GEOLOGICAL SURVEY OF CANADA

OPEN FILE 5696

Regional Stream Sediment and Water Geochemical Data, Finlayson Lake area, southeastern Yukon (NTS 105G)

P.W.B. Friske, E.H.W. Hornbrook, M.W. McCurdy, S.J.A. Day, R.J. McNeil

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1 CD-ROM

Cover illustration
View of Kudz Ze Kayah area, *courtesy of Alain Plouffe (GSC)*

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Introduction

Geological Survey of Canada (GSC) Open File 5696 contains analytical data for stream silt sediment, stream water and field data -originally published as Open File 1648, as well as Inductively Coupled Plasma Mass Spectrometry (ICP-MS) re-analysis data of stream silt sediment samples. This publication includes site-specific field observation and analytical data for stream sediments and waters from 914 sites. Silt sediments were analysed for 39 elements plus loss-on-ignition and corresponding waters were analysed for uranium, fluoride and pH.

National Topographic System (NTS) mapsheet 105G was sampled in 1987 as part of the Canada-Yukon Economic Development Agreement (1985-1989). The reconnaissance survey was undertaken by the Geological Survey of Canada in conjunction with the Department of Indian Affairs and Northern Development. Funding for the ICP-MS re-analysis was provided by the Yukon Geological Survey and supported by the GSC's Environment and Health Program.

Regional geochemical surveys have been carried out since 1976 in Yukon under the National Geochemical Reconnaissance (NGR) program. One centre-lake sediment and water surveys and 27 stream silt sediment and water surveys (Fig. 1) have been published or are in publication. Approximately 102,100 km^2 of Yukon have been covered at an average density of 1 sample or site per 13 km^2.

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).

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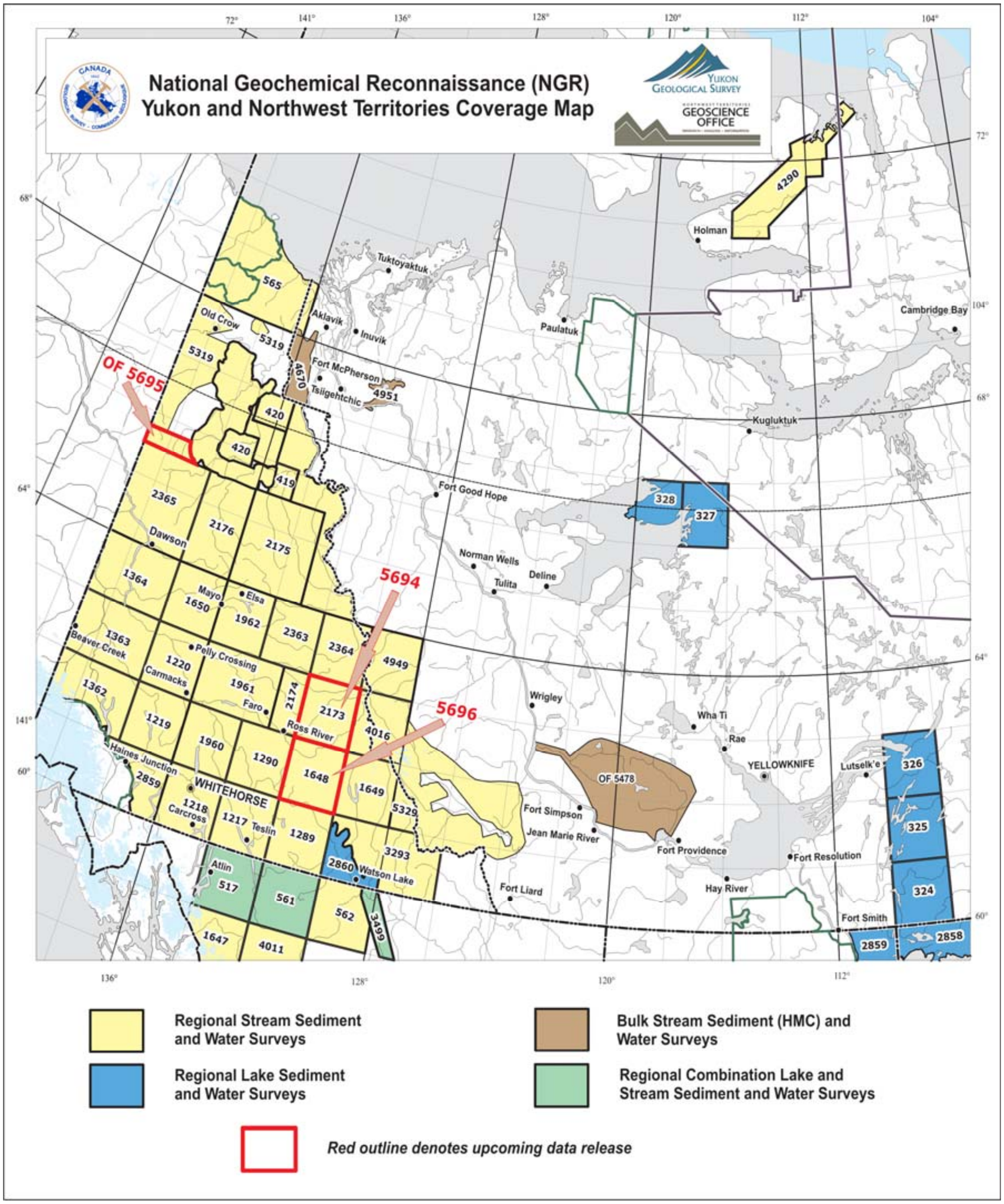

Figure 1: NGR coverage in NWT, Yukon and western Nunavut

Methods

Description of Survey and Sample Management

Helicopter-supported sample collection was carried out during the summer of 1987. Stream sediment and water samples were collected at an average density of one sample per 13 square kilometres throughout the 11,900 square kilometres of the south-central Yukon survey.

Sample site duplicate samples, or field duplicates samples, were routinely collected in each analytical block of twenty samples and were assigned Replicate Status (Rep Stat) values of 10 and 20. Routine samples were assigned a Rep Stat of 0. At the time of collection, one random sample position within each block of twenty samples is left blank for a control reference sample to be inserted prior to analyses.

Field observations were recorded on standard forms used by the Geological Survey of Canada (Garrett, 1974).

The sample site positions were marked on appropriate 1:250 000 scale NTS maps in the field. These maps were digitized at the Geological Survey in Ottawa to obtain the sample site UTM (NAD27) coordinates. Using GSC Open File 3939 (Lepard, 2008) these same coordinates were converted to NAD83 decimal degrees.

In Ottawa, field dried samples were air-dried and sieved through a minus 80 mesh (177 micron) screen, and ball-milled before analyses. At this time, control reference and blind duplicate samples were inserted into each block of twenty sediment samples. For the water samples, only control reference samples were inserted into the block. There were no blind duplicate water samples.

The sample site coordinates were checked as follows: a sample location map was produced on a Calcomp 1051 drum plotter using the digitized coordinates; the original sample location map produced in the field was then overlain on the Calcomp map; the two sets of points were checked for coincidence.

Thorough inspections of the field and analytical data were made to check for any missing information and/or gross errors.

Quality control and monitoring of the geochemical data was undertaken by a standard method used by the Exploration Geochemistry Subdivision (1989) and ICP-MS reanalysis QA/QC measures were carried out by Applied Geochemistry section staff (2007) at the Geological Survey of Canada.

Analytical Procedures

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 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.

Atomic Absorption Spectroscopy (AAS) and Other Analyses

For the determination of Zn, Cu, Pb, Ni, Co, Ag, Mn, Fe, Cd and As, a 1 gram sample is reacted with 3 mL concentrated HNO3 in a test tube overnight at room temperature. After digestion, the test tube is immersed in a hot water bath at room temperature and brought up to 90x C and held at this temperature for 30 minutes with periodic shaking. One mL of concentrated HCl is added and heating continues for another 90 minutes. The sample solution is then diluted to 20 mL with metal-free water and mixed. Zn, Cu, Pb, Ni, Co, Ag, Mn, Fe and Cd are determined by atomic absorption spectroscopy using an air-acetylene flame. Background corrections are made for Pb, Ni, Co, Ag, and Cd.

Arsenic is determined by atomic absorption using a hydride evolution method wherein the hydride (AsH3) is evolved and passed through a heated quartz tube in the light path of an atomic absorption spectrophotometer. The method is described by Aslin (1976).

Molybdenum and vanadium are determined by atomic absorption spectroscopy using a nitrous oxide acetylene flame. A 0.5 gram sample is reacted with 1.5 mL concentrated HNO3 at 90x C for 30 minutes. At this point, 0.5 mL concentrated HCl is added and the digestion continued at 90x C for an additional 90 minutes. After cooling, 8 mL of 1250 ppm Al solution are added and the sample solution diluted to 10 mL before aspiration.

Mercury is determined by the Hatch and Ott procedure with some modifications. The method is described by Jonasson et al. (1973). A 0.5 gram sample is reacted with 20 mL concentrated HNO3 and 1 mL concetrated HCl in a test tube for 10 minutes at room temperature prior to two hours of digestion with mixing at 90x C in a hot water bath. After digestion, the sample solutions are cooled and diluted to 100 mL with metal-free water. The Hg present is reduced to the elemental state by the addition of 10 mL 10% w/v SnSO4 in M H2SO4. The Hg vapour is then flushed by a stream of air into an absorption cell mounted in the light path of an atomic absorption spectrophotometer. Absorption measurements are made at 253.7 nm.

Loss-on-ignition is determined using a 500 mg sample. The sample, weighed into a 30 mL beaker, is placed in a cold muffle furnace and brought up to 500x C over a period of two to three hours. The sample is held at this temperature for four hours, then allowed to cool to room temperature for weighing.

Uranium is determined using a neutron activation method with delayed neutron counting. A detailed description of the method is provided by Boulanger et al.(1975). In brief, a 1 gram sample is weighed in a 7 dram polyethylene vial, capped and sealed. The irradiation is provided by the Slowpoke reactor with an operationg flux of 1012 neutrons/cm2/second. The samples are pneumatically transferred from an automatic loader to the reactor, where each sample is irradiated for 60 seconds. After irradiation, the sample is again transferred pneumatically to the counting facility where after a 10 second delay the sample is counted for 60 seconds with six BF3 detector tubes embedded in paraffin. Following counting, the samples are automatically ejected into a shielded storage container. Calibration is carried out twice a day as a minimum, using natural materials of known uranium concentration.

Antimony is determined as described by Aslin (1976). A 500 mg sample is placed in a test tube; 3 mL concentrated HNO3 and 9 mL concentrated HCl are added and the mixture allowed to stand overnight at room temperature. The mixture is heated slowly to 90x C and maintained at this temperature for at least 90 minutes. The solution is cooled and diluted to 10 mL. A one mL aliquot of this solution is diluted to 10 mL with 1.8 mL HCl. The antimony in an aliquot of this dilute solution is then determined by hydride evolution- atomic absorption spectrometry.

Fluorine is determined as described by Ficklin (1970). A 250 mg sample is sintered with 1 gram of a flux consisting of two parts by weight sodium carbonate and one part by weight potassium nitrate. The residue is then leached with water. The sodium carbonate is neutralized with 10 mL 10% (w/v) citric acid and the resulting solution diluted to 100 mL with water. The pH of the solution should range from 5.5 to 6.5. The fluoride content of the test solution is measured using a fluoride ion electrode. Standard solutions contain sodium carbonate and citric acid in the same quantities as the sample solution.

Gold is usually determined on a 10 g sediment sample; depending on the amount of sample available, lesser weights are sometimes used. This results in a variable detection limit: 2 ppb for a 5 g sample, 1 ppb for a 10 g sample... The sample is fused to produce a lead button, collecting any gold in the sample, which is cupelled in a muffle furnace to produce a siver (dore) bead. The silver beads are irradiated in a neutron flux for one hour, cooled for four hours, and counted by gamma ray spectrometry. Calibration is carried out using standard and blank beads.

Tungsten is determined as follows: a 0.2 g sample of sediment is fused with 1 g K2S2O7 in a rimless test tube at 575x C for 15 minutes in a furnace. The cooled melt is then leached with 10 mL concentrated HCl in a water bath heated to 85x C. After the soluble material has completely dissolved, the insoluble material is allowed to settle and an aliquot of 5 mL transferred to another test tube. 5 mL of 20% SnCl2 solution are then added to the sample aliquot, mixed and heated for 10 minutes at 85x C in a hot water bath. A 1 mL aliquot of dithiol solution (1% dithiol in iso-amyl acetate) is added to the test solution and the test solution then heated for 4 - 6 hours at 80x - 85x C in a hot water bath. This solution is then removed from the hot water bath, cooled and 2.5 mL of kerosene added to dissolve the globule. The colour intensity of the kerosene solution is measured at 630 nm using a spectrophotometer. The method is described by Quin and Brooks (1972). Detection level = 2 ppm.

Tin in stream sediments is determined by heating a 200 mg sample with NH4I: the sublimed SnI4 is dissolved in acid and the tin determined by atomic absorption spectrometry after solvent extraction of the tin into methyl isobutyl ketone containing trioctylphosphine oxide (TOPO). The method is described by E.P. Welsch and T.T. Chao (1976).

Barium is determined as follows: 2 mL of concentrated HCl are added to a 0.2 gram sample in a pressure tube and allowed to stand 20 minutes to drive off sulphides. Then, 1 mL HNO3, 1 mL HClO4 and 2 mL HF are added and the pressure tube capped and placed in a hot water bath for one hour to allow digestion. The tube is cooled, uncapped and filled with a 2.5% boric acid solution. After shaking, the solution is transferred to a 100 mL volumetric flask and diluted by a factor of 10 with a 10% cesium chloride solution.

Barium is determined by direct current plasma emission (DCP) spectroscopy.

Water Analyses

Fluoride in water samples is determined using a fluoride electrode.

Prior to measurement an aliquot of the sample is mixed with an equal volume of TISAB II buffer solution (total ionic strength adjustment buffer). The TISAB II buffer solution is prepared as follows: to 50 mL metal-free water add 57 mL glacial acetic acid, 58 g NaCl and 4 g CDTA (cyclohexylene dinitrilo tetraacetic acid). Stir to dissolve and cool to room temperature. Using a pH meter, adjust the pH between 5.0 and 5.5 by slowly adding 5 M NaOH solution. Cool and dilute to one liter in a volumetric flask. Detection limit = 20 ppb.

Hydrogen ion activity (pH) is measured with a combination glass-calomel electrode and a pH meter.

Uranium in waters is determined by a laser-induced fluorometric method using a Scintrex UA-3 uranium analyser. A complexing agent, known commercially as Fluran and composed of sodium pyrophosphate and sodium monophosphate (Hall, 1979) is added to produce the uranyl pyrophosphate species which fluoresces when exposed to the laser. Since organic matter in the sample can cause unpredictable behaviour, a standard addition method is used. Further, the reaction of uranium with Fluran can be delayed or sluggish; for this reason an arbitrary 24 hour time delay between the addition of the Fluran and the actual reading is incorporated into this method.

In practice, 500 ~L of Fluran solution are added to a 5 mL sample and allowed to stand for 24 hours. At the end of this period fluorescence readings are made with the addition of 0.0, 0.2 and 0.4 ppb U. For high samples the additions are 0.0, 2.0 and 4.0 (20 ~L aliquots of either 55 or 550 ppb U are used). All readings are taken against a sample blank.

A summary of analytical methods and detection limits is provided in Table 1.

Presentation and Interpretation of Gold Data

The following discussion reviews the format used to present the Au geochemical data and outlines some important points to consider when interpreting this data. This discussion is included in recognition of the special geochemical behaviour and mode of occurrence of Au in nature and the resultant difficulties in obtaining and analyzing samples which reflect the actual concentration level at a given site.

To correctly interpret Au geochemical data from regional stream sediment or lake sediment surveys requires an appreciation of the unique chemical and physical characteristics of Au and its mobility in the surficial environment. Key properties of gold that distinguish its geochemical behaviour from most other elements include (Harris, 1982):

1) Gold occurs most commonly in the native form which is chemically and physically resistant. A significant proportion of the metal is dispersed in micron-sized particulate form, and the high specific gravity of gold results in heterogeneous distribution, especially in stream sediment and clastic-rich (low LOI) lake sediment environments. Gold distribution appears to be more homogeneous in organic-rich fluviatile and lake sediments.

2) Gold typically occurs at low concentrations in the ppb range. Wheras gold concentrations of only a few ppm may represent economic deposits, background levels encountered from stream and centre-lake sediments seldom exceed 10 ppb, and commonly are near the detection limit of 2 ppb.

These factors result in a particle sparsity effect wherein very low concentrations of gold are heterogeneously enriched in the surficial environment. Hence, a major problem facing the geochemist is to obtain a representative sample. In general, areas where concentrations of gold in sediments are low, and/or grain sizes of the gold present relatively high require proportionally larger samples to reduce the uncertainty between subsample analytical values and actual values. Conversely, as actual gold concentrations increase or grain size decreases, the number of gold particles to be shared in random subsamples increases and variability of results decreases (Clifton et al., 1969; Harris, 1982). The limited ammount of material collected during the rapid, reconnaissance-style regional surveys and the need to analyze for a broad spectrum of elements, precludes the use of a significantly large sample weight for the gold analyses. Therefore, to obtain representative samples, grain size is reduced by sieving and ball milling of the dried sediments.

The following control methods are currently employed to evaluate and monitor the sampling and analytical variability which are inherent in the analysis of gold in geochemical media:

(1) For each block of 20 samples:

  (a) random insertion of a standard reference sample to control analytical accuracy and long-term precision;

  (b) collection of a field duplicate (two samples from one site) to measure sampling variance;

  (c) analysis of a second subsample (blind duplicate) from one sample to measure and control short-term precision.

(2) For both stream and lake sediments, routine repeat analyses on a second subsample are performed for all samples having values that are statistically above approximately the 90th percentile of the total total data set.

(3) For lake sediments only, a routine repeat analysis on a second subsample is performed on those samples with LOI values below 10%, indicating a large clastic component. Ongoing studies

suggest that the gold distribution in these samples is more likely to be variable than in samples with a higher LOI content.

Gold data listed in the open file may include initial analytical results, values determined from repeat analyses, together with sample weights and corresponding detection limits for all analyzed samples. Sample weights can be found in the silt data listing.

In summary, geochemical follow-up investigations for gold should be based on a careful consideration of all geological and geochemical information, and especially a careful appraisal of gold geochemical data and its variability. In some instances, prospective follow-up areas may be indirectly identified by pathfinder element associations in favourable geology, although a complementary gold response due to natural variability may be lacking. Once an anomalous area has been identified, field investigations should by designed to include detailed geochemical follow-up surveys and collection of large representative samples. Subsequent repeat subsample analyses will increase the reliability of results and permit a better understanding of natural variability which can then by used to improve sampling methods and interpretation.

TABLE 1. Summary of Analytical Data and Methods

Element	Analytical Method	Units of Measurement	Detection Limit
Silt			
Ag	AAS	ppm	0.2
Ag	ICP-MS	ppb	2
Al	ICP-MS	%	0.01
As	HY-AAS	ppm	1
As	ICP-MS	ppm	0.1
Au	FA-NA	ppb	1
Au_wt		g	0.1
Au1	FA-NA	ppb	1
Au1_wt		g	0.1
B	ICP-MS	ppm	1
Ba	DCP	ppm	40
Ba	ICP-MS	ppm	0.5
Bi	ICP-MS	ppm	0.02
Ca	ICP-MS	%	0.01
Cd	AAS	ppm	0.2
Cd	ICP-MS	ppm	0.01
Co	AAS	ppm	2
Co	ICP-MS	ppm	0.1
Cr	ICP-MS	ppm	0.5
Cu	AAS	ppm	2
Cu	ICP-MS	ppm	0.01
F	ISE	ppm	20
Fe	AAS	pct	0.02
Fe	ICP-MS	%	0.01
Ga	ICP-MS	ppm	0.2
Hg	CV-AAS	ppb	10
Hg	ICP-MS	ppb	5

K	ICP-MS	%	0.01
La	ICP-MS	ppm	0.5
LOI	GRAV	pct	1.0
Mg	ICP-MS	%	0.01
Mn	AAS	ppm	5
Mn	ICP-MS	ppm	1
Mo	AAS	ppm	2
Mo	ICP-MS	ppm	0.01
Na	ICP-MS	%	0.001
Ni	AAS	ppm	2
Ni	ICP-MS	ppm	0.1
P	ICP-MS	%	0.001
Pb	AAS	ppm	2
Pb	ICP-MS	ppm	0.01
S	ICP-MS	%	0.01
Sb	HY-AAS	ppm	0.2
Sb	ICP-MS	ppm	0.02
Sc	ICP-MS	ppm	0.1
Se	ICP-MS	ppm	0.1
Sn	AAS	ppm	1
Sr	ICP-MS	ppm	0.5
Te	ICP-MS	ppm	0.02
Th	ICP-MS	ppm	0.1
Ti	ICP-MS	%	0.001
Tl	ICP-MS	ppm	0.02
U	ICP-MS	ppm	0.1
U	NADNC	ppm	0.5
V	AAS	ppm	5
V	ICP-MS	ppm	2
W	COL	ppm	2
W	ICP-MS	ppm	0.1
Zn	AAS	ppm	2
Zn	ICP-MS	ppm	0.1
Waters			
F-w	ISE	ppb	20
pH	GCM	-	-
U-w	LIF	ppb	0.05

AAS - atomic absoprtion spectrometry
GCM - glass Calomel electrode and pH meter
GRAV - gravimetry
ICP-MS - inductively coupled plasma mass spectrometry
ISE - ion selective electrode
LIF - laser-induced fluorescence
NADNC - neutron activation, delayed neutron counting
HY-AAS - atomic absorption using hydride evolution
CV-AAS - cold vapour (flameless) atomic absorption

CREDITS (1989)

E.H.W. Hornbrook directed the survey.

P.W.B. Friske coordinated the operational activities of contract and Geological Survey of Canada staff.

Contracts were let to the following companies for sample preparation and analysis and were managed by the following staff of the Regional Geochemical Studies section:

Collection:	Monaghan Delph Miller Don Mills, Ontario E.H.W. Hornbrook P.W.B. Friske
Preparation:	Golder Associates Ottawa, Ontario J.J. Lynch
Analysis:	Bondar-Clegg and Company Ottawa, Ontario Chemex Labs (waters and Au) North Vancouver, British Columbia J.J. Lynch

H. R. Schmitt coordinated and edited open file production.

A.C. Galletta and D. Wright managed the digital geochemical data and provided computer processing support. Computing services were provided by the Computer Science Centre, EMR. The plotting was done by Canada Lands Data Systems staff at Environment Canada, Hull, Quebec.

H. Gross developed microcomputer software to produce data listings and summary statistics.

J. Yelle and F. Williams of the Geological Information Division supervised the preparation of open file base maps by Cartography Unit A-2 and Terra Surveys Ltd., Ottawa.

M. McCurdy and S. Cook provided technical assistance.

J.C. Belec provided word processing support.

CREDITS (2007)

ICP-MS	Acme Analytical Laboratories Vancouver, British Columbia P.W.B. Friske

S. Day edited and compiled this open file.

John Buckle of the GSC reviewed this open file and provided many useful comments and suggestions.

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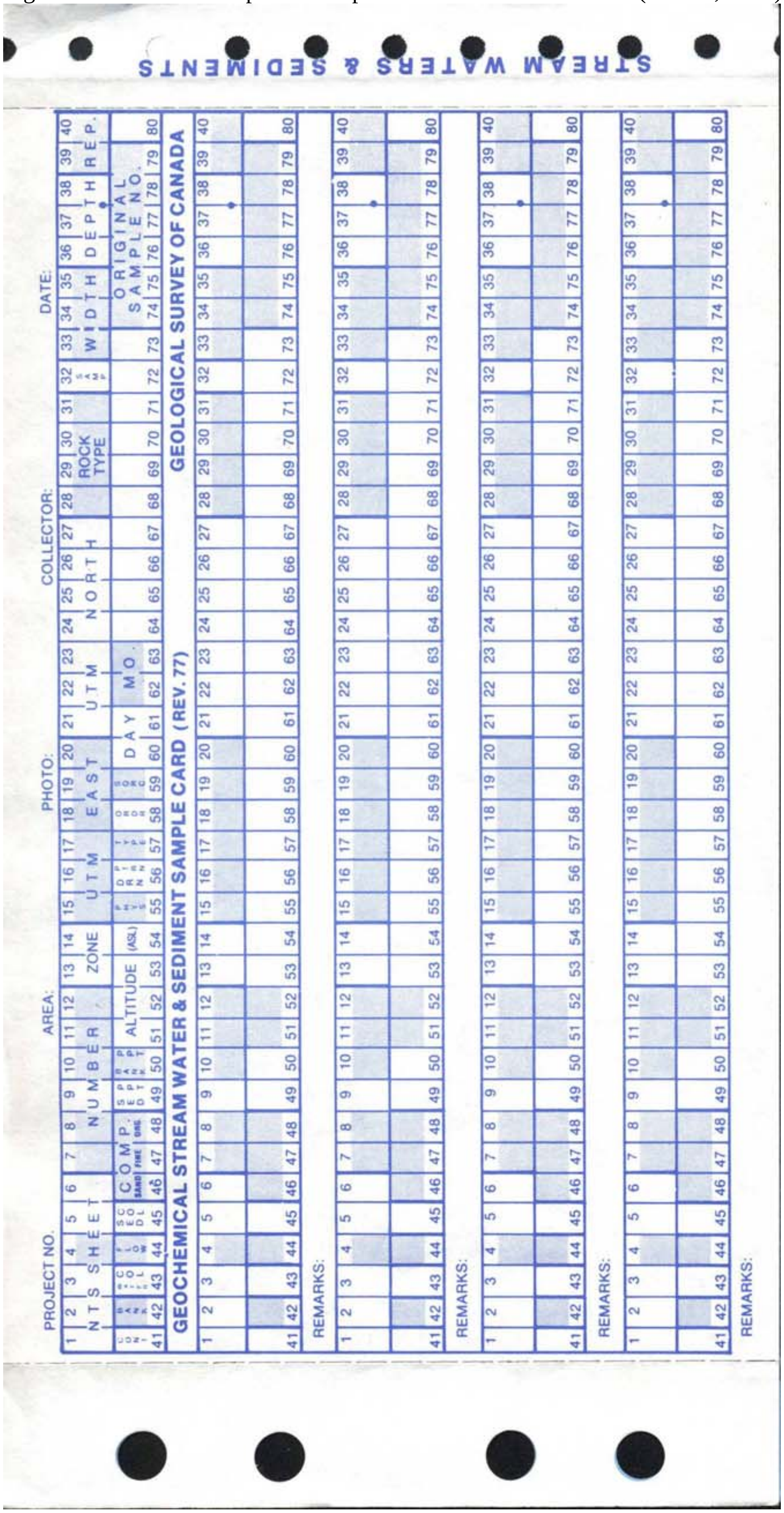
Figure 2: Card used to capture site-specific field observation data (Garrett, 1974)



STREAM WATERS & SEDIMENTS

PROJECT NO. AREA: PHOTO: COLLECTOR: DATE:

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	39	40
NTS SHEET						NUMBER						ZONE		UTM EAST						UTM NORTH								ROCK TYPE			SAMP	WIDTH			DEPTH			REP.	
CONT	BANK	WATR COL	FLOW	SED COL	COMP. SAND	FINE	ORG	SED PPT	BANK PPT	ALTITUDE			(ASL)	PHYS	DRN PTRN	TYPE	ORDR	SORC	DAY		MO.												ORIGINAL SAMPLE NO.						
41	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	80

GEOCHEMICAL STREAM WATER & SEDIMENT SAMPLE CARD (REV. 77) GEOLOGICAL SURVEY OF CANADA

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	39	40
41	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	80

REMARKS:

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REMARKS:

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41	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	80

REMARKS:

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	39	40
41	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	80

REMARKS: