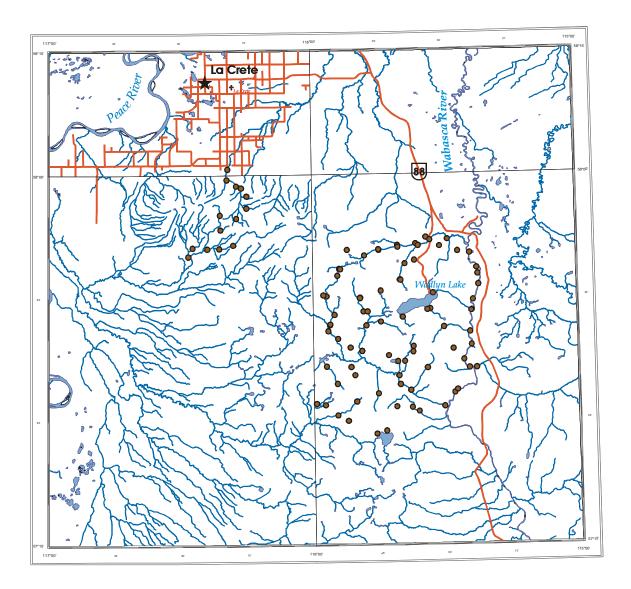


Figure 1. Location map shows area of survey south and southeast of the village of La Crete. Circles indicate the location of sample sites.

Sample Collection

Stream Sediments

At each site a pre-labelled Kraft paper bag (12.5 cm x 28 cm with side gusset) (Fig. 2) was two-thirds filled with silt or fine sand collected from the active stream channel. In practice, the silt sample was collected after water samples were collected but before a bulk sediment sample. Commonly, the sampler collected handfuls of silt from various points in the active stream channel while moving gradually upstream. If the stream channel consists mainly of clay, coarse material or organic sediment from which suitable sample material is scarce or absent, moss mat from the stream channel, which commonly contains trapped silt, may be added to the sample.



Figure 2. Pre-labelled Kraft paper bags and plastic bottles are used to collect samples of stream silts and stream waters. Flagging tape with a sample site number is used to mark sample sites.

Stream Waters

Waters were sampled in mid-channel, from flowing water where possible, at every site. At sites where sediments were taken for heavy mineral concentration (approximately every second site) two water samples were generally collected. Samples were contained in 125-ml Nalgene HDPE bottles (Fig. 2). Samples were collected after first rinsing bottles at least two times in flowing water before a final fill.

Heavy Mineral Concentrates

Ideal sites for the collection of sediments for the heavy mineral concentrate fraction are located at the upstream points of mid-channel bars. Material was collected from a single location where possible, or within close proximity otherwise. A 5-gallon (22.7 litre) plastic pail was lined with a heavy-duty polyethylene bag (18x24 inches, 4 Mil). Material was wet-sieved through a 12-mesh (1.68 mm) stainless steel sieve, placed on top of the pail, until a sample weight of 10-15 kg was attained. The sample was weighed in the pail before the opening of the bag lining the pail was taped shut with black plastic (electrical) tape and placed into a second bag with a sample number and taped. Samples were shipped directly to a commercial lab for preparation and analysis.

Sample Preparation

Stream Sediments (Silts)

The Kraft paper bags containing the silt samples were placed into plastic bags, taped with electrical tape and shipped directly to a commercial lab, where they were air-dried at temperatures below 40°C and sieved through a minus 80-mesh (177 μ m) screen. Control reference and blind duplicate samples were inserted into each block of twenty sediment samples.

Waters

One set of water samples, those collected at every site, remained unaltered and was packed and shipped to a laboratory for analysis. The second set of water samples, those collected at approximately every second site, was filtered within 24 hours of collection through single-use Millipore Sterivex-HV 0.45-µm filter units attached to 50-ml sterile plastic syringes. After 50 ml of water from the second set of samples was filtered into new 60-ml bottles, the remainder was used for the determination of pH and conductivity before being discarded. Using an Eppendorf pipettor with disposable plastic tips, 0.5 ml 8M HNO₃ was added to filtered water samples. Syringes were reused after rinsing with distilled, de-ionized water, but replaced daily. At this point, control reference samples (filter, acid and travel blanks^{*}) were inserted. Filtered and acidified waters were kept in a cool dark place until shipment to the lab. Control reference samples were inserted into each block of 20 water samples. No duplicate water samples were introduced.

Heavy Mineral Concentrates

Bulk sediment samples were progressively reduced by different laboratory procedures to concentrate heavy minerals. Initially a 500-g character sample was taken and stored before a low-grade table concentrate was prepared from the remainder. Gold grains were observed at this stage and counted, measured and classified as to degree of wear (i.e. distance of transport). The table reject was re-tabled to scavenge possible unrecovered kimberlite indicator minerals and magmatic massive sulphide indicator minerals. The concentrate from both tabling runs was separated in methylene iodide diluted with acetone to S.G. 3.20 to recover heavy minerals including Cr-diopside and forsterite olivine. Magnetite was removed after the heavy liquid separation and the remaining concentrate cleaned with oxalic acid to remove limonite stains. The dried concentrate was sieved to separate it into several size fractions, (<0.25, 0.25 to <0.5, 0.5 mm to <1.0 mm, \ge 1.0 mm to 2.0 mm). The <0.25 mm fraction was kept for chemical analysis and the 0.25 to 0.50 mm fraction was sorted with a Carpco® drum magnetic separator into strongly, moderately, weakly and non-paramagnetic fractions.

Analytical Procedures

Stream Sediment (Silt)Analyses

Instrumental Neutron Activation Analysis (INAA)

^{*} Filter (sample) blanks are 60-ml bottles filled with deionized water used in the field that has been filtered and acidified at the same time as routine samples; acid blanks are samples of the deionized water used in the field and acidified (but not filtered) at the same time as routine samples; travel blanks are bottles of deionized water pre-filled at the GSC lab in advance of field sampling and acidified in the field with the survey samples.

Weighed and encapsulated samples, normally 30 g, 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 with a high-resolution germanium detector. Typical counting times were 500 seconds. Elements determined by INAA are listed in Table 1.

ELEMENT		DETEC	DETECTION LEVEL	
As	Arsenic	0.5	ppm	
Au	Gold	2	ppb	
Ва	Barium	50	ppm	
Br	Bromine	0.5	ppm	
Се	Cerium	5	ppm	
Со	Cobalt	5	ppm	
Cr	Chromium	20	ppm	
Cs	Cesium	0.5	ppm	
Eu	Europium	1	ppm	
Fe	Iron	0.2	pct	
Hf	Hafnium	1	ppm	
La	Lanthanum	2	ppm	
Lu	Lutetium	0.2	ppm	
Мо	Molybdenum	1	ppm	
Na	Sodium	0.02	pct	
Ni	Nickel*	10	ppm	
Rb	Rubidium	5	ppm	
Sb	Antimony	0.1	ppm	
Sc	Scandium	0.2	ppm	
Sm	Samarium	0.1	ppm	
Та	Tantalum	0.5	ppm	
Tb	Terbium	0.5	ppm	
Th	Thorium	0.2	ppm	
Ti	Titanium*	500	ppm	
U	Uranium	0.2	ppm	
W	Tungsten	1	ppm	
Yb	Ytterbium	2	ppm	
* HMC	samples only			

Table 1Elements in stream sediments and the <0.25 mm fraction of heavy mineral concentrates
determined by Instrumental Neutron Activation analysis.

Data for silver, cadmium, iridium, selenium, tin, tellurium, zinc, and zirconium are not published because of inadequate detection limits and/or precision.

Inductively Coupled Plasma Mass Spectrometry (ICP-MS) and Other Analyses

For the determination of 36 elements listed in Table 2, a one-gram sample was leached with 6 ml of a mixture of HCl, HNO_3 , and distilled, deionized water (2:2:2 v/v) at 95° C for one hour. The sample solution was diluted with de-ionized water to 20 ml and analysed by inductively coupled plasma emission spectroscopy on a Jarell-Ash instrument and inductively coupled plasma mass spectroscopy on a Perkin-Elmer Elan instrument.

Loss-on-ignition was determined using a one-gram sample. The sample, weighed into a Leco® crucible, was placed into a 100°C muffle furnace and brought up to 500° C for

one hour. The oven was cooled to 100°C and crucibles transferred to a desiccator for cooling to room temperature. The crucibles were re-weighed, and the difference was reported as loss-on-ignition.

Tin in heavy mineral concentrates was prepared for analysis by heating a 0.2 g sample mixed with 1.5 g of LiBO₂ flux in a muffle furnace for 15 minutes at 1050°C in a graphite crucible. The molten mixture was removed and immediately poured into 100 mL of 5% HNO₃. The solution was shaken for two hours and then an aliquot was poured into a test tube. Analytical values were determined using a Perkin-Elmer Elan 6000 ICP-MS (Gravel, 2003).

Table 2	Variables in stream silts and the <0.25 mm fraction of heavy mineral concentrates determined by Inductively Coupled Plasma - Mass Spectrometry and other analytical
	methods

VARIABLE		DETECTION LEVEL		
٨a	Silver	2	nnh	
Ag Al	Aluminum	0.01	ppb pct	
As	Arsenic	0.01	ppm	
Bt	Boron	1	ppm	
Ba	Barium	0.5	ppm	
Be†	Beryllium	0.0	ppm	
Bi	Bismuth	0.02	ppm	
Ca	Calcium	0.01	pct	
Cd	Cadmium	0.01	ppm	
Ce†	Cerium	0.1	ppm	
Co	Cobalt	0.1	ppm	
Cr	Chromium	0.5	ppm	
Cst	Cesium	0.02	ppm	
Cu	Copper	0.01	ppm	
F	Fluorine	10	ppm	
Fe	Iron	0.01	pct	
Ga	Gallium	0.2	, ppm	
Get	Germanium	0.1	ppm	
Hft	Hafnium	0.02	ppm	
Hg	Mercury	5	ppb	
Int	Indium	0.02	ppm	
ĸ	Potassium	0.01	pct	
La	Lanthanum	0.5	ppm	
Li†	Lithium	0.1	ppm	
LOI*	Loss-on-ignition	1	pct	
Mg	Magnesium	0.01	pct	
Mn	Manganese	1	ppm	
Мо	Molybdenum	0.01	ppm	
Na	Sodium	0.001	pct	
Nb†	Niobium	0.02	ppm	
Ni	Nickel	0.1	ppm	
Р	Phosphorus	0.001	pct	
Pb	Lead	0.01	ppm	
Rb†	Rubidium	0.1	ppm	
Re†	Rhenium	1	ppb	
S	Sulphur	0.01	pct	
Sb	Antimony	0.02	ppm	
Sc	Scandium	0.1	ppm	
Se	Selenium	0.1	ppm	
Sn†	Tin (LiBO ₂ Fusion)	1	ppm	
Sn†	Tin	0.1	ppm	
Sr	Strontium	0.5	ppm	
Ta †	Tantalum	0.05	ppm	
Te	Tellurium	0.02	ppm	
Th Ti	Thorium	0.1	ppm	
Ti	Titanium	0.001	pct	

TI	Thallium	0.02	ppm	
U	Uranium	0.1	ppm	
V	Vanadium	2	ppm	
W	Tungsten	0.1	ppm	
Y†	Yttrium	0.01	ppm	
Zn	Zinc	0.1	ppm	
Zr† Zirconium *Silt samples only †HMC samples only		0.1	ppm	

Fluorine was determined using 0.25-gram sample splits weighed into Ni crucibles. One gram of sodium hydroxide was added and the mixture was fused at 600°C in a muffle furnace. The fusion product was dissolved in 7 ml of de-ionized water and 5 ml of 30% sulphuric acid. The solutions were transferred to plastic beakers and 5 ml of 30% ammonium acetate added. The volume was made up to 90 ml with de-ionized water. The pH was tested and adjusted to 7.8 with either sodium hydroxide or sulphuric acid. Fluorine content was determined using a fluorine selective ion electrode.

Water Analyses

The pH of stream waters was determined using an WTW MultiLine® P3 pH/LF-SET with automatic temperature compensation, a range of -2.00 to +16.0 pH, resolution of 0.01 pH and an accuracy of ± 0.01 pH. Meters were calibrated using commercial buffer solutions with pH values of 4.0, 7.0 and 10.0.

Conductivity of stream waters was determined using an WTW MultiLine® P3 pH/LF-SET with automatic temperature compensation and a range of 500 μ S/cm, a resolution of 1 μ S/cm and a full-scale accuracy of ±1%. Meters were calibrated using commercial conductivity standards.

Chloride (Cl⁻), fluoride (F⁻), nitrate (NO₃⁻) and sulphate (SO₄²⁻) in untreated waters were determined by ion chromatography (IC).

Uranium in untreated waters was determined by inductively coupled plasma-mass spectrometry (ICP-MS).

Trace and Major Elements

Acidified and filtered stream water samples were analyzed for trace metal and major elements. A complete list of elements and stated detection limits are given in Table 3.

Trace metal analysis was performed using a VG PQII ICP-MS with a Meinhard concentric glass nebulizer, Type K (solution uptake rate 1 ml min⁻¹), a quartz Scott-type double-pass chilled spray chamber (2°C) and a 27 MHz standard quartz torch. The argon flow-rates are: Cool 12.5 I min⁻¹, Auxiliary 0.85 I min⁻¹, and Nebulizer 0.9 I min⁻¹. The RF power is 1350 watts. Isotopes measured and corrections for spectral interferences are detailed in Hall et al. (1995) and Hall et al. (1996). Data for hafnium and zirconium are not published because these elements are not sufficiently stabilized

in waters by the addition of nitric acid. Data for indium, selenium, silver, tantalum and thulium are not published because of inadequate detection limits and/or precision.

ELEMENT		DETECTIO	ON LEVEL	METHOD	
Waters -	-Filtered, Acidified (Wat-FA)				
AI	Aluminum	2	ppb	ICP-MS	
As	Arsenic	0.1	ppb	ICP-MS	
B	Boron	0.1		ICP-MS	
			ppb		
Ba	Barium	0.2	ppb	ICP-MS	
Be	Beryllium	0.005	ppb	ICP-MS	
Cd	Cadmium	0.02	ppb	ICP-MS	
Ce	Cerium	0.01	ppb	ICP-MS	
Cs	Cesium	0.01	ppb	ICP-MS	
Co	Cobalt	0.05	ppb	ICP-MS	
Cr	Chromium	0.1	ppb	ICP-MS	
Cu	Copper	0.1	ppb	ICP-MS	
Dy	Dysprosium	0.005	ppb	ICP-MS	
Er	Erbium	0.005	ppb	ICP-MS	
Eu	Europium	0.005	ppb	ICP-MS	
Ga	Gallium	0.01	ppb	ICP-MS	
Gd	Gadolinium	0.005	ppb	ICP-MS	
Ge	Germanium	0.02	ppb	ICP-MS	
Ho	Holmium	0.005	ppb	ICP-MS	
La	Lanthanum	0.01	ppb	ICP-MS	
Li	Lithium	0.02	ppb	ICP-MS	
Lu	Lutetium	0.005	ppb	ICP-MS	
Mn	Manganese	0.1	ppb	ICP-MS	
Мо	Molybdenum	0.05	ppb	ICP-MS	
Nb	Niobium	0.00	ppb	ICP-MS	
Nd	Neodymium	0.005	ppb	ICP-MS	
Ni	Nickel	0.003	ppb	ICP-MS	
Pb	Lead	0.2	ppb	ICP-MS	
Pr		0.005		ICP-MS	
Rb	Praseodymium		ppb		
	Rubidium	0.05	ppb	ICP-MS	
Re	Rhenium	0.005	ppb	ICP-MS	
Sb	Antimony	0.01	ppb	ICP-MS	
Sm	Samarium	0.005	ppb	ICP-MS	
Sn	Tin		ppb	ICP-MS	
Sr	Strontium	0.5	ppb	ICP-MS	
Tb	Terbium	0.005	ppb	ICP-MS	
Те	Tellurium	0.02	ppb	ICP-MS	
Ti	Titanium	0.5	ppb	ICP-MS	
TI	Thallium	0.005	ppb	ICP-MS	
U	Uranium	0.005	ppb	ICP-MS	
V	Vanadium	0.1	ppb	ICP-MS	
W	Tungsten	0.02	ppb	ICP-MS	
Y	Yttrium	0.01	ppb	ICP-MS	
Yb	Ytterbium	0.005	ppb	ICP-MS	
Zn	Zinc	0.5	ppb	ICP-MS	
<u>Ca</u>	Coloium	0.02			
Ca	Calcium	0.02	ppm	ICP-ES	
Fe	Iron	0.005	ppm	ICP-ES	
K	Potassium	0.05	ppm	ICP-ES	
Mg	Magnesium	0.005	ppm	ICP-ES	
Na	Sodium	0.05	ppm	ICP-ES	
Р	Phosphorus	0.05	ppm	ICP-ES	
S	Sulphur	0.05	ppm	ICP-ES	
Si	Silicon	0.02	ppm	ICP-ES	
	-Filtered, not Acidified (Wat-Not FA)	100			
Cl	Chloride	100	ppb	IC	
F	Fluoride	50	ppb	IC	
NO ₃	Nitrate	50	ppb	IC	
SO4	Sulphate	50	ppb	IC	
Cond.	Conductivity	1	μS	ISE (ion selective electrode	
pН	-	0.1	-	ISE	

Table 3Major and trace elements determined in stream waters.

Major element analysis was performed using a Perkin-Elmer 3000DV Inductively Coupled Plasma – Emission Spectrometer (ICP-ES) with a cross-flow nebulizer (solution uptake rate 1 ml min⁻¹), a Ryton Scott-type double-pass spray chamber and a custom demountable quartz ICP-ES torch. The argon flow-rates are: Plasma 15.0 I min⁻¹, Auxiliary 0.5 I min⁻¹, and Nebulizer 0.7 I min⁻¹. The RF power is 1350 watts. All elements were analyzed in axial mode except for sodium and potassium. These elements were run in radial mode. Inter-element correction factors were applied as required to correct for various spectral interferences. Data for scandium are not published because of inadequate detection limits and/or precision.

Heavy Mineral Concentrate Analyses

Kimberlite indicator minerals (KIMs) were picked and identified from each of three size fractions (0.25-0.5 mm, 0.5 mm-1.0 mm, 1.0-2.0 mm). Fractions exceeding a 100 g threshold were characterized by a 100 g split and normalized to represent the total sample weight. The 0.25-0.5 mm fraction (minus KIMs) was further characterized by identifying and recording the mineralogy of 100 grains selected at random. The remaining heavy mineral grains >0.5 mm (minus KIMs) were recombined and archived. The <0.25 mm fraction of the heavy mineral concentrate was sent to a commercial lab where it was ground in a ceramic mill and analyzed by a combination of ICP-MS, INAA and specific methods. Kimberlite indicator mineral grains mm underwent electron microprobe analysis for chemical characterization.

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- Helicopter: Highland Helicopter Ltd. (Howard Vigneault pilot) Richmond, British Columbia
- Preparation: Acme Analytical Laboratories Vancouver, British Columbia

Overburden Drilling Management Ottawa, Ontario

Analysis: Acme Analytical Laboratories Vancouver, British Columbia

> Becquerel Laboratories Inc. Mississauga, Ontario

GSC Analytical Chemistry Laboratory Ottawa, Ontario

GSC Analytical Method Development Laboratory Ottawa, Ontario

GSC Mineralogical Laboratory (electron microprobe) Ottawa, Ontario

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References

Friske, P.W.B. and Hornbrook, E.H.W.

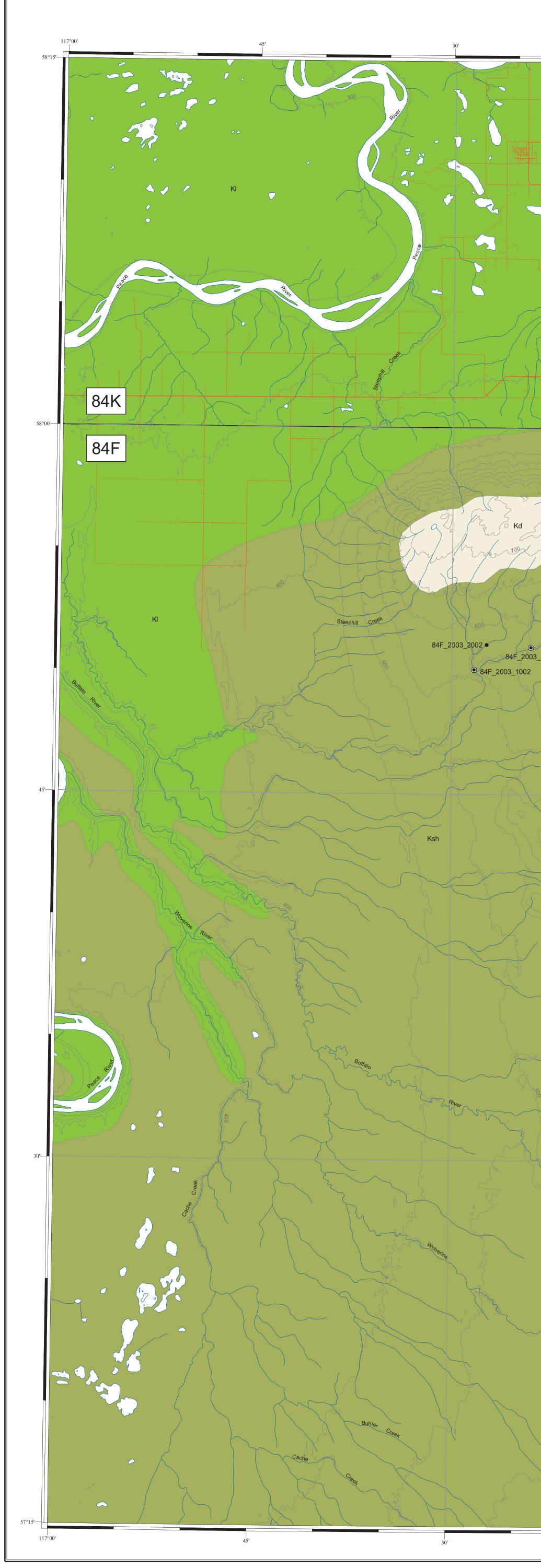
- 1991 Canada's National Geochemical Reconnaissance programme; Transactions of the Institution of Mining and Metallurgy, London, Section B; Volume 100, p. 47-56
- Gravel, J.
 - 2003 Method and specifications for Group 4B; document prepared for Acme Analytical Labs, Vancouver, British Columbia, 1 page.

Hall, G.E.M., Vaive, J.E., and McConnell, J.W.

1995 Development and application of a sensitive and rapid analytical method to determine the rare-earth elements in surface waters; Chemical Geology, Vol. 120, Nos. 1-2, pp. 91-109.

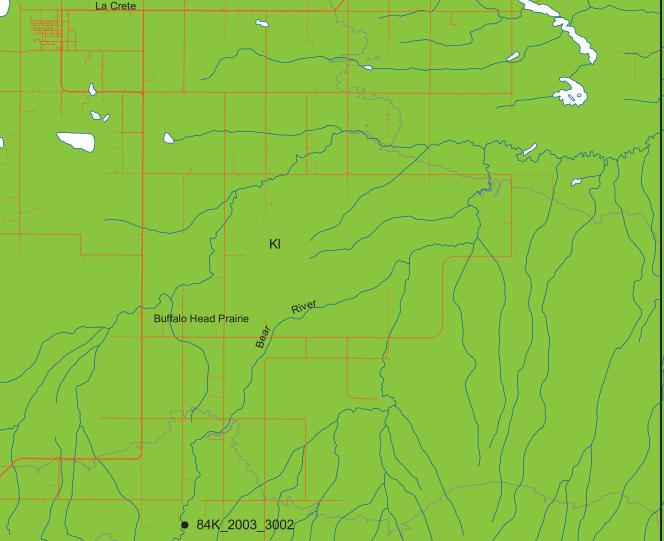
Hall, G.E.M., Vaive, J.E., and Pelchat, J.C.

1996 Performance of inductively coupled plasma – mass spectrometric methods used in the determination of trace elements in surface waters in hydrogeochemical surveys; Journal of Analytical Atomic Spectrometry, Vol. 11, pp. 779-786.



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Sample Locations

Alberta Geological Survey and

AGS Alberta Geological Survey of Canada Buffalo Head Hills, Northern Alberta (Parts of NTS 84F, 84G, 84K, 84J)

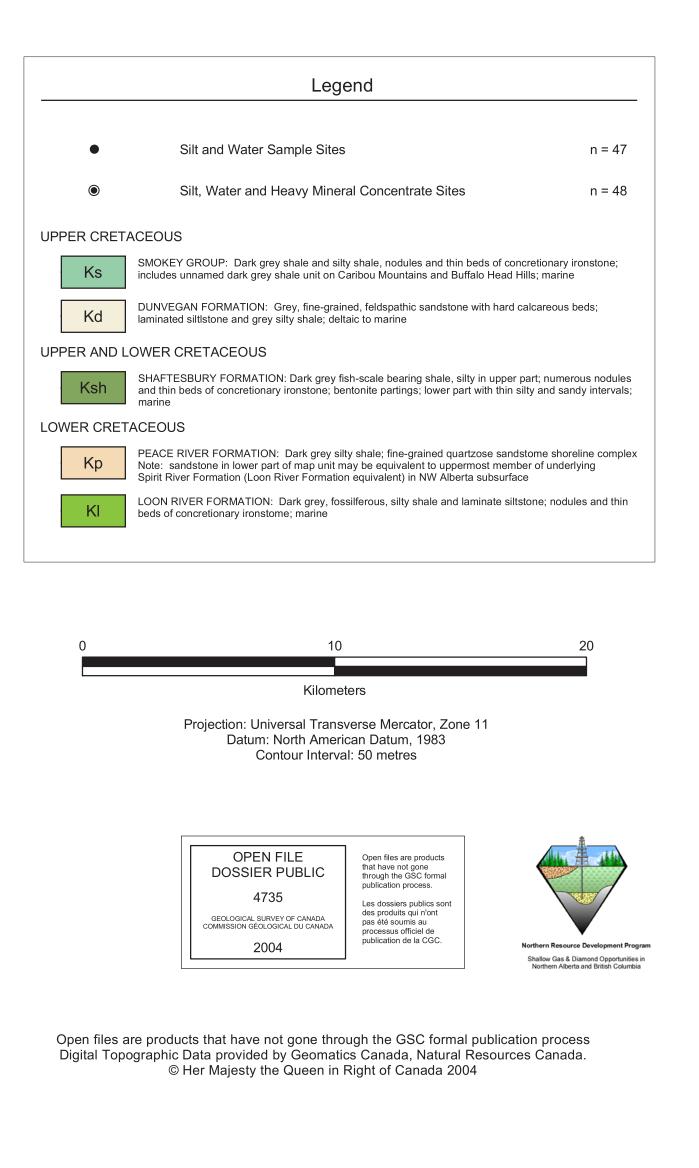
EUB/AGS SPE 71 - GSC Open File 4735

Staff of the Geological Survey of Canada (GSC) and the Alberta Energy and Utilities Board/Alberta Geological Survey (EUB/AGS) carried out a regional stream sediment and water survey in the northern Buffalo Head Hills area of northern Alberta in 2003. The 2003 data are part of an ongoing survey of the Buffalo Head Hills area initiated in 2001. This National Geochemical Reconnaissance project is aligned with Alberta's plan for a multi-year, multi-disciplinary study in the northern part of the province. The GSC, under the Northern Resource Development Program and the Targeted Geoscience Initiative II (2003-2005), and the EUB/AGS funded the 2003 survey.

Stream sediments and waters were collected from 95 sites in the northern Buffalo Head Hills during 2003. Analytical data for up to 48 elements in stream sediments and up to 58 variables in waters, determined by a number of analytical techniques, are available for locations indicated on this map. In addition, at 48 sites, indicated by a circled dot, bulk sediment samples were collected for heavy mineral concentration. Heavy minerals were characterized in the 0.25 to 0.5 mm fraction of the concentrate by a 100-grain identification after kimberlite indicator minerals were picked and identified separately. Kimberlite indicator minerals, picked from the 0.25 to 2.0 mm fraction, underwent analysis by electron microprobe to determine the chemistry of individual grains. Further geochemical analysis was carried out on the <0.25 mm fraction of the heavy mineral concentrate by ICP-MS. Detailed descriptions of collection, preparation and analytical methods are available in the text of the report accompanying this map.

Analytical results and field observations contribute to building a national geochemical database 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 analytical undertaking the analyses.





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