1971 Geological and Geochemical Report

3/89

TITLE

-..AUTHORS

DATE

COMMODITY

LOCATION - Area
Mining Division
Coordinates
NTS

Jura Station - Rok Claims

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August 1971

Cu-Mo

Princeton, British Columbia Similkameen 120°27'W longitude, 49°34'N latitude 92 H

AMAX Vancouver Office

Department of

Mines and Petrolaum Resources

ASSESSMENT REPORT

NO 3/89

MAG

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SUMMARY

Geological mapping and geochemical sampling were conducted on the 68 Rok claims situated seven miles north of Princeton, British Columbia.

Geologically the property lies in the so-called "Princeton-Aspen Grove Copper Belt", an area underlain by Triassic Nicola volcanic rocks comprised of a complex sequence of flows, pyroclastics, volcanic breccia and minor limestone that have been intruded by alkalic stocks. On the Rok property a zoned diorite sill is surrounded and intruded by rocks of the Okanagan batholith.

Pyrite, with rare molybdenite, are the only sulphide minerals observed on the property, with pyrite invariably associated with magnetite.

Geochemical soil sampling indicates that no anomalous areas of copper exist on the property.

CONCLUSION

The geological mapping and geochemical sampling leave little room for speculation on the presence of a viable orebody.

INTRODUCTION

The property was examined between May 24 and July 4, 1971 by a crew of 12 men under the supervision of R.L. Morton, geologist employed by Amax Exploration, Inc. During this period a picket line grid was cut, geological mapping was carried out at a scale of 1 inch = 400 feet, and a total of 790 soil geochemical samples were collected.

LOCATION AND ACCESS

The property is situated 120°27'W longitude and 49°34'N latitude about seven miles north of Princeton near the Jura rail—road station (Figure 1). Access is from Princeton via the Osprey Lake road, which is usuable year round. The property is covered by a network of old logging roads, many of which are suitable for four wheel drive vehicles.

PHYSICAL FEATURES

The claim group is situated in the relatively arid interior belt of British Columbia and consists, in the southeast, of rolling grasslands and open forest and is covered by a thick growth of pine, fir, spruce and poplar in the north and northwest. The property occupies the summit and east flank of a broad ridge that separates Summers and Hayes Creeks. Relief is about 2000 feet from Hayes Creek to the crest of the hill at 4500 feet. Annual precipitation, mostly in the form of snow during the winter months, is approximately twenty inches, summers are typically hot and dry.

Outcrop makes up 3% or less of the surface area, and overburden ranges from a few to several tens of feet thick.

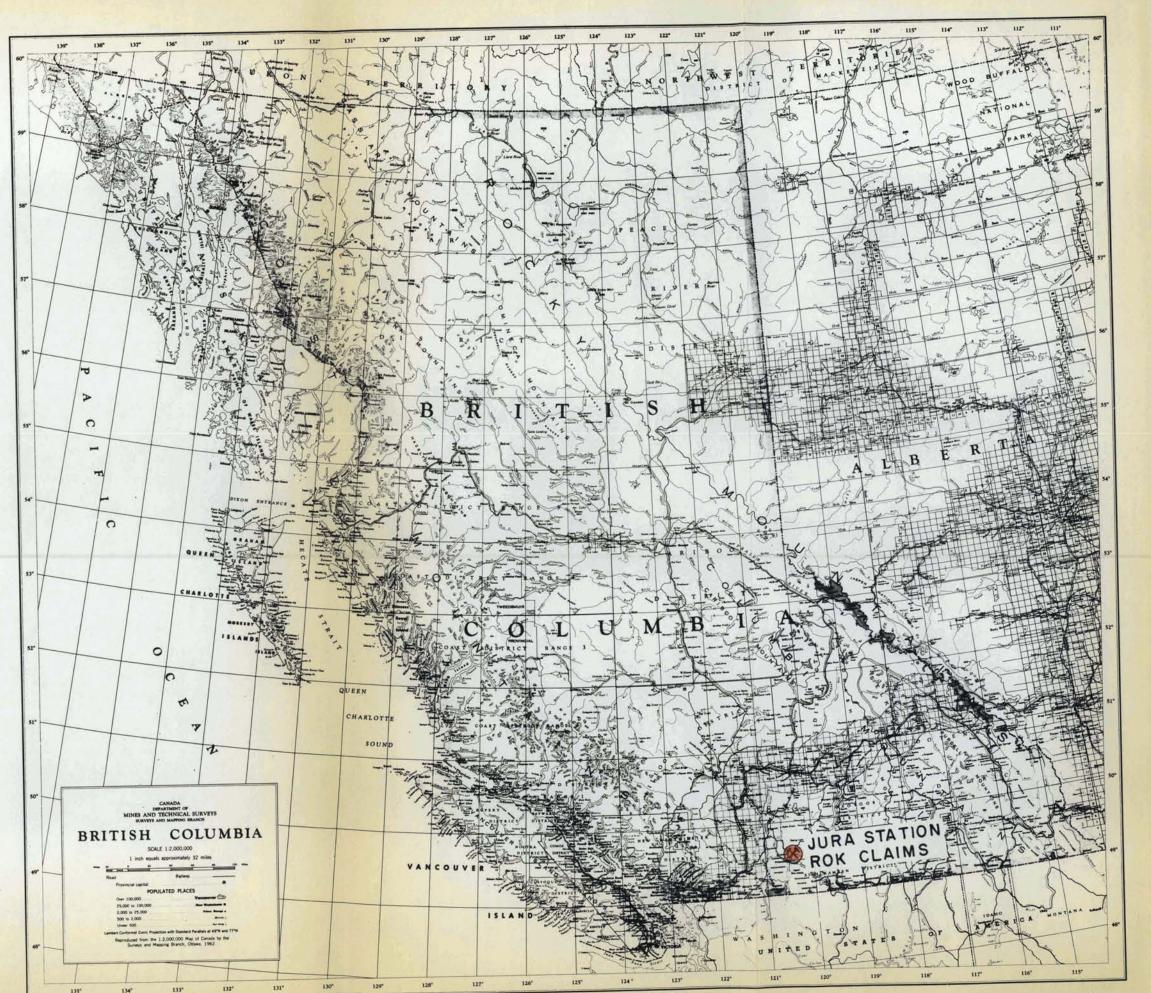
CLAIMS

The property consists of 68 claims (Figure 2) staked by AMAX in August, 1970 and these are listed below -

Claims Recording Date Record Numbers

Rok #1 - #68 inclusive August 25,1970 28104-28171 inclusive

Mines and Petroleum Resources
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NO. 3182 MAP



N.T.S. File: 92 H 9

LOCATION MAP

GEOLÓGY

General Statement

The property lies in the so-called "Princeton-Aspen Grove Copper Belt", an area underlain by Triassic Nicola volcanic rocks comprised of a complex sequence of flows, pyroclastics, volcanic breccia, and minor limestone that have been intruded by alkalic stocks. The eastern border of the belt is defined by Jurassic granitic rocks of the Okanagan batholith.

Local features characteristic of the Rok property are shown in Figure 2. A zoned sill 9600 by 2300 feet composed of microdiorite, hornblende-biotite diorite, and hornblende-magnetite-biotite diorite is surrounded and intruded by aplite, quartz monzonite, and porphyritic quartz monzonite of the Okanagan batholith. A narrow, westerly trending salient of the batholith extends along the western border of the claim group. Hornblendite and monzonite form narrow dykes in the sill, with dykes of microdiorite, quartz-feldspar porphyry, and aplite observed in the porphyritic quartz monzonite.

Sulphides consist of pyrite in the zoned sill, and minor molybdenite in the aplitic phase of the Okanagan batholith.

Lithology

Microdiorite (Unit 5, Figure 2) - forms the periphery of a zoned sill that trends northwest and occupies the western and southeastern part of the property. This unit has been intruded by a fine grained aplite and close to the contact small veins (.3-2mm in width) of aplite form a stockwork in the microdiorite. The microdiorite is a fine grained, equigranular rock consisting of subhedral hornblende (32%), euhedral-subhedral grey plagioclase (65%), and irregular, disseminated grains of magnetite (3%). The hornblende is partially altered to chlorite, magnetite to hematite, and plagioclase is replaced by cream coloured clay minerals. Minor amounts (up to 1%) of pyrite occur

as disseminated grains throughout the microdiorite. The contact between the aplite and microdiorite is sharp, and in places the microdiorite has been bleached.

Hornblende-Magnetite-Biotite Diorite (Unit 6) - forms a sharp, chilled, conformable contact with the microdiorite, and grades, over 25 to 50 feet, into a hornblende-biotite diorite. This unit varies from 200 to 1400 feet in width, and is the cause of the large magnetic anomaly that underlies the property. The hornblende-biotite-magnetite diorite is a porphyritic rock, mottled black and white, that consists of prismatic, aligned hornblende phenocrysts (35%) set in a medium grained matrix of euhedral-subhedral plagioclase (50%), books of hexagonal biotite (5%), and irregular grains and plates of magnetite (10%). The rock is fresh in appearance and only slightly fractured with crystals of grey to pink zeolite coating the fractures. Pyrite is disseminated throughout the rock, but is mostly concentrated (2-3%) in the southeastern part of the sill, associated with magnetite.

The hornblende-magnetite-biotite diorite is fine grained adjacent to the contact with the microdiorite, and angular fragments (1 - 6 feet wide) of microdiorite were observed in Unit 6 up to 30 feet from the contact, and these are often hornfelsed.

Lineation of the hornblende varies, trending northeast along the northern and southern contacts with the microdiorite and northwest in the nose of the sill. This indicates that the lineation is a flow feature and that the hornblende-magnetite biotite diorite is younger and has intruded the microdiorite.

Hornblende-Biotite Diorite (Unit 7) - grades into the hornblende-magnetite-biotite diorite described above and forms the core of the sill. This medium grained, equigranular unit is mottled black and white and is composed of subhedral-anhedral, grey plagioclase (60%), prismatic, aligned hornblende (25%),

hexagonal books of black, lustrous biotite (10%), and disseminated grains of magnetite (5%). No sulphides were observed in this unit.

Angular fragments of monzonite occur in the hornblendebiotite diorite, and these are thought to be associated with or derived from, the Copex stock to the south.

Hornblendite (Unit 8) - occurs as a large inclusion within the Okanagan batholith, and as dykes that cut the zoned sill. The hornblendite dykes parallel the trend of the sill, dip 55 - 70° to the southeast, and vary from 2 - 100 feet in width. These dark, medium grained rocks consist of subhedral hornblende (60%), prismatic augite (10%), and dark grey, subhedral plagioclase (30%). They are non-magnetic and contain no visible sulphides. The large (600 by 200 feet) hornblendite inclusion forms a sharp contact with the porphyritic quartz monzonite, is elongate north-south, and is cut by numerous, fine grained aplitic dykes.

Aplite (Unit 12) - forms two separate, crescent-shaped bodies, of undetermined extent, that grade into the porphyritic quartz monzonite. This unit is similar to the quartz monzonite found along Summers Creek and appears to be a late phase of the Okanagan batholith. The quartz monzonite outcrops along the northern and southeastern borders of the claim group, and is cut by numerous, northwest trending aplite dykes. This medium grained rock is composed of 40% grey, subhedral plagioclase, 30% tabular pink feldspar, and 15% anhedral, clear quartz. Prismatic hornblende, books of biotite, and minor disseminated magnetite form the remainder of the rock. No sulphides were observed in this unit.

<u>Porphyritic Quartz Monzonite</u> (Unit 11) - underlies a large portion of the claim group and contains angular-subrounded inclusions of hornblende-biotite diorite and microdiorite. The

rock varies from medium grained to pegmatitic, is greyish pink, and consists of tabular perthite (10%) and subhedral quartz (10%) phenocrysts set in a matrix of subhedral, grey plagioclase (20%), anhedral pink feldspar (35%), anhedral, clear quartz (20%), often intergrown with pink feldspar, and books of biotite (4%). This unit contains no sulphides.

Dyke Rocks (Units 9, 12, 14, 15) - Monzonite (Unit 9) thought to be related to the Copex stock, and fine grained aplite form narrow dykes in the zoned sill; with microdiorite (Unit 14), quartz feldspar porphyry (Unit 15), and aplite (Unit 12) forming north-northwest trending dykes in the porphyritic quartz monzonite. No sulphides were observed in any of these dyke rocks.

ECONOMIC GEOLOGY

Pyrite, with rare molybdenite, are the only sulphide minerals observed on the property. Pyrite is associated with large amounts of magnetite and is concentrated in Unit 6.

Molybdenite occurs as rare flakes in the aplitic border phase of the Okanagan batholith.

GEOCHEMISTRY

A total of 790 soil samples were collected within the claim area. The methods of collecting and analyzing these samples are given in Appendix II. Analyses were carried out at the Amax Geochemical Laboratory in Burnaby, B.C., with Cu, Mo, Ni, Pb, Zn, Mn, Ag, and Fe content determined for all samples. Sample locations were spaced at 400 foot intervals, along lines generally 400 feet apart. Analytical values were plotted on a distribution curve and were colour coded as follows:

Soils - Green 0 - 50 ppm Cu background Orange 51 - 100 ppm Cu positive Red +101 ppm Cu anomalous

Copper - out of the 790 samples collected only four contained anomalous values in copper and three of these are due to local high background in the aplitic border phase of the Okanagan batholith. Only one anomaly occurred over the zoned diorite sill (144 ppm) and its cause has not yet been ascertained.

<u>Molybdenum</u> - molybdenum is present in only trace amounts on the property and most samples with significant molybdenum content are from the aplitic phase of the Okanagan batholith suggesting local high background.

'AMAX Vancouver Office

R.L. Morton

C.J. Hodgson, P. Eng. (B.C.)

August 1971

APPENDIX I - ASSESSMENT DATA

Claims - ROK #1 to #68 inclusive

Record Numbers 28104 to 28171 inclusive

Period of Work - May 24 to July 4, 1971

Summary of Work

Geological Mapping - 4 square miles Geochemical Sampling - 4 square miles Line Cutting - 4 line miles Geochemical Analyses - 790 samples

Personnel Involved

R.L. Morton	-601-535 Thurlow Street, Vancouver	•						
	Staff Geologist; 18 days @ \$47.00	\$ 846.00						
D. Colley	-4359 Harder Road, Victoria							
	Junior Assistant; 24 days @ \$19.66	471.84						
M. Legros	-16 Lake St, Huntington, Quebec							
	Junior Assistant: 15 days @ \$16.24	243.60						
A. Sparham	-3255 W 10th Ave., Vancouver							
D 11-1-	Junior Assistant; 10 days @ \$19.66	196.66						
R. Hale	-General Delivery, Merritt	035 45						
D. Wheeler	Junior Assistant; 14 days @ \$15.39	215.46						
D. Wheeler	-General Delivery, Keremeos	60.45						
T. Underwood	Junior Assistant; 5 days @ \$13.69 -Box 150, Montrose	68.45						
1. Onact wood	Junior Assistant; 1 day @ \$20.52	20.52						
P. Marshall	-305 Wharncliffe Rd., London, Ontario	20.52						
I, IIII DIIII I	Senior Assistant: 8 days @ \$25.65	205.20						
D. Simon	-16223 NE 27 St., Bellevue, Wash.	203.20						
	Senior Assistant; 6 days @ \$20.52	123.12						
F. Ferguson	-1395 Cornwall St., N. Kamloops	120.12						
	Senior Assistant; 3 days @ \$41.00	123.00						
G.M. DePaoli	-5442 Inman Avenue, Burnaby	220.00						
,	Geophysict; 7 days @ \$50.00	350.00						
P. Stocker	-4753 Capilano Road, N. Vancouver							
	Senior Assistant; l day @ \$44.00	44.00						
•	_							
Board - 112 mai	n days @ \$5.00/day	560.00						
<u>Transportation</u> - 2 four wheel drive vehicles								
	30 days @ \$10.00	300.00						
Geochemical Sample Analyses								
790 samples for	r Cu, Mo, Ni, Pb, Zn, Mn, Ag, and Fe							
_	@ \$3.00/sample	2,370.00						
	_							

Work to be applied for one year on ROK #1-#12 inclusive and ROK #21-#68 inclusive

CJ. Hodgen

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

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 ($\frac{1}{2}$ 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. Scope 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 - Ag

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

- 1. Set 1 gamma/ml to read 40 equivalent to 20 gamma/gm Factor ½ x meter reading Check standards
- 2. Set 15 gamma/ml to 100 equivalent to 100 ppm Check standards

4, 10, 20, 40 ppm Ag in sample

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

Standards

- 1. 1000 gamma/ml Ag 0.720 gm Ag₂SO₄ dissolved in 20 mls $Hx10_3$ 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, H_2O , HCl, HNO_3 , $HClO_4$, fumed to $HClO_4$ - make up to 100 mls 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, 80, 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, 800, 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 \times 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% AlCl $_3$ and dilute to 100 mls with 20% HClO $_4$

This gives

.2, .5, 1, 2, 3, 5, 8, 10, 20, 30, 50, 80, 100 gamma/nul 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 x % - 0 to 10.0%

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

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% HC104 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

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SITE SY Current 10

Wave length 3415 Dial 312.5

Fule - Acetlylene Flow 14.0

Flow 14.0 Oxidant - Air

Burner AB 51 in line

квиде

0 - 20 damma/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

Wi in waters and very low ranges

Wave length 2320 Dial 118

gange 0 - 5 gamma/ml Factor lx - 0 - 100 ppm

Standards 10,000 gamma/ml

1.000 gm pure Ni metal dissolved in HCl, HNO3, HClO4 to

perchloric fumes, dilute to 100 ml H20

1000 gamma/ml and 100 gamma/ml Successive lox dilutions in 20% HClo.

in 2, 5, 8, 10 mis of 100 gamma/mi

2, 5, 8, 10 mis 1000 gamma/ml

2, 5, 8, 10 mls 10,000 gamma/ml - dilute to 100 mls in 20%

This gives HCIO4.

I' S' 2' 8' 10' 50' 20' 80' 100' 500' 200' 800' 1000 demme/mJ M:

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, H_2O , HCl, HNO_3 until dissolved, add $HClO_4$, fume dilute to lOO mls

1000 gamma/ml 10x dilution above in 20% HClO₄

2000 gamma/ml 20 mls 10,000 gamma/ml - dilute to 100 mls in 20% $HC10_4$

100 gamma/ml 10x dilution 1000 gamma/ml dilute to 100 mls in 20% HClO₄

200 gamma/ml 10x dilution 2000 gamma/ml dilute to 100 mls in 20% HClO₄

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 80. 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

- 2. 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 m1 20% KSCN, mix (if lemmon yellow; compare color standard 10x)
- 9. Add $\frac{1}{2}$ 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 1 gamma/ml and 1, 1.5, 2, mls of 10 gamma/ml dilute to 50 mls with demineralized H_2O , and continue step #2.

This equivalent to

1, 4, 10, 20, 40, 60, 80, 100, 200, 300, 400 ppb Mo Artificial color - Nabob orange extract dilute with 1:1 $\rm H_2O$ to methanol to match. Seal tightly

SnCl₂ - 15% in 15% HCl

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

KSCN - 5% in H2O

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 MEASUREMENTS

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.

