1981 Geological & Geochemical Assessment Report

TITLE

Blue Sheep Lake Property

CLAIMS

Sky 1-5 (67 units)

COMMODITY

W, Mo

LOCATED

160 kilometres south of Watson Lake, Y.T. Latitude 58045' Longitude 128018'

Liard Mining Division 104 I 9, 16

BY

W.H. Bentkowski and A.C. Hitchins

FOR

AMAX of Canada Limited

WORK PERIOD

August, 1981

AMAX VANCOUVER OFFICE

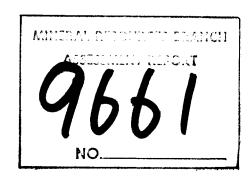


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RECOMMENDATIONS

- a) Trenching and diamond drilling to test the strike and dip extensions of the mineralized skarn.
- b) Detailed mapping and additional sampling over the breccia zone north of Blue Sheep Lake.

INTRODUCTION

The Blue Sheep Lake tungsten property is located (Figure 1) in the southern Cassiar Mountains (104 I 9, 16) approximately 110 kilometres, southeast of the townsite of Cassiar, B.C. The property is comprised of the Sky 1-5 claims (67 units) within the Liard Mining Division.

Access to the property is by helicopter from Watson Lake, 160 kilometres to the north, or from Dease Lake, 110 kilometres to the west southwest. Blue Sheep Lake is amenable to small float planes. An old cat trail from Dease Lake and running along the Turnagain River passes approximately 13 kilometres south of the property.

Geological mapping at a scale of 1:10,000 was conducted over approximately 36 of the 67 claim units. An area 200 metres by 380 metres over the main showing was mapped at 1:1,000 scale.

Twenty-one silt, thirty-one rock chip and approximately three hundred and eighty soil samples were collected on the Sky claims.

Previous Work

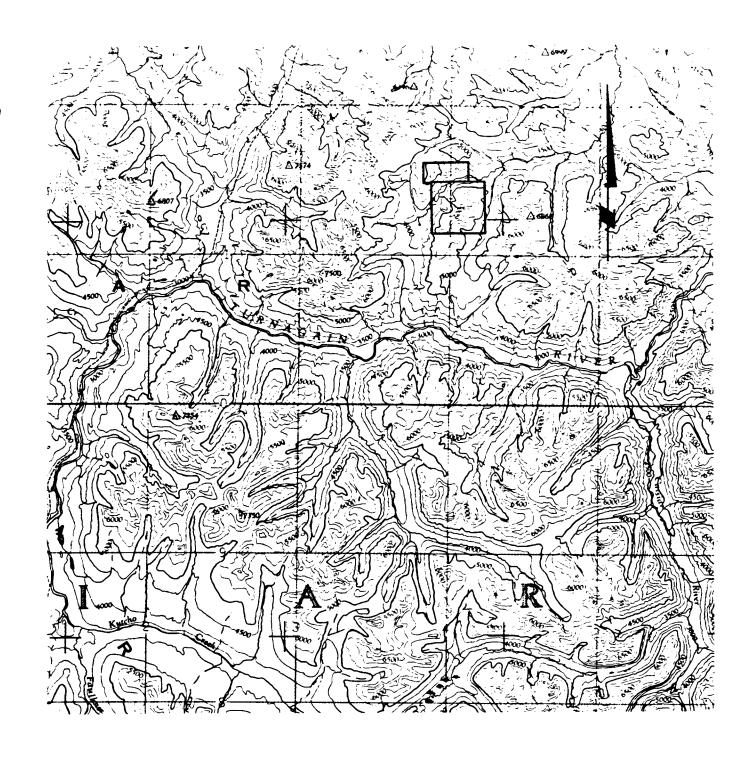
The property is located on the Cry Lake map sheet which has been mapped by H. Gabrielse for the G.S.C. in the 1950's and again in the late 1970's (map 29-1962 and O.F. 610).

Past work in the area includes trenching and drilling on a kaolinized granite cut by sphalerite-galena veins (Herb claims) 13 kilometres southeast of the Sky claims and work by several operators including Union Carbide, on the Ram-Ewe scheelite prospect 16 kilometres to the southeast.

Previous work on the ground covered by the Sky claims was restricted to EM-16 and magnetometer surveys over Pb-Zn-Ag-Cu mineralization in pyrrhotite float at a felsic porphyry-limestone contact. The surveys outlined several magnetic conductors (BCDM Assessment Report 3539) but the claims were dropped before further testing was carried out.

Physiography

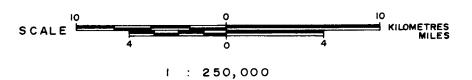
Most of the property lies above tree line, with thick bush toward the main valley. Smooth, rolling topography characterizes the south end of the northerly-trending ridge. To the north, the elevation reaches 2,100 metres at the head of a rugged cirque.



AMAX OF CANADA LIMITED

BLUE SHEEP LAKE PROPERTY SKY CLAIMS LIARD MINING DIVISION — BRITISH COLUMBIA

LOCATION MAP



CLAIMS

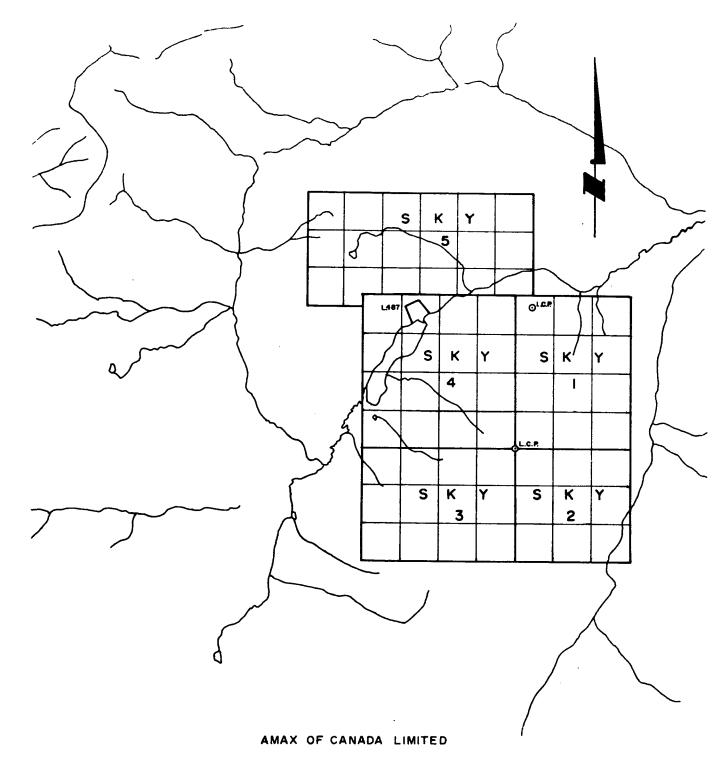
A total of 67 mgs. units in five adjoining claims (Sky 1-5) are located in the Liard Mining Division of northern British Columbia (Figure 2). Forty-nine units were staked in August, 1980 and an additional 18 units were staked in July, 1981. Sky 1-4 claims were recorded on August 11, 1980 and the Sky 5 claim was recorded on July 30, 1981.

Claim Name	Record No.	Expiry Date	
Sky 1 (12 units)	1525	August 11, 1981	
Sky 2 (9 units)	1526	August 11, 1981	
Sky 3 (12 units)	1527	August 11, 1981	
Sky 4 (16 units)	1528	August 11, 1981	
Sky 5 (18 units)	1972	July 30, 1982	

REGIONAL GEOLOGY

Folded and faulted northwest striking Precambrian to Silurian clastic and carbonate rocks are exposed in several mountain ranges east of the northwest trending Cassiar Batholith.

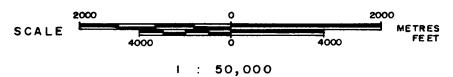
An elliptical lobe of the Cassiar Batholith quartz monzonite intrudes Hadrynian clastic and carbonate units and Cambrian to Silurian shales about five kilometres southeast of the property.



BLUE SHEEP LAKE PROPERTY

SKY CLAIMS
LIARD MINING DIVISION - BRITISH COLUMBIA

CLAIM MAP



PROPERTY GEOLOGY

The claims are underlain by southerly dipping (20-50°) Atan carbonate and overlying Kechika phyllites which have been intruded by a small Cretaceous(?) quartz-feldspar porphyry stock and associated dykes and sills. Intrusion of the stock has produced a local doming of the sediments and an extensive zone of hornfels within the phyllites. Low sulphide garnet-diopside skarn and overlying cherty, light green to brown hornfels are exposed at the dolomite-phyllite contact immediately west of the stock. This favourable contact is talus covered east of the intrusion but cherty and phyllitic hornfels are exposed above the talus slope.

Numerous kaolinized and manganese stained leucocratic quartz-feldspar porphyry dykes and felsic sills associated with the stock are exposed in the carbonate and hornfels units on the northern portion of the property. North of Blue Sheep Lake a zone of brecciated unaltered phyllite and altered dolomite has been intruded by fine grained felsic dykes.

Tertiary alkaline basalt dykes cut both the hornfels and carbonate units but are most common within the hornfelsed sediments.

Brief descriptions of the unaltered carbonates and phyllites and their altered equivalents follows in Appendix I.

MINERALIZATION

Mineralization present on the property includes scheelite, molybdenite, pyrite, pyrrhotite, galena and sphalerite, chalcopyrite, powellite(?), arsenopyrite and rhodochrosite. Galena-sphalerite and pyrrhotite occur in diopsidequartz veins within dolomite and in discontinuous pods with pyrite and chalcopyrite along the phyllite-dolomite contact. Pyrrhotite is common throughout all the rock units but is most abundant within the phyllitic and cherty hornfels as disseminations, veinlets and massive pods. Chalcopyrite blebs and veinlets are commonly associated with the massive pyrrhotite pods and are also locally present within the dolomite and cherty hornfels. Massive arsenopyrite was observed in float with pyrite, galena-sphalerite and quartz. Rhodochrosite occurs in vertical, black weathering carbonate veins with minor disseminated sphalerite, chalcopyrite, arsenopyrite and galena.

Economic interest in the property is centered on scheelite, powellite and molybdenite within the light green skarn, garnet skarn and magnetite skarns. Scheelite occurs as blue and yellow fluorescing, coarse, crystals up to two centimetres long in the magnetite skarn and fine grained to powdery disseminations in the light green and garnet skarns. Coarse scheelite crystals are zoned with a yellow fluorescent core and blue fluorescent rim. The magnetite skarns are narrow pipe like or lenticular pods probably parallel to bedding with little economic potential. Scheelite and powellite in the skarns is concentrated in a zone 200 metres long by up to 35 metres wide.

Molybdenite occurs as disseminations and streaks within the light green and brown cherty hornfels and flakes along narrow fractures in the hornfels. Traces of disseminated

molybdenite are also present in the light green skarn and garnet skarn. Estimated grades of approximately 0.5% WO $_3$ + MoS $_2$ over five metre thickness occur in the light green skarn.

GEOCHEMISTRY

Just over 400 soil and silt samples were collected from talus slopes and creek beds usually on a contour line around the mountain below the favourable dolomite-phyllite contact. A small soil grid was placed over the breccia zone north of Blue Sheep Lake.

Most of the samples were collected from the loose surface of talus fans at the base of scarps. Where soil was present the thin vegetation - humus layer was removed and unaltered mineral soil from a depth of 3-10 centimetres was collected. Generally soil profiles were very poorly developed.

Samples were placed in Kraft envelopes and analysed by Rossbacher Laboratory, Burnaby for W, Zn, Pb, Ag, Fe, Ni, Co, Mn and Mo.

Results for W, Zn, Pb and Ag are plotted on Figure 4 and analytical values for the other elements are presented in Appendix IV.

Dato

A.C. Hitchins

APPENDIX I

DESCRIPTION OF ROCK TYPES

Description of Rock Types

Atan Carbonates

Limestone and dolostone of the Cambrian Atan Formation are the oldest lithologies exposed on the property. Grey weathering, light grey, finely crystalline limestone is overlain and interbedded with pink to tan weathering dolostone. The weathered surface of the dolostone is usually pitted and jointing is common. Alteration within the dolostone, especially proximal to the porphyry stock includes local silicification and irregular pods and veins of yellow-green serpentine. Tremolite and actinolite have developed adjacent to siliceous pods and veins. In general, the limestone appears to be less altered than the dolostone.

Kechika Phyllites

The variably calcareous, black, unaltered phyllites, which overlie the Atan carbonates exhibit a well developed intersecting foliation and cleavage producing a bladed talus. Local rusty zones are present with only minor disseminated pyrite visible along cleavage planes. Irregular quartz carbonate veins up to two metres thick are common but do not contain sulphides.

Cretaceous Porphyry Stock

A weakly to intensely kaolinized quartz-feldspar porphyry stock intrudes the Atan carbonates and Kechika phyllites and is exposed over an area 400 by 500 metres on the northwest side of the mountain. Quartz phenocrysts vary in shape from rounded to euhedral doubly terminated. The generally euhedral kaolinized feldspars weather out producing a vuggy surface. Alteration colour varies from a chalky

yellow to manganese stained dark brown-black. The eastern contact is generally sharp with only minor dykes and sills. A zone of brecciated biotite hornfels is up to eight metres thick and with either a porphyry or carbonate matrix rims the stock's southern contact.

Cretaceous Dykes and Sills

Quartz-feldspar porphyry dykes similar in texture to the stock intrude both the hornfelsed phyllites and the dolomite. The dykes vary in thickness from 1-12 metres wide and contacts with the surrounding rocks are generally sharp. Phenocrysts within the porphyry dykes vary from rounded to well terminated quartz crystals up to one centimetre long, fresh to completely kaolinized feldspar up to two centimetres and muscovite plates probably altered from original biotite. The matrix is fine grained to aphanitic and locally exhibits flow banding along contacts. Iron oxides and carbonate are present in minor amounts. Manganese staining is often intense. Fine grained pyrite is a minor consituent of some dykes.

Several fine grained light coloured cherty rhyolite dykes and sills intrude the hornfels.

A zone of intense brecciation intruded by fine grained felsic dykes is exposed on a weakly rusty hump just north of Blue Sheep Lake. In contrast to intensely altered dark brown, altered dolostone fragments the phyllite fragments are unaltered, rarely displaying even a reaction run. Dolostone clasts are often mineralized with disseminated galena, sphalerite and pyrite. Felsic dyke fragments are scattered through the breccia but appear to be concentrated towards the base of exposure.

Tertiary Alkali Basalt Dykes

These mafic dykes crosscut the hornfels, limestone and dolostone parallel to northerly striking joints. The dykes are composed of fine grained pyroxene, biotite, feldspar and magnetite with quartz and calcite filled amygdules. No contact effects are observed.

Skarn and Hornfels

Intrusive activity has altered the dark grey Kechika phyllites through a brown phyllitic hornfels to a bleached or light green cherty hornfels. Light green skarn, garnet skarn and magnetite skarn developed along the cherty hornfels-dolomite contact. The bulk of the scheelite and molybdenite occur in the skarn and in the gradational contact between skarn and cherty hornfels.

Hornfelsed Phyllites

Weak hornfelsing of fine grained pelitic and calcareous sediments has given rise to laminated purple and greygreen calcareous phyllites which become less limy and phyllitic towards the stock. Thin veins of pyroxene-quartz-calcite with a bleached margin up to one centimetre wide crosscut the phyllitic hornfels. Intensity of veining increases toward the stock. Actinolite veins and beds up to five metres thick occur within the hornfels. Pyrrhotite is ubiquitous in the hornfels and occurs as massive pods and veins with traces of fine grained scheelite, as dissemination and blebs in concordant to subconcordant quartz veins and as discontinous laminae parallel to foliation. A prominent narrow rusty bed of the siliceous phyllitic hornfels occurs between the garnet skarn and cherty hornfels.

Cherty Hornfels

Light green to brown cherty hornfels is fine grained and laminated on a millimetre scale but breaks across banding, often with a concoidal fracture. Bands and veins of garnet and actinolite skarn occur within this unit and are most prominent proximal to the porphyry stock. Minor pyroxene veining is present, with abundant bleaching along joints. Pyrrhotite is common as dissemination within the hornfels and in association with garnet rich knots and lenses parallel to the foliation. Minor scheelite is present in narrow garnet skarn bands in the cherty hornfels immediately above the main skarn bands. Disseminations and streaks of molybdenum are common within the cherty hornfels.

Light Green Skarn

A very distinctive massive, buff to light green weathering skarn occurs above the dolomite west of the porphyry stock. This unit can be traced over a length of 320 metres with a maximum thickness of about 30 metres. It is very porous and vuggy and weathers to a distinctive a pock-marked surface. The skarn consists of fine grained crystalline calcite, diopside (?), actinolite and locally has diopside crystals up to five millimetres. Pods and veins of coarse garnet occur within this skarn and increase in abundance towards a gradational contact with the garnet skarn unit.

Tungsten is present as both coarse scheelite crystals and a fine-grained yellow fluorescent powellite coating. The coarse crystals are sometimes zoned with yellow fluorescent powellite cores and blue fluorescent scheelite rims. Minor disseminated molybdenum also occurs locally within the skarn.

Garnet Skarn

Red-brown garnet skarn gradationally overlies the light green skarn over a length of 320 metres and is up to 10 metres thick. The red-brown garnet crystals are medium to coarse-grained and occur as bands within a diopside-calcite-quartz skarn. Locally, garnet bands are vuggy with the development of garnet and calcite crystals within the cavities. Scheelite occurs as a fine yellow fluorescent powder and as medium to coarse grained crystals. Molybdenum is present as disseminations within interbedded light green cherty hornfels.

Magnetite Skarn

A fine to coarse crystalline massive magnetite skarn is exposed within the dolomite north of the skarn zone. It is subparallel to bedding and covers an area of approximately 10 x 2 metres. Coarse blue and yellow fluorescing scheelite is most abundant near the sharp magnetite-dolomite contact with the development of scattered calcite veins bordered by coarse crystalline magnetite. Finely interbedded magnetite-quartz float, devoid of scheelite has not been traced to outcrop.

A magnetite lens exposed in a gully immediately northwest of the stock contains no visible scheelite. APPENDIX II

STATEMENT OF COSTS

Statement of Costs

Work - Geological and Geochemical Survey -					
<u>Period</u> - July 10 - 29, 1981					
Personnel Employed -					
A.C. Hitchins-601-535 Thurlow Street, Vancouver, B.C. Staff Geologist; 14 days @ \$146.40/day	\$2,049.60				
W.H. Bentkowski-l Rockingham Court, London, Ontario Senior Assistant; 12 days @ \$67.06/day	804.72				
A.C. Smallwood-1054 Duchess Avenue, West Vancouver, B.C. Junior Assistant; 3 days @ \$63.12/day	189.36				
Kathleen Dorey-110 Lawrence Avenue, Cornwall, Ontario Junior Assistant; 4 days @ \$49.31	197.24				
Stewart McIlwraith-38 Rhydwen Avenue, Scarborough, Onta Junior Assistant; 4 days @ \$53.25	rio 213.00				
Board - 37 man days @ \$30/day	1,110.00				
Helicopter - 19.7 hours @ \$295/hr - Hughes 500 Fuel 433.4 gallon @ \$4.77/gal.	5,811.50 2,067.31				
Quasar Aviation LtdRichmond Inv.#81249					
Fixed Wing - Otter - 495 miles @ 2.56/miles B.C. Yukon Air Service-Inv.#2092, 2253	1,267.20				
Assays and Geochem - 13 Assays Mo & W					
394 soil and rock chip samples for six elements	1,554.80				
Rossbacher Laboratory, Burnaby, B.CInv.#1386					
Topographic Map - Pacific Survey Corporation, Vancouver 880.00					
Report Preparation and Drafting	500.00				
PAC Withdrawal	16,753.28 1,547.00				
	18,300.28				

³ years to Sky 1-4, 2 years to Sky 5

APPENDIX III

STATEMENT OF QUALIFICATIONS

NAME: A.C. Hitchins

ADDRESS: 601-535 Thurlow Street,

Vancouver, B.C. V6E 3L6

EDUCATION: University of Toronto - B.A. Sc. 1970

University of Toronto - M.Sc. 1973

EXPERIENCE: AMAX of Canada Limited - Staff Geologist - 1972 to present

NAME

A.C. Smallwood

ADDRESS

7580 Burris Street

Burnaby, B.C.

EDUCATION

3rd year standing at Simon Fraser University

EXPERIENCE

1977 Newmont Mining - Field Assistant

1978 ANAX Minerals - Field Assistant

1979 AMAX Minerals - Field Assistant

1980 AMAX of Canada Limited - Field Assistant

NAME

S. McIlwraith

ADDRESS

38 Rhydwen Avenue, Scarborough, Ontario

M1N 2E1

EDUCATION

University of Toronto - Arts & Science - Geology -

1979 to present

EXPERIENCE

Birchmount Park Science Department - Senior Lab.

Assistant - 1974 to 1978

Ministry of Natural Resources - Junior Ranger Program -

1977

Derry, Michener and Booth (Mining Consultants) - Junior

Assistant Geologist - 1979

Golden Eagle Oil and Gas Ltd. - Junior Assistant

Geologist - 1980

AMAX of Canada Limited - Field Assistant - 1981

NAME

K. Dorey

ADDRESS

110 Lawrence Avenue,

Cornwall, Ontario

K6H 4H3

EDUCATION

Completed 2nd year of four year B.Sc. geological

geophysics

EXPERIENCE

1980 - summer research assistant with E. McClelland-Brown (University of Leeds, postdoctorate)

core sampling Huronian 1981 - AMAX of Canada Limited - Field Assistant

NAME

W.H. Bentkowski

ADDRESS

1 Rockingham Court,

London, Ontario

N6C 4L7

EDUCATION

University of Western Ontario - Science - Geology

1978 to present

University of Western Ontario - Science - General 1977 to 1978

Catholic Central High School - 1972 to 1977

EXPERIENCE

Shell Canada Resources - 1979 - Junior Geological

Assistant

Texasgulf Inc. - 1980 - Geological Assistant

AMAX of Canada Limited - 1981 - Geological Assistant

APPENDIX IV

ANALYTICAL METHODS

Kossbacher Laboratory

GEOCHEMICAL ANALYSTS & ASSAYERS

CANADA
TELEPHONE 299 6910
AREA CODE 604

April 30, 1974

SUMMARY OF SOME ANALYTICAL TECHNIOUFS CURRENTLY IN USE AT ROSSBACHER LABORATORY

A ANALYTICAL TECHNIQUES FOR GEOCHEMICAL SAMPLES

SIMPLE 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 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 pulverizer with plates set such that 95% of the product will pass through a 100 mesh

Rock Chine

composite rock chin samples conversity consist of some ten small fragments broken from unweathered outcrop with a steel hammer. Each fragment weighs some 50 cms. 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.

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.

Septorber 1970

STUPE S COLLECTION

Soil-

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 syampy deprecians.

Samples are taken by hand from a small excevation made with a cast iron mattock. Approximately 200 gms of Energrained 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 sale 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 scaled, polythone bottle. Observations are made at each site recarding the environment and nature of the sample.

<u>Calibration</u>

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

2. 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

- Samples higher than 1000 ppm should be re-analyzed by assay procedure
- 5. Background correction for sample reading between 1 to 5 ppm $\mbox{\footnote{A}}$ 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 Ag_2SO_4 dissolved in 20 mls $Hx10_3$ and dilute to 500 mls
- 2. 150 gamma/ml Ag 10 mls of above + 20 mls HClO4, dilute to
 100 mls

3. Recovery spiked standard

5 gamma/ml Ag = 5 mls lú0 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 1.00 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 g 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.

'l ppm = .0292 oz/ton

conversion factor

 $oz/ton = .0292 \times ppm Ag$

serior. From naturity are appropriately married tranching the material is matrix after jaw or ching by means of a Jones uplitter. After privarising the sample is mixed by rolling on paper and in them placed in a Kraft paper bag.

SIMILE DIC CURRON

Discretion types (100 x 10 mm) are marked at the 5 ml level with a Marcad pencil. Types are cleaned with hot water and concentrated Hol. 0.5 g samples are weighed accurately, using a F ster Dial-C-Graw balance, and placed in the appropriate types.

To each of the camples this propared are added 2 ml of an acid mixture comprising 15% nitric and 15% perchloric apids. Naths of these are then placed on an electrical hot plate, brought to a gentle boil (! hour) and digested for 4½ hours. Samples unusually rich in organic material are first larged in a percelain cricible heated by a bunsen burner before the acid mixture is added. Digestion is performed in a stainless steel fine bood.

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 Ct. Mo. Pt. Zh. Ag. Fe. Mn. Ni and Co determination by a Perkin-Richt 2 GB atomic absorption spectrophotometer. Analytical procedures and given on the following pages.

ANALYTICAL PROCEDURES

Silver

- Scope This procedure covers a range of silver in the sample from less than .5 to 1000 ppm
- 2. <u>Summary of Method</u> 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 3231A Dial 287.4

Fuel - acetylene - flow - 14

Oxidant - air - flow - 14

Burner - techtron AB 51 in line

Maximum Conc. 3 to 4x

Zn Goodhemical 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 x

Pure Standard 10,000 gamma/ml

1 gm Zn dissolved, $\rm H_2O$, $\rm HC1$, $\rm HNO_3$, $\rm HClO_4$, 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 Plow 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 funed into \mbox{HClO}_4 , dilute to l liter

Pipette

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

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

Ma 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, 3, 10, 15, 20 ml of 10,000 gamma/ml dilute to 100 mls with 20% $HClO_4$. This gives
- 5, 10, 20, 30, 50, 80, 100, 200, 300, 500, 300, 1000, 1500, 2000 gamma/ml

Mo Goothemical 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 x 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 5.0 mls 100 gamma/ml - 10 x dilution

Pipette

- .2, .5, 1, 2, 3, 5, 8, 10 mls of 100 ganma/ml
- 2, 3, 5, 9, 10 mls of 1000 genuma/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/ml Mo

Fc 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/nd 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 160 mls with 20% HClO4 to give

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

Ni Goochemical 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 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 $\rm H_2O$

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

1, 2, 5, 8, 10 mls of 100 ganma/ml

2, 5, 9, 10 mls 1000 gamma/ml

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, 860, 1000 grama/ml 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 1x 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% 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% $HClO_{\Delta}$

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

Pipette

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

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

Combined standards Cu, Ni, Co, Pb, Zn

Ph 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 ${\rm HNO_3}$, fumed to ${\rm HClO_4}$ make up to 100 mls in 20% ${\rm HClO_4}$

1,000 gamma/ml and 100 gamma/ml. Successive 10x dilutions in 20%~HC104

Pipette

- 1, 2, 5, 3, 10 mls 100 ganuna/ml
- 2, 5, 3, 10, 20 mls 1000 gamma/ml dilute to 100 mls in 20% $HClO_4$ this gives
- 1, 2, 5, 8, 10, 20, 50, 30, 100, 200 gamma/m.l
 Combined Standards Cu, Ni, Co, Pb, Zn, are used as working standards

W in Soils and Silts

Reagents and apparatus

Test tubes - pvrex disposable

Test tubes - screw cap

Bunsen Burner

Flux - 5 parts Na₂CO₂

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
- Add 1 ml 20% KSCN, mix (if lemmon yellow; compare color standard 10x)
- 9. Add 1 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 demineral_zec $\rm H_2O$, as continue step #2.

T is equivalent to

1, 4, 10, 20, 40, 60, 30, 100, 200, 300, 400 ppb Mo $\label{eq:hammon} \text{Artificial color} - \text{Nabob orange extract dilute with 1:1 H_2O to methanol to match. Seal tightly}$

SnCl2 - 15% in 15% HCl

300 gm SnCl₂ . 2H₂O + 300 mls HCl, until SnCl₂ 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

xxi

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 400m/wand subtract from sulphate reading
- 5. Calculate ppm sulphate from the graph

Reagent

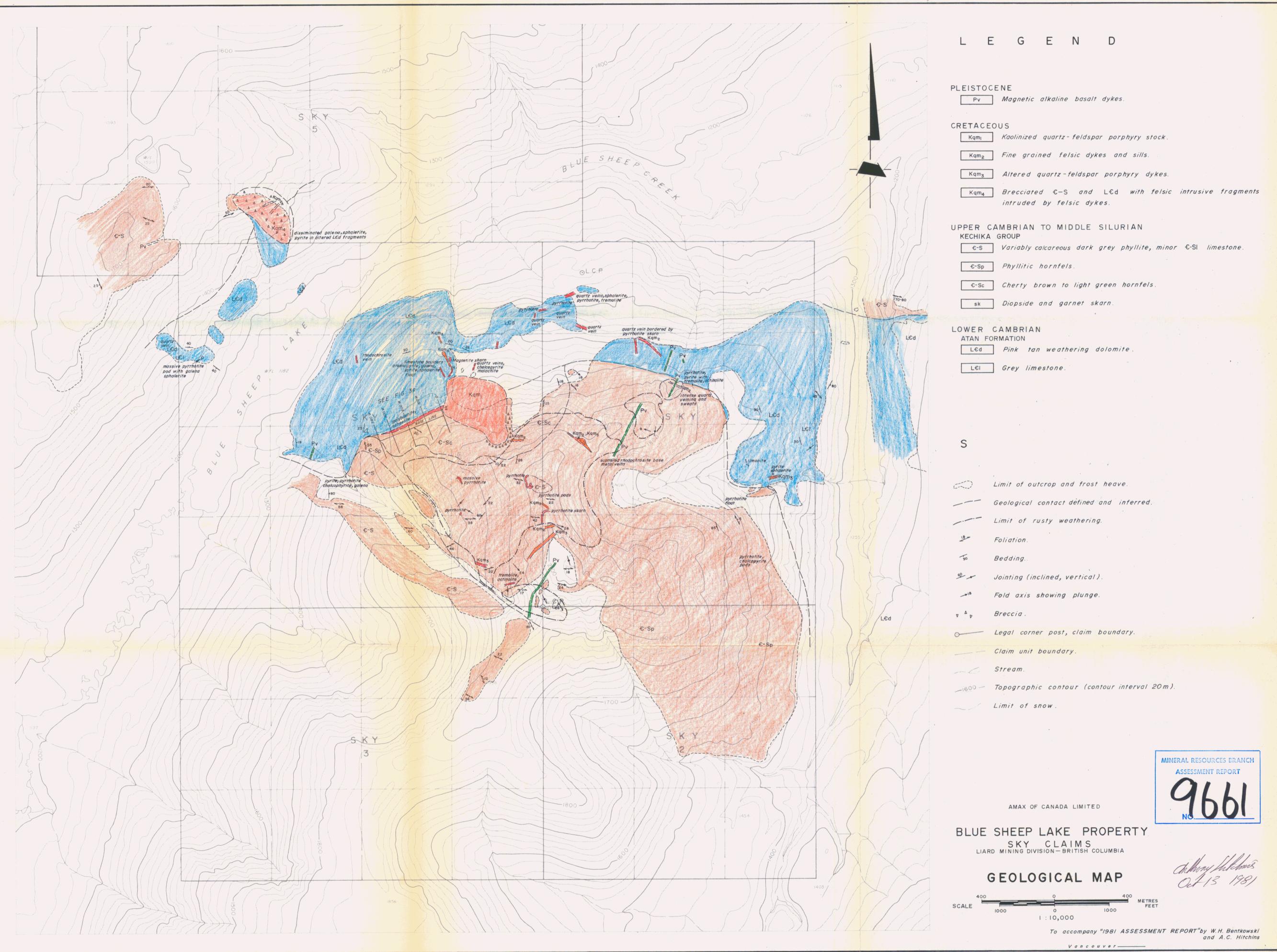
Dissolve 54 grams red mercuric oxide (J.T. Baker 2620- Can Lab) in 185 ml 70% perchloric acid and 20 ml $\rm H_2O$, shake for one hour. Add 46.3 grams ferric perchlorate $\rm II Fe(ClO_4)_3$. $\rm 6H_2OI$ (GFS 39) and 47 grams aluminum perchlorate $\rm II Al$ (ClO4) $\rm _3$. $\rm 3H_2OI$ (GFS 2) Add 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 Acument 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.

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N.T.S. Ref. 104 | 9 and 16 FIG. 3

