

REGIONAL LAKE SEDIMENT AND WATER GEOCHEMICAL RECONNAISSANCE DATA, ONTARIO 1977.

GSC-OF 507, NGR 18-1977, NTS 52A, 52H(S/2)

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OPEN FILE 507 IS ONE OF TWO OPEN FILES (506 AND 507) COVERING ONTARIO - THUNDER BAY SURVEY AREA.

THE RECONNAISSANCE SURVEY WAS UNDERTAKEN BY THE GEOLOGICAL SURVEY OF CANADA IN CONJUNCTION WITH THE ONTARIO MINISTRY OF NATURAL RESOURCES UNDER THE TERMS OF THE CADADA-ONTARIO AGREEMENT ON A URANIUM RECONNAISSANCE PROGRAM. FISHERIES AND ENVIRONMENT CANADA PROVIDED FUNDS FOR THE DETERMINATION OF MERCURY.

E.H.W. HORN BROOK DIRECTED GEOLOGICAL SURVEY OF CANADA ACTIVITIES. CONTRACTS LET FOR SAMPLE COLLECTION, PREPARATION AND ANALYSIS WERE SUPERVISED AND/OR MONITORED BY STAFF OF THE GEOCHEMISTRY SECTION AS FOLLOWS:

COLLECTION - MARSHALL MACKLIN MONOGHAN LIMITED, TORONTO.  
- E.H.W. HORN BROOK, W.B. COKER

PREPARATION - GOLDR ASSOCIATES, OTTAWA.  
- J.J. LYNCH

ANALYTICAL - CHEMEX LABS. LIMITED, VANCOUVER.  
- BARRINGER RESEARCH LIMITED, TORONTO.  
- ATOMIC ENERGY OF CANADA LIMITED, OTTAWA.  
- J.J. LYNCH

AT THE GEOLOGICAL SURVEY OF CANADA, N.G. LUND WAS RESPONSIBLE FOR OPEN FILE PRODUCTION AND DATA MANAGEMENT AND WAS SUPPORTED BY F. WILLIAMS OF THE CARTOGRAPHIC SECTION WHO SUPERVISED MAP PREPARATION. DIRECT PHOTO-HEAD PLOTTING FACILITIES WERE MADE AVAILABLE BY THE MAP PRODUCTION DIRECTORATE, SURVEYS AND MAPPING BRANCH, E.M.R.

PROVINCIAL LIASION WAS WITH R.B. BARLOW, ONTARIO MINISTRY OF NATURAL RESOURCES.

LAKE SEDIMENT AND WATER SAMPLES WERE COLLECTED AT AN AVERAGE DENSITY OF ONE SAMPLE PER 13 SQUARE KILOMETRES (5 SQUARE MILES) THROUGHOUT THE 27700 SQUARE KILOMETRE (10700 SQUARE MILE) TOTAL THUNDER BAY SURVEY AREA. THE HELICOPTER SUPPORTED SAMPLE COLLECTION WAS CARRIED OUT DURING THE SUMMER OF 1977. SAMPLE SITE AND GRID CELL DUPLICATE SAMPLES WERE ROUTINELY COLLECTED IN EACH ANALYTICAL BLOCK OF TWENTY SAMPLES.

IN OTTAWA, FIELD DRIED SAMPLES WERE AIR-DRIED, CRUSHED AND BALL MILLED. THE MINUS 80 MESH (177 MICRONS) FRACTION WAS OBTAINED AND USED FOR SUBSEQUENT

## ANALYSES.

AS REQUIRED, AT THIS TIME, CONTROL REFERENCE AND BLIND DUPLICATE SAMPLES WERE INSERTED INTO EACH ANALYTICAL BLOCK OF TWENTY SEDIMENT AND WATER SAMPLES. NO OTHER SAMPLE PROCESSING IN OTTAWA WAS CARRIED OUT ON THE WATER SAMPLES.

THE DETERMINATION OF ZN, CU, PB, NI, CO, AG, MN, FE, MO, HG, AS AND LOSS ON IGNITION IN LAKE SEDIMENTS WAS CARRIED OUT BY CHEMEX LABS LIMITED.

THE DETERMINATION OF U IN LAKE SEDIMENTS WAS CARRIED OUT BY ATOMIC ENERGY OF CANADA LIMITED.

THE DETERMINATION OF U, F AND PH IN LAKE WATERS WAS CARRIED OUT BY BARRINGER RESEARCH LIMITED.

FOR THE DETERMINATION OF ZN, CU, PB, NI, CO, AG, MN AND FE, A 1 GRAM SAMPLE WAS REACTED WITH 6 ML OF A MIXTURE OF 4M HCL AND M HNO<sub>3</sub> IN A TEST-TUBE OVERNIGHT AT ROOM TEMPERATURE.

AFTER THE OVERNIGHT DIGESTION THE TEST-TUBE WAS IMMERSSED IN A HOT WATER BATH AT ROOM TEMPERATURE AND BROUGHT UP TO 90C AND HELD AT THIS TEMPERATURE FOR 2 HOURS WITH PERIODIC SHAKING.

THE SAMPLE SOLUTION WAS THEN DILUTED TO 20 ML WITH METAL FREE WATER AND MIXED. ZN, CU, PB, NI, CO, AG, MN AND FE WERE DETERMINED BY ATOMIC ABSORTION SPECTROSCOPY USING AN AIR-ACETYLENE FLAME.

BACKGROUND CORRECTIONS WERE MADE FOR PB, NI, CO AND AG.

ARSENIC WAS DETERMINED COLORIMETRICALLY USING SILVER DIETHYLDITHIOCARBAMATE. DECOMPOSITION WAS ACCOMPLISHED BY HEATING A 1 GRAM SAMPLE WITH 20 ML OF 6M HCL AT 90C FOR 1.5 HOURS. COLORIMETRIC MEASUREMENTS WERE MADE AT 520 NM.

MOLYBDENUM WAS DETERMINED BY ATOMIC ABSORTION SPECTROSCOPY USING A NITROUS OXIDE-ACETYLENE FLAME.

A 0.5 GRAM SAMPLE WAS REACTED WITH 1.5 ML CONCENTRATED HNO<sub>3</sub> AT 90C FOR 30 MINUTES.

AT THIS POINT 0.5 ML CONCENTRATED HCL WAS ADDED AND THE DIGESTION WAS CONTINUED AT 90C FOR AN ADDITIONAL 90 MINUTES.

AFTER COOLING, 8 ML OF 1250 PPM AL SOLUTION WERE ADDED AND THE SAMPLE SOLUTION WAS 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 GRAM SAMPLE WAS REACTED WITH 20 ML CONCENTRATED HNO<sub>3</sub> AND 1 ML CONCENTRATED HCL IN A TEST-TUBE FOR 10 MINUTES AT ROOM TEMPERATURE PRIOR TO 2 HOURS OF DIGESTION WITH MIXING AT 90C 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 W/V

SNSO<sub>4</sub> IN M H<sub>2</sub>SO<sub>4</sub>.

THE HG VAPOUR WAS THEN FLUSHED BY A STREAM OF AIR INTO AN ABSORTION CELL MOUNTED IN THE LIGHT PATH OF AN ATOMIC ABSORTION SPECTROPHOTOMETER. ABSORTION MEASUREMENTS WERE MADE AT 253.7 NM.

LOSS ON IGNITION WAS DETERMINED USING A 500 MG SAMPLE. THE SAMPLE, WEIGHED INTO 30 ML BEAKER, WAS PLACED IN A COLD MUFFLE FURNACE AND BROUGHT UP TO 500C OVER A PERIOD OF 2-3 HOURS. THE SAMPLE WAS LEFT AT THIS TEMPERATURE FOR 4 HOURS, THEN ALLOWED TO COOL TO ROOM TEMPERATURE FOR WEIGHING.

URANIUM WAS 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 INTO A 7 DRAM POLYETHYLENE VIAL, CAPPED AND SEALED.

THE IRRADIATION IS PROVIDED BY THE SLOWPOKE REACTOR WITH AN OPERATING FLUX OF 10\*\*12 NEUTRONS/SQ. CM./SEC.

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 BF<sub>3</sub> DETECTOR TUBES EMBEDDED IN PARRAFIN.

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.

URANIUM, FLUORIDE AND PH WERE DETERMINED IN LAKE WATER SAMPLES.

UPON RECEIVING A BATCH OF SAMPLES, FLUORIDE AND PH WERE DETERMINED BY SPECIFIC ION ELECTRODE AND GLASS-CALOMEL COMBINATION ELECTRODE RESPECTIVELY.

AFTER THESE TWO DETERMINATIONS WERE COMPLETED, THE REMAINING WATER IN THE SAMPLE BOTTLE (APPROX. 225 ML) WAS ACIDIFIED WITH 3 ML CONCENTRATED HNO<sub>3</sub>.

TWO WEEKS AFTER ACIDIFICATION, A 5 MICROLITRE ALIQUOT OF THE SAMPLE WAS THEN REMOVED FOR THE DETERMINATION OF URANIUM BY FISSION TRACK ANALYSES.

THE TWO WEEK WAITING PERIOD WAS TO INSURE THAT ALL PRECIPITATED URANIUM WAS REDISSOLVED.

TO DETERMINE URANIUM, SAMPLE ALIQUOTS WERE PLACED ON A POLYCARBONATE TAPE AND DRIED.

THE TAPE WAS THEN IRRADIATED IN A NUCLEAR REACTOR AT MCMASTER UNIVERSITY FOR 1 HOUR IN A FLUX OF 10\*\*13 NEUTRONS/SQ. CM./SEC.

THE TAPE WAS SUBSEQUENTLY ETCHED WITH 25% NAOH SOLUTION AND THE FISSION TRACKS WERE COUNTED WITH AN OPTICAL COUNTER FITTED TO A MICROSCOPE.

THE NUMBER OF TRACKS WAS PROPORTIONAL TO THE URANIUM CONCENTRATION.

EACH TAPE CONTAINED ITS OWN CALIBRATION STANDARDS, BLANKS AND SAMPLE DUPLICATES.

FLUORIDE IN LAKE WATER SAMPLES WAS DETERMINED USING AN ORION FLUORIDE ELECTRODE AND A MODEL 401 ORION SPECIFIC ION METER.

PRIOR TO MEASUREMENT AN ALIQUOT OF THE SAMPLE WAS MIXED WITH AN EQUAL VOLUME OF A MODIFIED TISAB SOLUTION (TOTAL IONIC STRENGTH ADJUSTMENT BUFFER). THE MODIFICATION CONSISTED OF ADDING 60 ML 8M KOH SOLUTION TO THE BUFFER. THIS PERMITTED THE RE-ANALYSIS OF FLUORIDE IN ACIDIFIED WATER SAMPLES WHEN REQUIRED. WHEN THIS ANALYSIS WAS REQUIRED, ACIDIFIED STANDARD SOLUTIONS WERE USED FOR CALIBRATION.

HYDROGEN ION ACTIVITY (PH) WAS MEASURED WITH A BECKMAN COMBINATION ELECTRODE AND A MODEL 401 ORION SPECIFIC ION METER.

ON RECEIPT, FIELD AND ANALYTICAL DATA WERE PUNCHED ONTO 80 COLUMN CARDS AND ALL SUBSEQUENT PROCESSING WAS CARRIED OUT WITH THE AID OF COMPUTERS. THE FIELD DATA WERE RECORDED BY THE FIELD CONTRACT STAFF ONTO STANDARD LAKE SEDIMENT FIELD CARDS (REV. 74) 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 COORDINATES. THE DOMINANT ROCK TYPES IN THE LAKE CATCHMENT BASINS WERE IDENTIFIED ON APPROPRIATE GEOLOGICAL MAPS USED AS THE BEDROCK GEOLOGICAL BASE ON NGR MAPS.

THE ANALYTICAL DATA WERE RECORDED AS FOLLOWS (SEE GARRETT, 1974, FOR DETAILS) AND FOR CONVENIENCE THE DETECTION LIMITS OF THE ANALYTICAL METHODS USED ARE ALSO GIVEN-

ELEMENT	ANAL. CARD	COLUMNS	DETECTION LIMIT	
SEDIMENT				
ZN	1	21-25	2	1
CU	1	26-30	2	1
PB	1	31-35	2	1
NI	1	36-40	2	1
CO	1	41-45	2	1
AG	1	46-50	0.2	0.1
MN	1	51-55	5	2
AS	1	56-60	1	0.5
MO	1	61-65	2	1
FE %	1	66-70	0.02	0.01
HG PPB	1	71-75	10	5
LOI %	1	76-79	1.0	0.5
U	2	21-25	0.2	0.1
WATER				
U PPB	3	21-25	0.01	0.005
F PPB	3	26-30	20	10

UNLESS OTHERWISE NOTED THE UNITS OF MEASUREMENT FOR THE ANALYSES ARE PPM. THE SECOND FIGURE UNDER DETECTION LIMIT IS THE FIGURE TO WHICH VALUES WERE ARBITRARILY SET IF THEY FELL BELOW THE DETECTION LIMIT.

GENERAL INSPECTIONS OF THE FIELD AND ANALYTICAL DATA WERE MADE TO CHECK FOR ANY MISSING INFORMATION AND/OR GROSS ERRORS. THE SAMPLE SITE COORDINATES WERE CHECKED BY PLOTTING SAMPLING LOCATION MAPS ON A FLAT-BED PLOTTER FROM THE DIGITIZED COORDINATES AND THEN OVERLAYING THESE OVER THE FIELD CONTRACTOR'S SAMPLE LOCATION BASE MAPS.

QUALITY CONTROL AND MONITORING OF THE GEOLOGICAL DATA WAS UNDERTAKEN BY A STANDARD METHOD USED BY THE GEOCHEMISTRY SECTION AT THE GEOLOGICAL SURVEY OF CANADA.

#### REFERENCES

BOULANGER, A., EVANS, D.J.R. AND RABY, B.F. (1975) URANIUM ANALYSIS BY NEUTRON ACTIVATION DELAYED NEUTRON COUNTING: PROC. OF THE 7TH ANNUAL SYMP. OF CANADIAN MINERAL ANALYSTS. THUNDER BAY, ONTARIO, SEPT. 22-23, 1975.

GARRETT, R.G. (1974) FIELD DATA ACQUISITION METHODS FOR APPLIED GEOCHEMICAL SURVEYS AT THE GEOLOGICAL SURVEY OF CANADA: GEOL SURV. CAN. PAPER 74-52

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: GEOL. SURV. CAN. PAPER 73-21.

## DATA LIST LEGEND

MAP-	NATIONAL TOPOGRAPHIC SYSTEM(NTS)- LETTERED QUADRANGLE (SCALE 1:250000). PART OF SAMPLE NUMBER	ROCK TYPE:	DIBS- DIABASE SMRK- SEDIMENTARY ROCK ARGL- ARGILLITE CHRT- CHERT GRNT- GRANITE GRNG- GRANITE GNEISS UMFC- ULTRAMAFIC CGLM- CONGLOMERATE GRCK- GRAYWACKE BEXV- BASIC EXTRUSIVE MVCC- META VOLCANIC UKNN- UNKNOWN
SAMPLE-	REMAINDER OF SAMPLE NUMBER- YEAR(2), FIELD CREW(1), SAMPLE SEQUENCE NUMBER(3)		
UTM COORDINATES-	UNIVERSAL TRANVERSE MERCATOR(UTM) COORDINATE SYSTEM- SAMPLE COORDINATES		
ZN-	ZONE		
EAST-	EASTING (METERS)		
NORTH-	NORTHING (METERS)		
ROCK TYPE-	MAJOR ROCK TYPE OF LAKE CATCHMENT AREA	LAKE AREA:	POND- POND LT 1- 1/4 TO 1 SQ KM 1-5- 1 TO 5 SQ KM GT 5- GREATER THAN 5 SQ KM
LAKE AREA-	AREA OF LAKE SAMPLED		
SMP DTH-	SAMPLE DEPTH MEASURED TO THE NEAREST FOOT		
RP ST-	REPLICATE STATUS- RELATIONSHIP OF SAMPLE WITH RESPECT TO OTHERS WITHIN THE SURVEY	RP ST:	00- ROUTINE REGIONAL SAMPLE 10- FIRST OF FIELD DUPLICATE 20- SECOND OF FIELD DUPLICATE 70- CELL DUPLICATE SITE SAMPLE
REL-	RELIEF OF THE SURROUNDING LAKE CATCHMENT BASIN		
GEL-	PRESENCE OF AN ORGANIC GEL OR GYTJA	REL:	L- LOW M- MEDIUM H- HIGH
CONT-	CONTAMINATION- HUMAN OR NATURAL (WORK-DRILL/TRENCH, CAMP, FUEL OR GOSSAN)		
SAMP COLOR-	SEDIMENT COLOUR		
SUSP-	SUSPENDEED MATTER	GEL:	BLANK- ABSENT
ZN-	ZINC BY ATOMIC ABSORPTION SPECTROSCOPY (PPM)		
CU-	COPPER BY ATOMIC ABSORPTION SPECTROSCOPY (PPM)		
PB-	LEAD BY ATOMIC ABSORPTION SPECTROSCOPY (PPM)	CONT:	BLANK- NONE 1- PRESENT
NI-	NICKEL BY ATOMIC ABSORPTION SPECTROSCOPY (PPM)		
CO-	COBALT BY ATOMIC ABSORPTION SPECTROSCOPY (PPM)		
AG-	SILVER BY ATOMIC ABSORPTION SPECTROSCOPY (PPM)		
MN-	MANGANESE BY ATOMIC ABSORPTION SPECTROSCOPY (PPM)	SAMP COLOR:	TN- TAN YL- YELLOW GN- GREEN GY- GREY BR- BROWN BK- BLACK
AS-	ARSENIC COLORIMETRICALLY (PPM)		
MO-	MOLYBDENUM BY ATOMIC ABSORPTION SPECTROSCOPY (PPM)		
FE-	IRON BY ATOMIC ABSORPTION SPECTROSCOPY (%)		
HG-	MERCURY BY FLAMELESS SPECTROSCOPY (PPB)		
LOI-	LOSS ON IGNITION BY WEIGHT DIFFERENCE (%)		
U-	URANIUM BY DELAYED NEUTRON ACTIVATION (PPM)		
U-W-	URANIUM IN WATERS BY FISSION TRACK (PPB)		
F-W-	FLUORINE IN WATERS BY SPECIFIC ION ELECTRODE (PPB)	SUSP:	BLANK- NONE L- LIGHT H- HEAVY
PH-	PH BY COMBINATION GLASS - CALOMEL ELECTRODE		