1973 Geophysical and Geochemical Report
EAGLE CREEK COPPER PROPERTY

Claims WA 1-16 incl.

Located 16 miles north of Lac La Hache, B.C. Latitude 52°02'N Longitude 121°26'W Cariboo Mining Division 93 A 3

By G.M. DePaoli, B.Sc. Geophysicist and J.F. Allan, P.Eng. (B.C.)

Work was carried out during October 3-5,1972 and May 25-30,1973



TITLE

AUTHORS

DATE

COMMODITY

LOCATION-Area

- -Mining Division
- -Coordinates
- -NTS

Eagle Creek Copper Property (WA Claims)

G.M. DePaoli, B.Sc. Geophysicist and J.F. Allan, P.Eng. (B.C.)

July 1973

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Lac La Hache Cariboo Latitude 52°02'N Longitude 121°26'W 93 A 3

AMAX Vancouver Office

Department of

Mines and Petroleum Resources

ASSESSMENT REPORT

NO. 4556 MAP

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SUMMARY

During the fall of 1972 and spring of 1973 several geophysical and geochemical traverses were completed on the Eagle Creek Copper Property.

A coincident magnetic and resistivity anomaly were obtained on one traverse and a weak induced polarization anomaly was obtained at depth on an adjacent line. The variability in the remaining data can be explained by changes in lithology.

Geochemical results were negative and sparse outcrop exposure suggests that the property is overlain by a thin veneer of Tertiary basalts and fluvio-glacial sands and gravels.

INTRODUCTION

The Eagle Creek Copper Property consists of sixteen mineral claims owned by Amax Potash Limited. During the period October 3-5, 1972 two coincident magnetic and battery powered induced polarization traverses were surveyed over the property. In addition soil samples were taken every two hundred feet along each traverse. In the following year during May 25 - 30 two subparallel lines were cut on the property and subsequently surveyed with a higher powered I.P. unit. The following report describes the instrumentation, field procedure and results obtained from the above surveys.

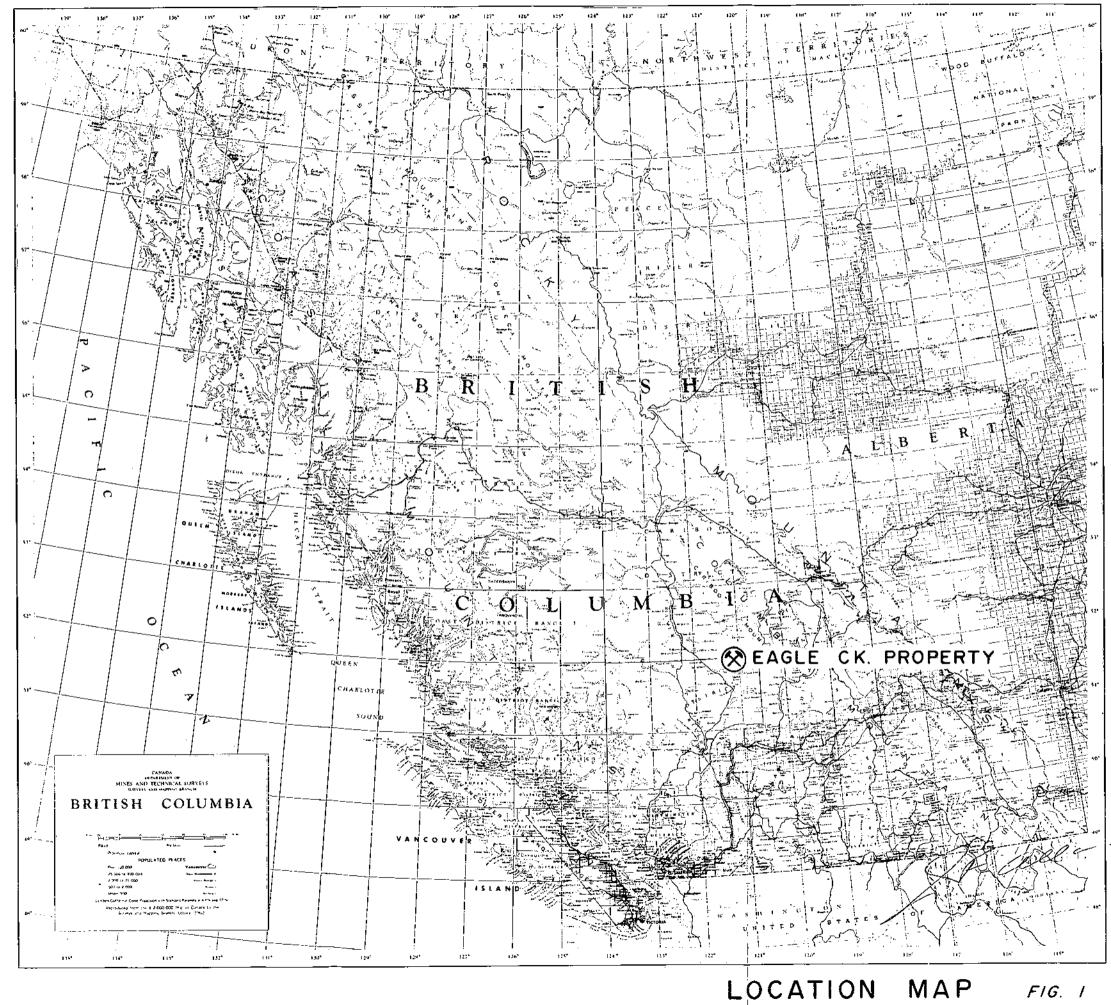
Location and Access

The property is located within the Interior Plateau of south-central British Columbia, sixteen miles north of Lac La Hache. It is accessible by two wheeled drive vehicle via the Murphy Lake gravel road. It lies in the Cariboo Mining Division at 52°02'N latitude and 121°26'W longitude (see Figure 1).

Grid Control

Control for the initial traverses conducted in 1972 consisted of chain and compassed flagged lines. The traverses

Department of Mines and Petroleum despurces ASSESSMENT REPORT NO 4556 MAP#1



MAP

were named #1 and #2 and are 2200 feet and 2600 feet long respectively. Control for the later I.P. work was established by two chain and compassed, cut and picketed lines both 2800 feet long. They have been marked as the north and south I.P. lines. All four traverses are shown in plan view on Figure 2.

GENERAL GEOLOGY

Regionally the property is situated on the northwestern segment of an annular magnetic high which is coincident with Nicola strata that lie peripheral to a monzonite stock. Outcrop on the property is almost nonexistent and to date only one exposure has been found. It is a porphyritic basalt typical of Tertiary flows in the area.

MAGNETOMETER SURVEY

Introduction and Theory

The ground magnetometer traverses were undertaken to accurately locate the axis of an aeromagnetic anomaly visible on aeromagnetic map sheet 93 A 3.

The magnetism of all rocks is controlled by their content of ferromagnetic material, i.e. substances possessing a relatively high susceptibility and capable of acquiring permanent magnetization. Often skarn mineral assemblages include considerable amounts of mineral magnetite. High intensity magnetic anomalies may therefore be used as an indicator of skarn environments.

Instrument and Procedure

The instrument employed was the Model MF-2 magnetometer manufactured by Sharpe Instruments, a division of Scintrex Ltd., Downsview, Ontario. It operates on the fluxgate principle and measures the vertical component of the earth's magnetic field.

LEGEND Blozed lines Traverse line; soil sample site sample number. Claim post, claim location line Claim boundary Department of Mines and Petrale on Resources ASSESSME.IT REPORT NO 4556 MAP#2 AMAX POTASH LIMITED EAGLE CREEK PROPERTY CLAIMS CARIBOO MINING DIVISION --- BRITISH CLAIM MAP TRAVERSE LOCATIONS I" = 1,000' SCALE TO accompany: "1973 GEOPHYSICAL AND GEOCHEMICAL REPORT -EAGLE CREEK COPPER PROPERTY " by G.M. De Pagif and J.F. Allan.

The MF-2 circuitry is temperature compensated to less than 1 gamma per °C from -40°C to +40°C and its measurement range is from +100,000 gammas to -100,000 gammas. On the most sensitive scale the sensitivity is 20 gammas per scale division or a readability of 10 gammas.

Using the latitude adjust control the instrument was set to its most sensitive scale at the western end of traverse line #1. A closed loop survey was completed back to this point in less than two hours. No diurnal change was noted and no diurnal corrections were applied to the data.

Results and Discussion

The data is plotted in profile format after page 3.

The highest magnetic anomaly, which reached a peak value of 6000 gammas was obtained on traverse line #1. The intensity of this anomaly suggests the presence of a magnetite concentration within 100 feet of ground surface. A resistivity high and background I.P. effects are associated with the anomaly.

Two smaller magnetic anomalies having maximum values of 2,500 gammas were obtained on traverse line #2. A change in the background chargeability values is also noted near these two anomalies. The coincidence of both parameters may be reflecting a lithological contact.

INDUCED POLARIZATION SURVEYS

Introduction and Theory

During the period October 3 - 5, 1972 traverse lines #1 and #2 were surveyed with a portable time domain battery powered induced polarization unit. The survey was executed by AMAX personnel who concurrently collected soil samples.

The following year, during May 25 - 30, 1973 the north and south I.P. lines were cut and then surveyed employing a frequency domain McPhar P660 induced polarization system,

powered by a 2.5 kilowatt motor generator. This work was contracted to Dennis F. Morrison of Gravenhurst, Ontario.

In both surveys a 200 foot dipole-dipole configuration was used. In the first survey only the first separation measurements were taken, while in the second survey five separations were obtained (n=5).

The term induced polarization means electrical polarization (i.e. separation of charges) induced by an applied electric field. The cause of this polarization is changes in the mobilities of ions within a rock. At the interfaces between zones of different mobilities, excesses of deficiences of ions occur. The concentration gradients developed oppose the current flow and cause a polarizing effect. When mineral grains block the pore passages of rocks and a current is applied, a concentration of ions builds up at the electrolyte(water)-metal interface while awaiting an electrochemical reaction which must occur before the electric charge can be transferred from an ion in the electrolyte to a free electron in the metal. The forces which oppose the current flow are said to polarize the interface and the added voltage necessary to drive the current across this barrier is known as "overvoltage".

In the pulse-transient or time domain method that was employed, the interfaces within the rock were polarized by applying a steady direct current. The current was then abruptly terminated and measurement was made of the small decaying voltage caused by the polarized charges returning to equilibrium.

It takes a finite time to build up overvoltages and one finds that the impedances of these zones (Warburg Impedance) decreases with increasing frequency. In the frequency domain system that was employed, the decrease in the Warburg Impendance was measured between current applied at 0.3 hertz (Ac 1) to current applied at 5.0 hertz (AC 2).

Resistivity information is useful in inferring overburden depths, defining abrupt lithological changes, and assessing the importance of any I.P. effects obtained.

Instruments and Procedures

AMAX's portable I.P. unit was used for the first survey on Traverse lines 1 and 2. The equipment consists of the IPR-7 Newmont-type receiver (15 pounds) and the IPC-7 25 watt battery powered transmitter (13 pounds). The receiving dipole consisting of a 200 foot length of wire connected to porous pots filled with a saturated solution of CuSO₄. The transmitting dipole employed a 200 foot length of wire connecting four foot stainless steel rod electrodes.

Survey procedure required four men equispaced 200 feet apart along the line. The advance man prepared the electrode site for the lead potential electrode by digging a small hole. When moving the array the lead man advanced the potential dipole wire two hundred feet. The second man operated the receiver. He normally situated his electrode in the same site the lead man prepared. Because distance permits, the receiver operator signals moves and transmitter "on" periods by voice. The third man operates the transmitters. He establishes his current electrode and advances the 200 foot current dipole wire. The trailing man prepares the second current electrode site. Using vicegrip pliers the last two "current" men retract the stainless steel rods they have hammered into the ground and reuse them on the next set-up.

On the second I.P. survey a multiple frequency domain McPhar P660 induced polarization system was employed. The transmitter is a manually variable voltage source. The output current can be selected from both polarities and varies from direct current to automatically alternating output frequencies of 0.05, 0.1, 0.3, 1.25, 2.5 and 5.0 hertz. Power was obtained

from a $2\frac{1}{2}$ kilowatt - 400 hertz motor generator. The maximum output voltage is 690 volts. The receiver is a potentiometer-type where the amplified and filtered signal is compared with a reference voltage. It is powered by six 8V alkaline transistor batteries and draws 7.5 m.a.

Survey procedure required the preparation of a set-up position near the center of each line. The transmitter and its motor generator power supply remained stationary at the set-up position and wires in increasing two hundred foot intervals were strung out in both directions. Care was taken to ensure that the wires were well separated to prevent inductive coupling effects. The ends of the wires were connected to aluminum foil electrodes which had been prepared earlier. The receiving dipole consisting of the receiver and a 200 foot "read" wire also utilized the aluminum foil electrodes where possible. However, once the receiving dipole moved past the last foil emplaced for the transmitting set-up, ground connections were made via porous pots containing a solution of copper sulphate. Radio contact between the receiver and transmitter operations coordinated power "on" and "off" periods.

Results and Discussions

The initial battery I.P. survey results are plotted in profile format on Figures 3(a) and 3(b) while the latter survey is plotted in pseudosection format on Figures 4(a) and 4(b).

No significant chargeability values were obtained with the battery equipment which effectively tested the first 100 feet below ground surface. The same result was obtained on the first separation of the second survey however, the north I.P. line indicates a very weak anomaly on the last two separations in the vicinity of 12+00w. Testing of this anomaly is warranted only with further geophysical, geological or geochemical support. The source of this anomaly is interpreted to be at a depth

greater than 200 feet. On the southern I.P. line a change in the apparent per cent frequency effect is noted on the 2nd to 5th separations inclusive. An increase in apparent resistivity accompanies the change suggesting a rock contact or possibly an increase in the depth of overburden to the east. The surface projection of this transition is at 12+00W on the southern I.P. line.

GEOCHEMICAL SURVEY

A total of 26 soil samples were collected at 200 foot intervals on traverse lines #1 and #2. The sample sites were numbered consecutively beginning with 720LS-1 to 720LS-26 and the station positions have been noted along the bottom of the geophysical profiles in Figures 3(a) and 3(b).

The samples were analyzed in AMAX's Burnaby laboratory for Mo, Cu, Ni, Co, Mn, Fe%, Ag, Zn and Pb. The analytical results and procedures are shown in Appendix II.

Accepting a regional threshold of 50 ppm Cu, no anomalous copper values were obtained.

G.M. DePaoli, B.Sc.

Allan, P.Eng. (B.C.

July 1973

	APPENDIX I	STATEMENT OF COS	<u>TS</u>	
	Record Number	Claim Number	Due Date	Group
	63675-63690 incl.	WA 116 incl.	July 22,1973	Red
	Period of Work - Octo May	ber 3, 1972 to Oc 25, 1973 to May 3		
1972	•	uced Polarization	Survey - 5000	feet
1973	Summary of Work - Line	chemical Survey e Cutting uced Polarization	- 1.1	line miles
	Personnel			
	G.M. DePaoli - 601-53 Geophysicist	Oct. 3-5/72	and May 28-30/	73
-	G.M. Leary - 601-535 Geologist	Oct. 3-5/72	ancouver 5, B.C	•
	Nick Sworyk - Box 235 Labourer	Oct. 3&4/72	· -	174.00
	Allan Jones - General Labourer	Oct. 3/72	Hache, B.C.	52.98
	F.J. Ferguson - 601-53 Geological Technicia	an May 25&26/7	, Vancouver 5, 3	25.00 B.C.
	T.E. Gilchrist - 7210 Jr. Assistant	May 25,26,2	rth Delta, B.C. 8,29/73	86.00
	D.R. Morrison - Box 41		Ontario	68.40
	IP Contractor Marcel Arsenault - Box IP Helper	2 days @ 220 k 28, R.R. #3 Abra 2 days	·	440.00 E.I.
	Board - 22 man days @	9 \$10.00/day		220.00
	Vehicle	7 days @ \$10	0.00/day	70.00
	Induced Polarization F			
	Scintrex IPR-7 Recei October 3-5/72	iver and IPC 25-Wa 3 days @ \$40		120.00

Declared before me at the Leff

Manuscon, in the

since of British Columbia, this 3/0 A Commissioner for taking Affidavits within British Columbia or A Notary Public in and for the Province of British Columbia. Sub mining Recorder

Magnetometer Rental

Scintrex MF-2 October 3-5/73

3 days @ \$10.00/day

30,00

Geochemical Analyses

26 samples for Mo, Cu, Ni, Co, Mn, Fe, Ag, Zn, Pb, and pH every 4th sample @ \$3.00/sample

78.00

Report Preparation and Drafting

200.00

\$1,888.38

legaleed K Doyg

The work is to be applied for one year on WA 1-16 incl

Declared/before me at the

Province of British Columbia, this

day of

A Commissioner for taking Affidavits within British Columbia or A Notary Publicin and for the Frovince of British Columbia;

Sub mining Recorder

APPENDIX II

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INDUCED POLARIZATION SURVEYS

AMAX EXPLORATIONS INC.

601-535 THURLOW STREET,

VANCOUVER, BRITISH COLUMBIA.

In account with D.F. MORRISON, BOX 418, GRAVENHURST, ONT.

TO I. P. SURVEY:

W.A. CLAIMS.

DATE MAY 28429, 1973

6A OPERATING DAYS.

6B STANDBY AND TRAVEL DAYS.

NUMBER OF OPERATING DAYS 2

@ \$220.00 per day =

NUMBER OF TRAVEL AND

STANDBY DAYS O @ \$110.00 per day =

SUB TOTAL OF BASIC FEES

7A EXPENSES RECEIPTS ATTACHED

Plus 10% Overhead

TOTAL EXPENSES

7B SALARIES RECEIPTS ATTACHED

SUBTOTAL OF EXPENSES PLUS 20% Overhead

AMOUNT DUE AND PAYABLE P.O. BOX 418, GRAVENHURST, ONT.

	APPROVE			_DATE>	
	Project	Exp.	Code	UOMA	11
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PHONE 705-687-2009

Procedures for Collection and Processing of Geochemical Samples

Analytical Methods for Ag, Mo, Cu, Pb, Zn, Fe, Mn, Ni, Co and W in sediments and soils; Mo, Cu, Zn, Ni and SO4 in waters.

Amax Exploration, Inc. Vancouver Office.

September 1970

R.F. Horsnail

SAMPLE COLLECTION

Soils

B horizon material is sampled and thus organic rich topsoil and leached upper subsoil are avoided. Occasionally organic rich samples have to be taken in swampy depressions.

Samples are taken by hand from a small excavation made with a cast iron mattock. Approximately 200 gms of finer grained material is taken and placed in a numbered, high wetstrength, Kraft paper bag. The bags are closed by folding and do not have metal tabs.

Observations as to the nature of the sample and the environment of the sample site are made in the field.

Drainage Sediments

Active sediments are taken by hand from tributary drainages which are generally of five square miles catchment or less. Composite samples are taken of the finest material available from as near as possible to the centre of the drainage channel thus avoiding collapsed banks. More than one sample is taken if marked mineralogical or textural segregation of the sediments is evident.

Some 200 gm of finer material is collected unless the sediment is unusually coarse in which case the weight is increased to 1 kg. Samples are placed in the same type of Kraft paper bag as are employed in soil sampling. Water samples are taken at all appropriate sites. Approximately 100 mls are sampled and placed in a clean, screw sealed, polythene bottle. Observations are made at each site regarding the environment and nature of the sample.

Rock Chips

Composite rock chip samples generally consist of some ten small fragments broken from unweathered outcrop with a steel hammer. Each fragment weighs some 50 gms. Samples are placed in strong polythene bags and sealed with non-contaminating wire tabs. Samples are restricted to a single rock type and obvious mineralization is avoided.

Soil, sediment and rock samples are packed securely in cardboard boxes or canvas sacks and dispatched by road or air to the AMAX geochemical laboratory in Vancouver.

SAMPLE PREPARATION

Packages of samples are opened as soon as they arrive at the laboratory and the bags placed in numerical sequence in an electrically heated sample drier (maximum temperature 70°C).

After drying soil and sediment samples they are lightly pounded with a wooden block to break up aggregates of fine particles and are then passed through a 35 mesh stainless steel sieve. The coarse material is discarded and the minus 35 mesh fraction replaced in the original bag providing that this is undamaged and not excessively dirty.

Rock samples are exposed to the air until the outside surfaces are dry; only if abnormally wet are rocks placed in the sample drier. Rock samples are processed in such manner that a fully representative $\frac{1}{2}$ g sample can be obtained for analysis. The entire amount of each sample is passed through a jaw crusher and thus reduced to fragments of 2 mm size or less. A minimum of 1 kg is then passed through a pulverized with plates set such that 95% of the product will pass through a 100 mesh

screen. Where samples are appreciably heavier than 2 kg the material is split after jaw crushing by means of a Jones splitter. After pulverizing the sample is mixed by rolling on paper and is then placed in a Kraft paper bag.

SAMPLE DIGESTION

Digestion tubes (100 x 16 mm) are marked at the 5 ml level with a diamond pencil. Tubes are cleaned with hot water and concentrated HCL. 0.5 g samples are weighed accurately, using a Fisher Dial-O-Gram balance, and placed in the appropriate tubes.

To each of the samples thus prepared are added 2 ml of an acid mixture comprising 15% nitric and 85% perchloric acids. Racks of tubes are then placed on an electrical hot plate, brought to a gentle boil $(\frac{1}{2} \text{ hour})$ and digested for $4\frac{1}{2}$ hours. Samples unusually rich in organic material are first burned in a porcelain crucible heated by a bunsen burner before the acid mixture is added. Digestion is performed in a stainless steel fume hood.

After digestion tubes are removed from the hot plate and the volume is brought up to 5 ml with deionized water. The tubes are shaken to mix the solution and then centrifuged for one minute. The resulting clear upper layer is used for Cu, Mo, Pb, Zn, Ag, Fe, Mn, Ni and Co determination by a Perkin-Elmer 290B atomic absorption spectrophotometer. Analytical procedures are given on the following pages.

ANALYTICAL PROCEDURES

Silver

- 1. <u>Scope</u> This procedure covers a range of silver in the sample from less than .5 to 1000 ppm
- 2. Summary of Method The sample is treated with nitric and perchloric acid mixture to oxidize organics and sulphides. The silver then is present as perchlorate in aqueous solution. The concentration is determined by atomic absorption spectrophotometer
- 3. <u>Interferences</u> Silver below 1 gamma/ml is not very stable in solution. Maintaining the solution in 20% perchloric prevents silver being absorbed on the glass container. Determination must be completed on the same day as the digestion.

Samples high in dissolved solids, especially calcium, cause high background absorbance. This background absorbance must be corrected using an adjacent Ag line.

Silver AA Settings P.E. 290

Lamp - Aq

Current 4 ma position 3

Slit 7 A

Wavelength 3281A Dial 287.4

Fuel - acetylene - flow - 14

Oxidant - air - flow - 14

Burner - techtron AB 51 in line

Maximum Conc. 3 to 4x

Calibration

Standards

- Set 1 gamma/ml to read 40 equivalent to 20 gamma/gm
 Factor ½ x meter reading
 Check standards
 - 4, 10, 20, 40 ppm Ag in sample
- Set 15 gamma/ml to 100 equivalent to 100 ppm
 Check standards
 40, 100 ppm
 Factor directly in ppm Ag
- 3. Rotate burner to maximum angle

 Set 10.0 gamma/ml Ag to read 100

 Check standards

 100,200,400,1000 ppm Ag

 Factor 10x scale reading
- 4. Samples higher than 1000 ppm should be re-analyzed by assay procedure
- 5. Background correction for sample reading between 1 to 5 ppm
 Calibrate AA in step 1
 Dial wavelength to 300 (peak)
 Read the samples again
 Subtract the background reading from the first reading
- 1. 1000 gamma/ml Ag 0.720 gm Ag_2SO_4 dissolved in 20 mls $Hx1O_3$ and dilute to 500 mls
- 2. 100 gamma/ml Ag 10 mls of above + 20 mls HClO₄, dilute to 100 mls

3. Recovery spiked standard

5 gamma/ml Ag - 5 mls 100 gamma/ml dilute to 100 mls with "mixed" acid

Working AA Standards

Pipette .2, .5, 1, 2, 5, 10 mls of 100 gamma/ml and 2, 5 mls 1.000 gamma/ml dilute to 100 mls with 20% HClO₄. This equivalent to 4, 10, 20, 40, 100, 200, 400, and 1000 ppm Ag in the sample .50 gm diluted to 10 mls.

Recovery Standard

Pipette 2 mls of 5 gamma/ml Ag in mix acids into a sample and carry through the digestion. This should give a reading of 20 ppm Ag + original sample content.

Follow the general geochemical procedure for sample preparation and digestion.

For low assay Ag, the same procedure is used. Ag is then calculated in oz/ton.

1 ppm = .0292 oz/ton conversion factor oz/ton = .0292 x ppm Ag

Zn Geochemical AA Setting

Lamp Zn

Current 8 #3 Slit 20A

Wave length 2133 Dial 84.9

Fuel - Acetylene Flow 14

Oxidant - Air Flow 14

Burner - P.E. short path 90°

Range

0 - 20 gamma/ml Factor 4x - 0 to 400 ppm

0 - 50 gamma/ml Factor 10x -0 to 1000 ppm

For Waters - Burner AB- 51 in line 1 gamma/ml read 100 to give 0 to 1000 ppb

High Zn Burner Boling in line. Wavelength 3075. Dial 250 Slit 7A
Fuel 14 Air 14.5

0 to 1000 gamma/ml read 0 to 20 Factor 400 \times

Pure Standard 10,000 gamma/ml

1 gm Zn dissolved, $\rm H_2O$, $\rm HCl$, $\rm HNO_3$, $\rm HClO_4$, fumed to $\rm HClO_4$ - make up to 100 mls $\rm H_2O$

1000, 100 gamma/ml and 100 ml by dilution in 20 % HClO₄
0 to 200 gamma/ml Zn use combined Cu, Ni, Co, Pb, Zn standards
Pipette

1, 2, 3, 5, 8, 10 mls of 10,000 gamma/ml - dilute to 100 mls with 20% HClO₄ to give

100, 200, 300, 500, 800, 1000 gamma/ml Zn for high standards

Co Geochemical AA Setting

Lamp - 5 multi element

Current 10 #4 Slit 2A

Wavelength 2407 Dial 133.1

Fuel - Acetylene Flow 14

Oxidant - Air Flow 14

Burner - AB 51 in line

Range

- 0 10 gamma/ml read 100 Factor 2 x reading to 200 ppm
- 0 20 gamma ml read 100 Factor 4 x reading to 400 ppm

 Burner at maximum angle
 - 0 100 gamma/ml read 100 Factor 20 x reading to 2000 ppm
- 0 200 gamma/ml read 100 Factor 40 x reading to 4000 ppm Standards - 1000 gamma/ml
 - 1.000 gm cobalt metal dissolved in HCl, HNO, and fumed into \mbox{HClO}_4 , dilute to 1 liter

Pipette :

1, 2, 10, 20 mls into 100 ml vol flasks diluted to mark with 20% HClO_4

This gives

10, 20, 100, 200 gamma/ml Co

Mixed - combination standards of Cu, Ni, Co, Pb, Zn

of

1, 2, 5, 10, 20, 30, 50, 30, 100, 150, 200 gamma/ml are used for calibration

Mn Geochemical AA Setting

Lamp Multi element Ca, Ni, Co, Mn Cr

Current 10 #4 Slit 7A

Wave length 4030.8 Dial 425.2

Fuel - Acetylene Flow 14.0

Oxidant - Air Flow 14.0

Burner - P.E. short path (or AB 50)

Range

- 0 100 gamma/ml Factor 20x 0 to 2000 ppm
- 0 200 gamma/ml Factor 40x 0 to 4000 ppm Burner 90°
 - 0 1000 gamma/ml Factor 200x 0 to 20,000 ppm
 - 0 2000 gamma/ml Factor 400x 0 to 40,000 ppm

EDTA Extraction - use AB 51 in line

0 - 20 gamma/ml Factor 4x - 0 to 400 ppm Standards

Fisher 10,000 gamma/ml (ml)

10x Dilution 1000 gamma/ml

Pippette

- .5, 1, 2, 3, 5, 8, 10, ml of 1000 gamma/ml
- 2, 3, 5, 8, 10, 15, 20 ml of 10,000 gamma/ml dilute to 100 mls with 20% HClO₄. This gives
 - 5, 10, 20, 30, 50, 80, 100, 200, 300, 500, 300, 1000, 1500, 2000 gamma/ml

Mo Geochemical AA Setting

Lamp ASL H/C Mo

Current 5 #5 Slit 7A

Wavelength 3133 Dial 260.2

Fuel - Acetylene Flow 12.0 to give 1" red feather

Oxidant - Nitrous oxide Flow 14.0

Burner - AB 50 in line

Caution read the operation using N_2O and acetylene flame at end of general AA procedure

Range

0 - 10 gamma/ml Factor 2x - 0 to 200 ppm Rotate burner to max. angle

0 - 50 gamma/ml Factor 10×0 to 1000 ppm

0 - 100 gamma/ml Factor 20 x 0 to 2000 ppm

Standards 1000 gamma/ml

Dissolve .750 gms MoO3 (acid molybdic) with 20 mls $\rm H_2O$, 6 lumps NaCH, when all dissolved, add 20 mls HCl, dilute to 500 mls 100 gamma/ml - 10 x dilution

Pipette

.2, .5, 1, 2, 3, 5, 8, 10 mls of 100 gamma/ml

2, 3, 5, 8, 10 mls of 1000 gamma/ml add 5 mls 10% AlCl3 and dilute to 100 mls with 20% $HClO_{\Delta}$

This gives

.2, .5, 1, 2, 3, 5, 8, 10, 20, 30, 50, 80, 100 gamma/ml Mo

Fe Geochemical AA Setting

Lamp - Fe

- Do not use multi element Fe

Current 10 #4 Slit 2A

Wavelength 3440.6 Dial 317.5

Fuel - Acetylene Flow 14:0

Oxidant - Air Flow 14.0

Burner - PE Short Path 90°

Range

0 - 5000 gamma/ml $0.1 \times \% - 0 \text{ to } 10.0\%$

 $0 - 10,000 \text{ gamma/ml} \quad 0.2 \times \% - 0 \text{ to } 20.0\%$

Higher Fe - 10 x dilution

Standards 10,000 gamma/ml

Weigh 5.000 gms iron wires, into beaker, add $\rm H_2O$, $\rm HCl$, $\rm HNO_3$, $\rm HClO_4$, heat to $\rm HClO_4$ fumes. Add $\rm HClO_4$ to 100 mls + 100 mls $\rm H_2O$, warm, dilute to 500 mls

Pipette

1, 5, 10, 20, 30, 50, 80 mls 10,000 gamma/ml dilute to 100 mls with 20% HClO4 to give

100, 500, 1000, 2000, 3000, 5000, 8000 gamma/ml to be equivalent to .2, 1.0, 2.0, 4.0, 6.0, 10.0%, 16.0% Fe in geochem sample

Ni Geochemical AA Setting

Lamp P.E. H/C. Ni or multi element Cu, Ni, Co, Mn, Cr

Current 10 #4, Slit 2A

Wave length 3415 Dial 312.5

Fule - Acetlylene Flow 14.0

Oxidant - Air Flow 14.0

Burner AB 51 in line

Range

0 - 20 gamma/ml Factor 4x - 0 - 400 ppm

0 - 100 gamma/ml Factor 20x - 0 - 2000 gamma

 45° 0 - 200 gamma/ml Factor 40x - 0 - 4000 ppm

0 - 500 gamma/ml Factor 100x - 0 - 10,000 ppm

Ni in waters and very low ranges

Wave length 2320 Dial 113

Range 0 - 5 gamma/ml Factor 1x - 0 - 100 ppm

Standards 10,000 gamma/ml

1.000 gm pure Ni metal dissolved in HCl, HNO3, HClO $_4$ to perchloric fumes, dilute to 100 ml $_{\rm H2}{\rm O}$

1000 gamma/ml and 100 gamma/ml Successive lox dilutions in 20% HCl(

- 1, 2, 5, 8, 10 mls of 100 gamma/ml
- 2, 5, 8, 10 mls 1000 gamma/ml
- 2, 5, 8, 10 mls 10,000 gamma/ml dilute to 100 mls in 20% HClO4. This gives
- 1, 2, 5, 8, 10, 20, 50, 80, 100, 200, 500, 300, 1000 gamma/ml 1
 Combined Standards Cu, Ni, Co, Pb, Zn is used as a working
 standard

Cu Geochemical AA Setting

Lamp Single Cu or

5 multi element

Current 10 for multi element #4 Slit 7A

4 for single #3 Slit 7A

Wavelength 3247 Dial 280

Burner Techtron AB 51 (For Cu in natural waters)

P.E. Short Path (For geochem)

Fuel Acetylene Flow 14

Oxidant Air Flow 14

Range

- 0 5 gamma/ml Factor lx to 100 ppm (for low Cu)
 - 0 20 gamma/ml Factor 4x to 400 ppm

Burner 90°

0 - 200 gamma/ml Factor 40x to 4000 ppm

Wavelength 2492 Dial 147

Burner in line

Range

- 0 1000 gamma/ml Factor 200x to 20,000 ppm
- 0 2000 gamma/ml Factor 400x to 40,000 ppm

Higher range than 40,000 ppm requires 10x dilution

Standards

10,000 gamma/ml

1.000 gm metal powder, $\rm H_2O$, $\rm HCl$, $\rm HNO_3$ until dissolved, add $\rm HClO_4$, fume dilute to 100 mls

1000 gamma/ml 10x dilution above in 20% ${
m HClO}_4$

2000 gamma/ml 20 mls 10,000 gamma/ml - dilute to 100 mls in 20% HClO_{A}

100 gamma/ml 10x dilution 1000 gamma/ml dilute to 100 mls in 20% $HC10_4$

200 gamma/ml 10x dilution 2000 gamma/ml dilute to 100 mls in 20% $HC10_4$

Pipette

1, 2, 3, 5, 8, 10 mls 100 gamma/ml - dilute to 100 mls with 20% HClO₄ to give 1, 2, 3, 5, 8, 10 gamma/ml

Combined standards Cu, Ni, Co, Pb, Zn

1, 2, 5, 10, 20, 30, 50, 80, 100, 150, 200 gamma/ml

Pb Geochemical AA Setting

Lamp ASL H/c Pb

Current 5 ma Slit 7A

Wave length 2833 Dial 208

Fuel - acetylene Flow 14

Oxidant - air Flow 14

Burner AB 51 in line

Range

- 0 20 gamma/ml to read 0 to 30. Factor 5x 0 to 500 ppm
- 0 200 gamma/ml to read 0 to 80. Factor 50x 0 to 5000 ppm Standards 10,000 gamma/ml
- 1.000 pure metal, dissolved in HNO3, fumed to HClO4 make up to 100 mls in 20% HClO4

1000 gamma/ml and 100 gamma/ml Successive 10x dilutions in 20% HClO4

Pipette

- 1, 2, 5, 8, 10 mls 100 gamma/ml
- 2, 5, 8, 10, 20 mls 1000 gamma/ml dilute to 100 mls in 20% $HClO_4$ this gives
- 1, 2, 5, 8, 10, 20, 50, 80, 100, 200 gamma/ml
 Combined Standards Cu, Ni, Co, Pb, Zn, are used as working
 standards

W in Soils and Silts

Reagents and apparatus

Test tubes - pyrex disposable

Test tubes - screw cap

Bunsen Burner

Flux - 5 parts Na₂CO₃

4 parts NaCl

1 part KNO3 pulverized to -80 mesh

7% SnCl₂ in 70% HCl

20% KSCN in H2O

Extractant - 1 part tri-n-butyl phosphate

9 parts carbon tetrachloride

Standards

1000 gamma/ml W

.18 gms Na_2WO_4 $2H_2O$ dissolved in H_2O , make up to 100 mls

100 gamma/ml, 10 gamma/ml by dilution

Standardization -

Pipette .5, 1, 2, 3, 5, 8, 10 ml of 10 gamma/ml

and 1.5, 2 mls of 100 gamma/ml - dilute to 10 mls

continue from step #4

Artificial colors - Nabob pure Lemon Extract, dilute with 1:1 ethanol and water to match. Tightly seal these for permanent standards

Procedure

1. Weigh 1.0 gram sample, add 2 gm flux, mix

- Sinter in rotary for 2 to 3 minutes (Flux dull read for one minute)
- 3. Cool, add 10 mls $\rm H_2O$, heat in sand bath to boiling, cool, let sit overnight
- 4. Stir, crush, and mix. Let settle
- 5. Take 2 ml aliquot into screw cap test tube
- 6. Add 7 mls SnCl2, heat in hot water bath for 5 minutes (80°C)
- 7. Cool to less than 15°C
- 8. Add 1 ml 20% KSCN, mix (if lemmon yellow; compare color standard 10x)
- 9. Add ½ ml extractant, cap, shake vigorously 1 minute
- 10. Compare color

Molybdenum in Water Samples

- 1. Transfer 50 mls to 125 separatory funnel
- 2. Add 5 ml .2% ferric chloride in conc HCl
- 3. Add 5 mls of mixed KSCN and SnCl2
- 4. Add 1.2 mls isopropyl ether, shake for 1 minute, and allow phases to separate
- 5. Drain off water
- 6. Compare the color of extractant

Standardization

Pipette 0, .2, .5, 1, 2, 3, 4, 5, mls of l gamma/ml and l, l.5, 2, mls of 10 gamma/ml dilute to 50 mls with demineralized $\rm H_2O$, and continue step #2.

This equivalent to

 $SnCl_2 - 15\%$ in 15% HCl

300 gm $SnCl_2$. $2H_2O$ + 300 mls HCl, until $SnCl_2$ dissolved dilute to 2 liters

KSCN - 5% in H_2O

Mixed SnCl2 - KSCN

3 parts SnCl2 to 2 parts KSCN

Water Samples Run for AA

- 1. Cu 2 gamma/ml reads 80 scale therefore 1 unit = 25 ppb
 - 2. Zn 1 gamma/ml reads full scale therefore 1 unit = 10 ppb
 - 3. Ni 2.5 gamma/ml reads 50 scale therefore 1 unit = 50 ppb

Burner: long slot techtron burner in line

Sulphate in Natural Waters

- 1. Pipette 0.5 ml sulphate reagent mix into a colorimetric tube
- 2. Add 5 ml water sample and mix
- 3. Read at 343 Muagainst a demineralized water blank
- 4. Read again at 400 muand subtract from sulphate reading
- 5. Calculate ppm sulphate from the graph

Reagent

Dissolve 54 grams red mercuric oxide (J.T. Baker 2620- Can Lab) in 185 ml 70% perchloric acid and 20 ml H₂O, shake for one hour. Add 46.3 grams ferric perchlorate I Fe(ClO₄)₃ . 6H₂O I (GFS 39) and 47 grams aluminum perchlorate I Al (ClO₄)₃ . 8H₂O I (GFS 2) Add 400 ml water to dissolve, let settle overnight, decant into bottle and make to 1 liter

PH MEASUREMEN'S

Soil and drainage sediment samples are dampened with water in a glass beaker to a pasty consistency. Demineralized water is used for this purpose as it has a low buffer capacity and thus does not influence the pH of the sample. Measurement is made with a Fisher Acumet pH meter. Electrodes are stored in buffer overnight. A 30 minute warm up time is allowed for the instrument each morning. A 10 ml aliquot is taken from water samples for pH measurement.

