

# ENVIRONMENTAL LABORATORY QUALITY ASSURANCE / QUALITY CONTROL PLAN

WATER LICENCE N7L2-0925



**ECHO BAY MINES LTD.**

LUPIN OPERATIONS

CONTWOYTO LAKE, NWT

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## INTRODUCTION

Quality Assurance is an integral component of laboratory management. Establishing and maintaining a sound quality assurance program is a definite necessity. The lab must assure that data generated is of known, proven, and defensible quality. For the purpose of this plan, Quality Assurance (QA) can be recognized as the system of activities implemented to ensure that the quality control job is done effectively.

Quality Control (QC) can be described as the use of established procedures to achieve a standard of measurement encompassing the two principals of quality;

- Accuracy, defined as the measurement of how closely a value approximates a standard or true value.
- Precision, defined as a measure of the closeness or repeatability of a set of values.

Lupin's Environmental lab has established a set of operating principles that constitutes the quality assurance program. The QA Plan includes the following;

- sample control and documentation
- standard operating procedures for analytical methods
- equipment preventative maintenance
- calibration procedures
- internal quality control activities
- performance audits
- data assessment procedures
- data validation and reporting

As always, within any process, improvements can be made to the system which will make it more efficient and thorough. The text that follows lists the procedures and methods incorporated by the Environmental lab at Lupin and comprises the basis of the QA Plan. Routine review and revision will ensure that the Plan maintains its' goal of Quality Assurance in the laboratory.

# 1 QUALITY ASSURANCE

## 1.1 SAMPLE COLLECTION

### 1.1.1 TYPE

Grab sampling is used for both static and kinetic water sample collection. Manual techniques are used for all sampling without the aid of automatic sampling devices. Van Dorn samplers are used for collection on lakes and ponds. Separate samplers are used on tailings ponds and lakes to avoid contamination.

### 1.1.2 LOCATION

Sample stations located around the Lupin mine site are designated within the Surveillance Network Program (SNP) annexed to water licence N7L2-0925, dated June 1, 1995. The SNP locations are indicated with signs for position consistency. At certain locations buoys and landmarks are utilized to locate sampling stations on tailings ponds and lakes respectively, while sign post positioning marks stream sample stations.

### 1.1.3 SAMPLING POINTS

Collection within lakes and ponds is at mid depth as well as surface when required. Samples are occasionally collected from various depths and isolated individually (grab samples). Depth profiles are conducted regularly to maintain consistency in locating the sample points.

Stream samples are sampled at a stream depth 0.6 of the depth of the stream (just over half the depth). In cases where shallow conditions are encountered, a smaller bottle is used to collect the sample.

### 1.1.4 SAMPLING METHODS

Lakes and ponds; Once at a sample station, the Van Dorn sampler is set to accept the sample. It is lowered to mid depth (predetermined by depth profile). The messenger is released to close the sampler and the sample is retrieved to surface where contents are emptied from the sampler.

Repeat collection but wash sample container using two rinses and collect the sample on the third fill. Leave approximately 2% of sample container capacity to provide for mixing, preservative addition, and thermal expansion. Add appropriate preservative immediately after collection and mix well.

Streams; Sampling is done manually in mid stream where water flows freely and is clear of debris. After calculating the mid-stream depth, wait until any stirred up material from the stream bed has cleared. Plunge bottle upstream with mouth facing downstream at 0.6 of stream depth. Turn bottle mouth toward the current and allow bottle to fill. If stream depth is insufficient for collection into main sample bottle, a smaller capacity (100 mL) bottle can be used. Using the same procedure, fill the smaller vessel several times and transfer until the main bottle is full. Again, a minimum of three rinses per container with the sample is required before the final collection. Bottles are filled to near capacity to allow room for preservative addition & mixing.

#### **1.1.5 SAMPLING FREQUENCY**

The times at which samples are collected is extremely important. Collection frequency is again, outlined within the SNP. The Lupin Environmental Lab personnel collect additional samples routinely at various times and locations to gain further information regarding current conditions within the system (ie. within the mill circuit, tailings ponds).

#### **1.1.6 FIELD BLANKS**

Field blanks of ultra pure water are included with every sample set collected. Treat these "samples" in exactly the same manner as all other samples. The Blanks are taken into the field and handled and preserved as part of the sampling program. Using blanks will give an indication of possible sample contamination and general sample integrity.

#### **1.1.7 REPLICATE SAMPLES**

During each sampling campaign, one sample point is chosen for the collection of replicate samples.

This involves sampling that station in triplicate and individually analyzing each sample which will provide "representation" for that sample and sampling precision. Collection of samples in triplicate has been performed only at Station 925-22. Additional replicates will be included where and when it is deemed necessary (ie. weekly at 925-10 and monthly at 925-14 during time of discharge, as they are two sampling locations of prime importance).

## 1.2 SAMPLE HANDLING

### 1.2.1 CONTAINERS

All samples are collected in containers constructed of high density polyethylene (HPDE) plastic (see Table I). New bottles are preferred but sample containers may be used repeatedly with proper handling measures. Use of new high density polyethylene bottles will be incorporated more often. High priority samples (high priority meaning SNP samples or samples of very low concentration) are to be collected in new bottles.

A system has been created for logging the utilization of bottles. A rotational scheme for bottle usage is as follows;

- (1) Use new bottles for blank controls, Sun Bay samples, and sampling of other "natural" water bodies. These bottles are marked in a manner so they can be used over for the same sampling location. After three uses, bottles are moved to a lower designation as in (2) below.
- (2) Bottles retired from a (1) designation are used for monitoring of Tailings ponds, Sewage lagoons, Stations 925-10 and 925-20. Again, it is necessary to assign a bottle to a fixed sampling location and use it for that location only.

Three uses at this designation and bottles can be withdrawn for use at the final designation.

- (3) Bottles retired from a (2) designation are to be used in the sampling of Cells within the Tailings Containment Area (TCA) and Mill circuit locations. Bottles which are; utilized for containment of highly contaminated material, visibly fouled, or suspect in any way shall be disposed of in a proper manner. Cleaning processes will not be applied and these bottles will not be re-used. To ensure that these bottles do not re-enter the "circuit", all bottles will be marked with a permanent, smear proof pen to ease in the identification of locations and designation.

Analysis of each sample set will include a cleaning check. The cleaning check will be administered for (i) trace metals, (ii) total arsenic, (iii) total cyanide analyses;

- (i) Trace metals - A blank sample of ultrapure water is contained in a "clean" sample bottle with a nitric acid addition (< ph of 2). The bottle is allowed to "soak" for at least 48 hours prior to analysis. Water within the sample bottle is analyzed with the sample set by the approved method.
- (ii) Total arsenic - A blank sample of ultrapure water is contained in a "clean" sample bottle. The bottle is allowed to sit for at least 48 hours prior to analysis. Water within the container is then analyzed with the sample set by the approved method.
- (iii) Total cyanide - A blank sample of ultrapure water is contained in a "clean" sample bottle with a sodium hydroxide addition (> ph of 11). The bottle is allowed to "soak" for at least 48 hours prior to analysis.



Water within the container is then analyzed with the sample set by the approved method.

Any contaminants present after cleaning can be detected, providing the analyst with confidence or suspect in the cleaning technique. If the contaminant being determined produces unusually high concentrations when compared to a blank control contained within a (1) designated bottle, then further examination with the cleaning procedures is justified. The set of bottles in question can be re-checked, designated to a lower level, or discarded if need be.

#### **1.2.2 SAMPLE PRESERVATION**

Methods of preservation are limited and are intended to retard biological action and hydrolysis, reduce volatility and reduce absorption effects. Preserve upon collection with appropriate preservative and amounts (see Table I on Page 15).

#### **1.2.3 SAMPLE IDENTIFICATION**

Every sample collected is recorded and as mentioned, identified by marking bottles with water resistant, non-smear felt pen. Record sufficient information to provide positive sample identification even by other persons not involved in the sampling. Include the following: name of sample collector, date, time, exact location/station, and other data such as weather conditions, water level, stream flow, etc.

#### **1.2.4 TRANSPORTATION/RECEIVING**

Ensure sample integrity from time of collection to data reporting. After labelling, seal sample and store upright in box with other samples to provide a snug, non moving storage space during transfer. Deliver to the lab as soon as possible and log all samples in a designated log book. Assignment of sample log number and required analyses can be carried out once in the laboratory.

### 1.3 INSTRUMENT PERFORMANCE

This section briefly discusses certain aspects of the instruments and equipment used in Lupin's Environmental Lab. Standard operational procedures for each particular component within the laboratory will not be discussed. Emphasis will be placed on calibration guidelines and maintenance control to assure adequate performance for water quality measurement.

#### 1.3.1 INSTRUMENTATION/CALIBRATION

Calibration is part of the analytical method. The need for accurate calibration of equipment is crucial for the detection of instrument malfunctions or degradation that may directly affect the quality of results. There are several variables that justify the frequency of calibration for instruments and equipment. (ie. amount of use, sensitivity, importance of the sample results, environment that it is used in).

Calibration schedules are followed in the laboratory. Results are documented to develop instrumental integrity and to enable the monitoring of trends in data. (Table II on Page 16) provides a basic summary of calibration guidelines in place at the Lupin Env. Laboratory.

NOTE : Full procedures, methods, and techniques for each equipment calibration are not listed. They would be far too comprehensive for this text and are detailed in their individual operation manuals.

#### 1.3.2 CALIBRATION WITH STANDARDS

In general, 6-8 standard dilutions are prepared containing the actual species to be measured covering the widest possible concentration range of interest. Verify the standard curve by analyzing one or more of the standards within the linear range. Reportable results are within the range of the standard dilutions used. The slope is a measure of the sensitivity of the method. Again, verify the calibration prior to each analysis and check regularly with a standard and blank during each sample set.

In addition to the verification of calibration with prepared standards and blanks, a specialized certified reference material is utilized.

The manufacturing quality and standard of certified references is more exacting and confidence limits are higher for these materials. Standard solutions prepared "In lab" are within + or - 1 % of their stated value (ie. Fischer Scientific Arsenic reference solution is at 1000 ppm + or - 1 % of certified value). The certified values of externally manufactured quality control standards are equal to 100 % of the parameter in the indicated standard.

### 1.3.3 MAINTENANCE

Preventative maintenance is practised in the Lupin Environmental Lab. The laboratory itself is isolated in a separate area of the main mill complex and is provided with separate ventilation. Delicate and sensitive instruments are kept and used in rooms set aside solely for their purpose (ie. atomic absorption unit). Components that need frequent replacement or items that require regular servicing are kept in stock. As an alternative to following a highly expensive maintenance schedule provided by the instruments manufacturer (and due to the remote location of the lab), a simpler, more common sense approach for maintaining equipment is achieved by applying the following:

- Monitor trends in data that might suggest instrument degradation or malfunction.
- Frequent calibration and repeat analysis will also give an indication of the instrument's integrity.
- Routine maintenance operations are carried out by Lupin's instrumentation and electrical departments.
- If a problem is not repairable on site, the instrument is sent out to the manufacturer's service department.
- In cases where instrument shipping is very difficult (ie. atomic absorption unit) or the instrument is needed immediately, a certified instrument technician is flown in to the site for maintenance endeavours.

All repairs and problems encountered are documented and recorded in a log book.

Other important factors which play a direct role in preventive maintenance are keeping a very clean work environment for the analytical instrument and ensuring proper training and instruction for the correct operation of each particular instrument.

#### 1.3.4 GLASSWARE

The use of Class A volumetric glassware constructed of borosilicate glass can deliver accuracy of 0.1 - 0.2%. The cleaning of most lab glassware is now done by the aid of a lab dishwasher unit. It produces extremely high temperature washes with tap water and ultrapure water rinses. Specialized cleaning agents specifically designed for lab ware are used. Test tubes, vials, pipettes, burettes, and graduated cylinders are not cleaned via the lab washer. They are emersed in a solution of synthetic detergent, scrubbed with a brush, and then put through several tap and Ultrapure™ water rinses. The individual glassware just mentioned, when used in the analysis of specialized parameters (ie. trace metals) must be cleaned in a more thorough manner. An "aqua regia" solution consisting of three parts concentrated HCl to one part concentrated HNO<sub>3</sub> diluted 50-50 with de-ionized water is excellent. The glassware is soaked in this solution for at least an hour followed by six rinses of both hot tap water and de-ionized distilled water. Again, cleaning checks are administered for glassware the same as they are for plastic sample containers as in Section 1.2.1 in this plan. All glassware should be kept separate and isolated for its segregated use only. Also, all solutions should be measured at room temperature (20°C), as glassware was calibrated by the manufacturer at this temperature. Periodic calibration checks of new glassware with water using a calibrated analytical balance is considered good practice. Any discrepancy with the manufacturer's specifications should result in disposal or return of that glassware.

#### 1.4 ANALYSIS

Approved Analytical Methods: Echo Bay Mines Ltd. strictly adheres to the use of EPA approved methods and test procedures. The frequency of various analytical determinations is governed by the Surveillance Network Program (SNP). A list of approved test procedures for selected parameters at EBM Lupin is included (see Table III on Page 17).

#### 1.4.1 WATER SUPPLY

DISTILLED/DEIONIZED WATER SUPPLY: Reagent grade water is now being produced by a new Barnstead / Thermolyne distillation unit. Ultra pure water (Type 1) is produced with a Barnstead NANOpure II purification unit. The integrity of the water is checked in the analysis of every sample set by control blanks. Documentation for maintenance and cleaning schedules is kept via a log. Checks are a necessity because they provide a measure for inorganic and organic content. Inorganics are best detected by measuring electrical conductivity. Acceptable grade is  $2\mu\text{s}$  or less at  $25^{\circ}\text{C}$  with cleaning or regeneration of the system being required if excessive values result. For metals analysis, the water must be checked using AA spectroscopy. Even trace indications of metals such as copper, lead and zinc will require the replacement of research grade ion exchange resin. The presence of organics is determined by a permanganate test.

#### 1.4.2 REAGENTS/CHEMICALS

CHEMICAL REAGENTS: All standards and reagents are purchased from certified suppliers that meet the American Chemical Society "Analytical Reagent Grade" requirements. Contents, preparation date and expiry date are documented in a log book with proper storage procedures being followed.

### 2 QUALITY ASSURANCE MEASURES

The Environmental lab at Lupin utilizes several measures to ensure data is generated within known probability limits and is of acceptable accuracy and precision. The following principles and procedures apply to metal, arsenic and cyanide analyses at Lupin.

#### 2.1 ANALYSIS OF EXTERNALLY SUPPLIED REFERENCE MATERIALS

The analysis of external reference materials is performed within every sample set.

Only certified reference materials are used for laboratory control (ie. Environmental Resource Associates, EPA). Control standards with a concentration between 5 and 50 times the MDL (Minimum Detection Limit) or near sample ambient levels are used. (ie. ERA Cyanide WasteWatR™ Lot 9937 where certified value = 0.313 mg/L CN with advisory range for acceptable recovery = 0.23 - 0.40 mg/L CN).

## 2.2 ANALYSIS OF SAMPLES IN REPLICATE

With each series of samples, duplicate analysis of individual samples is included. This aids in assessing precision of the method and technique. Analyze 5% or more of the samples in duplicate. In some cases, it is practical and involves little extra effort to analyze all samples in duplicate. Acceptable limits for duplicate analysis at low levels are less stringent than those for known additions (75 - 100%). At higher levels however, they are more strict (90% - 110%).

Replicate analyses are useful for an overall precision assessment of the analyst (ie. Preparation and analysis of 10 replicate samples created from a single sample collection).

## 2.3 RECOVERY OF KNOWN ADDITIONS

Spiked sample analysis through the recovery of known additions is an important procedure for determining positive/negative interference within the sample. This is administered to previously analyzed samples and to blank samples as well. Known additions are applied to at least 10% of the number of samples. Additions should be at 50%, 100% and 150% of the anticipated sample analyte concentration. Also, only use additions within the demonstrated linear range of the method. Acceptable recovery limits are 80 - 120% for metals, organics, and inorganic parameters. For reference to limits mentioned above, please see (Standard Methods for the Examination of Water and Wastewater., 17th Edition, Part 1020B-Quality Control, Item 4., Page 1-8, Table 1020I.)

## 2.4 ANALYSIS OF REAGENT BLANKS

Reagent blanks are used in every sample set.

This is due to the new reagents required each time for the specific method (ie. Potassium iodide (KI) solution blanks in Arsenic analysis), and the need to correct the method for any interferences introduced by the reagents. A minimum of 5% of the sample load is analyzed as reagent blanks.

## 2.5 CONTROL CHARTS

Control charts are constructed to verify that quality is held within acceptable limits of variation (ie accuracy & precision). Chart analysis allows for a visual assessment of accuracy by utilizing warning limits; (upper warning levels - UWL and lower warning levels - LWL). These levels are the guideline between acceptable and unacceptable data entries.

A Means chart is commonly used for standards (lab control standards, calibration check standards) and reagent blank analyses while Range charts are used for duplicate analysis (an example of a data table and control chart is shown in Table IV on Page 18).

Both Mean and Range charts are to be used as supplements to the data validation process. A consideration may be to compile a year end summary of the control charts generated. Another consideration is the comparison of internal lab checks with external control charts.

## 2.6 INTERLABORATORY COMPARISON STUDIES

The Lupin Environmental Lab participates in an annual Interlaboratory study in which the Water Resources Division of Indian and Northern Affairs Canada sponsors. Samples are provided containing various constituents at unknown levels. This Interlaboratory study is an unbiased way to assess the quality of the laboratory's analytical measure and may also assist in isolating problem areas within methods of analysis or procedures.

## 2.7 EXTERNAL LAB ANALYSIS

External lab checks shall be administered 4 times per year for main priority sample analysis (ie. SNP sample assessment).

Other random external checks will also be applied as necessary. External analytical work is carried out by Norwest Labs Ltd. of Edmonton, Alberta. In all cases, a QC report is requested and is kept on file.

## 2.8 ROUND ROBIN ANALYSIS

The Lupin Environmental lab has proposed an "Intersite" or round robin analysis program. The draft proposal for the program is completed and ready for distribution. Both the Royal Oak Giant mine and the Miramar Con mine have committed to participation in the program. The program will be initiated in May of 1996.

## 2.9 DATA RECORDING AND VALIDATION

- Record data on rough data sheets.
- Check validity of results by applying quality control measures as outlined in Section 2 of the plan.
- Results not valid shall be re-run.
- When results are assessed and acceptable by the analyst, they are passed on to the Department Supervisor or Environmental Affairs manager for validation.
- Rough data can now be entered into the lab computer.
- Entries are checked for transpositional error.
- Files are backed up on disc.
- Files are then transferred for back up on the main frame.



### 3      REFERENCES

Dux, J.P. (1986). "Handbook of Quality assurance for the Analytical Chemistry Laboratory"; pp 18-34, 48-57; Van Nostrand Reinhold.

Environmental Resource Associates (1988). "A Guide to Quality Control Practices For Waste and Potable Water Analysts"; pp 3-24; ERA.

Mining Association of British Columbia (1992). "Technical Guide for the Environmental Management of Cyanide in Mining"; sec. 4-10,4-17; prepared by T.W. Higgs Associates.

"Standard Methods for the Examination of Water and Wastewater" (1989). sec. 1-4, 1-26; AHPA, AWWA and WPCF, 17th edition.

Indian and Northern Affairs, Water Resources Division (1990). "Generic Quality Assurance (QA) Plan Guidelines for use by Licensees in meeting SNP requirements for submission of a QA Plan"; INAC.

TABLE I

SAMPLING SPECIFICATIONS FOLLOWED BY THE LUPIN ENVIRONMENTAL LAB

Parameter	Container	Preservative	Storage Time	Volume Required
Arsenic	Polyethylene	Cool, 4°C	6 months	200
BOD	Polyethylene	Cool, 4°C	4 hours	1000
Copper	Polyethylene	2mL conc. HNO <sub>3</sub> /L sample	6 months	200
Cyanide	Polyethylene	4g NaOH/L sample	14 days	500
Hardness	Polyethylene	Cool, 4°C	24 hours	100
Iron	Polyethylene	2mL conc. HNO <sub>3</sub> /L sample	6 months	200
Lead	Polyethylene	2mL conc. HNO <sub>3</sub> /L sample	6 months	200
Nickel	Polyethylene	2mL conc. HNO <sub>3</sub> /L sample	6 months	200
pH	Polyethylene	None	6 hours	25
Residue	Polyethylene	Cool, 4°C	7 days	100
Specific Conductance	Polyethylene	Cool, 4°C	24 hours	100
Turbidity	Polyethylene	Cool, 4°C	7 days	100
Zinc	Polyethylene	2mL conc. HNO <sub>3</sub> /L sample	6 months	200

**TABLE II**  
**CALIBRATION GUIDELINES FOR LABORATORY INSTRUMENTS & EQUIPMENT**

INSTRUMENT/EQUIPMENT	CALIBRATION FREQUENCY	SPECIFICATIONS	COMMENTS
<b>Analytical Balance</b>  *Mettler AE 160	Bi-weekly. Prior to critical usages. (ie. preparation of standards)	Readability: 0.1 mg Weighing Range: 0-162g Tare Range: 0-162g Stabilization time: 5 sec. Integration Time: 1.5/3/6 sec Reproducibility: 0.1 mg Linearity: +/- 0.2 mg	Correct optical scale sensitivity Check reproducibility of scale Check internal consistency of the balance weights.
<b>PH Meter</b>  *Fisher Accumet 810	Prior to every usage.	Ranges: 0.00-14.00 pH +/- 1999mV Resolution: 0.01 pH, 1mV Repeatability: +/- 0.01 pH +/- 1mV Relative Accuracy +/-0.02 pH +/- 2mV Stability: < +/- 0.01 pH +/- 1mV/24hr. Temp. Compensation 0-100°C Readout: 4 digit	Calibrate with pH buffers 4,7, & 10 at room temperature. Check drift, stabilization, and if electrodes in proper condition.
<b>Ultraviolet(U.V) Spectrophotometer.</b>  *LKB 4050 Ultrospec.	Every 3 months through winter using a set of various standards. Once a week during summer months.(when sampling schedule is heavy).	Wavelength Rg: 202-900nm Accuracy: +/- 1nm Reproducibility: +/- 0.5 nm Transmission Rg: 0-250% Absorbance Rg: -0.999-2.5 Conc.& Factor Rg: 0-9999 Bandwidth: 5nm Straylight: < 0.1% @ 210nm Stability +/- 0.002 A/hr. @ 0 A Noise @ 600nm. +/- 0.001 A +/- 0.002 A near 2 A Linearity. +/- 1 % to 2.5 A or +/- 0.005 A Photometric Rep: Within 0.5 % of Abs. value.	Check Spectral Bandwidth. Check Wavelength Accuracy. Check for Stray Light. Check Photometric Linearity. Check Length & Positioning of sample cells. Calibrate using solution of 0.0 gm potassium chromate / L in 0.05M potassium hydroxide for UV & visible regions. A solution of potassium permanganate is used for more defined areas in the visible range.
<b>Atomic Absorption Spectrometer</b>  *Varian Spectra AA/20	Calibration is carried out every time the instrument is used.	The diversity and complexity of this instrument is too great for a listing of specifications. Many variables & factors contribute to instrument performance. The instrument conditions change for varying parameters.	Check sensitivity. Measure linear working range Continued verification of baseline noise. Calibrate against 6-8 certified standards.
<b>Turbidimeter.</b>  *(H.F. Inst.)DRT 15b portable	Calibration done prior to instrument use.	Ranges (NTU). 4 ranges: 0-1, 0-1.0-10, 0-100, 0-200. Linearity: +/-1% of full scale. Repeatability: +/-1% of scale Sensitivity: 0.02 NTU change.	Calibrate against an internally prepared suspension such as formazin. Externally supplied standards of latex can be used.
<b>Conductivity Meter.</b>  *Jenway 4020	Instrument used infrequently by Lupins' environmental lab. Calibrated on a once/month basis.	Autoranging: 0 to 1,999,000 us/cm Accuracy: +/-0.5 % Temp. Range: 0 to 100 °C Temp. Coefficient: 0 to 100°C Response Time: <5sec to 200 mmho	Calibrate against standard KC or NaCl solution. Determine range of acceptable accuracy.(< 1% acceptable)
<b>Utility Oven.</b>  *Blue M Stabil-Therm	Unit used infrequently and is calibrated on a once per 3 month basis.	Temperature Range : +38 °C to +260 °C	Thermometer built into oven is certified by the NBS ( National Bureau of Standards ). External Thermometer is used to check calibration of internal gauge.
<b>Automatic Pipettes &amp; Dispensers</b>  *Oxford dis./pip. bottles  *Eppendorf Adjustable & Fixed Volume Pipettes.	Calibrate before first usage and prior to all critical measurements.  Calibrate before first usage and prior to all critical measurements.	Capacity : 1 Litre. Range: 2-10 mL Accuracy: 0.5 % coefficient of variation at one S.D. Adjustable Increment : 0.2 mL  Size : 0.5 uL to 2500 uL Accuracy: +/-5.0% for 0.5 uL +/- 0.6 % for 2500 uL Precision: </-2.8% for 0.5 uL </- 0.2 % for 2500 uL	Calibrated gravimetrically with distilled water at room temp. A beaker is then weighed on the analytical balance. The desired aliquot is pipetted into beaker and weighed immediately. This is repeated several times so a mean and Standard Deviation can be calculated. Compare to manufacturers specifications.

TABLE III

EPA APPROVED ANALYTICAL METHODS FOR SELECTED PARAMETERS

PARAMETER	METHOD DESCRIPTION	REFERENCE METHOD - 17TH EDITION STANDARD METHODS / EPA METHOD NO.	PERFORMED BY	DETECTION LIMIT
ARSENIC	CONTINUOUS GASEOUS HYDRIDE GENERATION	3114 B PG. 3-43 to 53 / 206.3	EBM	0.002 mg/L
ACIDITY	TITRIMETRIC METHOD	2310 B PG. 2-30 to 34/	EBM**	3 ppm
ALKALINITY	TITRIMETRIC METHOD	2320 B PG 2-35 to 39 / 310.1	EBM**	3 ppm
BOD	5 DAY BOD PROCEDURE	5210 B PG 5-2 to 10/405.1	EBM	2 mg/L
CONDUCTIVITY	SELF CONTAINED METER	2510 B PG 2-59 to 61/	EBM	N/A
CYANIDE	TOTAL CN-COLORIMETRIC	4500 CN-E PG 4-31 to 32/ 335.2	EBM	0.020 mg/L
	WEAK ACID DISSOCIABLE	4500 CN-I PG 4-38 to 39 / 335.3	EBM	
HARDNESS	EDTA TITRIMETRIC METHOD	2340 C PG 2-53 to 57 / 130.2	EBM**	N/A
TRACE METALS (Zn,Cu,Pb,Ni)	ATOMIC ABSORPTION DIRECT AIR-ACETYLENE FLAME METHOD.	3111 B PG 3-20 to 23 / 289.1, 220.1, 239.2, 236.1, 249.1	EBM	Zn-0.005, Cu- 0.01, Ni-0.002, Pb-0.05 mg/L
MERCURY	COLD VAPOR AA SPEC.	3112 B PG 3-118 to 120 / 245.1	*NORWEST LABS	0.002 mg/L
CADMIUM	(ICP) AA METHOD	3120 B PG 3-54 to 63 / 200.7	*NORWEST LABS	0.004 mg/L
pH	ELECTROMETRIC METHOD	4500 HB PG 4-95 to 102 /	EBM	+/- 0.001 pH
SUSPENDED SOLIDS	TOTAL FILTERABLE SOLIDS	2540 D PG 2-75 to 77 / 160.2	EBM	N/A
TEMPERATURE	THERMOMETER	2550 B PG 2-80 to 81 /	EBM	N/A
TURBIDITY	NEPHELOMETRIC METER	2130 B PG 2-13 to 16 / 180.1	EBM**	N/A
FECAL COLIFORMS	MOST PROBABLE NUMBER (MPN) METHOD	9221 C PG 9-75 to 76 /	*NORWEST LABS	N/A

\*\* NOT REQUIRED FOR SNP

\*NORWEST LABS

External quality control checks are conducted by a "Standards Council of Canada" accredited laboratory. Echo Bay Mines Environmental Lab at Lupin utilizes Norwest Labs Ltd. of Edmonton, AB. for its external analytical services. The following briefly outlines what sample stations are tested, what parameters are being monitored, and the frequency at which they are being applied.

SAMPLE STATION	PARAMETERS	FREQUENCY OF TESTING AT STATION
925-01	As,Cu,Pb,Ni,Zn,Hg,Cd,C,S,FC,pH	Once per year - May
925-10	As,CN,Cu,Pb,Ni,Zn,S,pH	Once during discharge - July
925-14	As,Cu,Pb,Ni,Zn,S,pH,BOD,FC	Once prior to, Once during discharge - August
925-15	As,CN,Cu,Pb,Ni,Zn,S,pH	Once during treatment - August
VARIOUS NON DESIGNATED SAMPLE SITES	VARIOUS ANALYSIS As,CN,Cu,Pb,Ni,Zn,Fe,Alk,S, A,pH,BOD,B,NH4,H,C,T,TFC	External monitoring performed monthly except during winter when sampling is minimal. Testing performed on locations within mill circuit as well.

As-Arsenic,CN-Cyanide,Cu-Copper,Pb-Lead,Ni-Nickel,Zn-Zinc,Fe-Iron,Alk-Alkalinity,S-Suspended Solids,Acid-Acidity,pH,BOD-Biochemical Oxygen Demand,B-Bioassay,NH4-Ammonia,H-hardness,C-Conductivity,T-Temperature, FC-Fecal Coliforms,TFC-Total & Fecal Coliforms.

**TABLE IV**  
**EXAMPLE CONTROL CHART FOR EXTERNALLY SUPPLIED REFERENCE MATERIAL**

**ECHO BAY MINES LTD.**

**ENVIRONMENTAL LAB**

**QUALITY CONTROL SUMMARY ; 1992**

**ANALYSIS :     ARSENIC**

**ANALYSIS : ARSENIC**

**METHOD OF ANALYSIS :     3114B\***

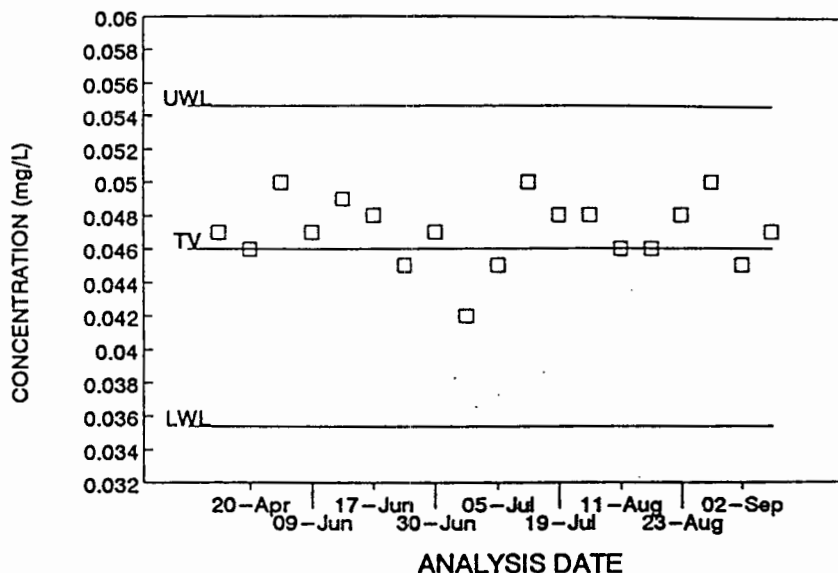
**REFERENCE MATERIAL SOURCE :     USEPA**

**LOT OR BATCH NO. :     WS378**

**CERTIFIED VALUE (mg/L) :     0.046**

ANALYSIS DATE	RESULT (mg/L)
26-Feb	0.047
20-Apr	0.046
06-Jun	0.050
09-Jun	0.047
10-Jun	0.049
17-Jun	0.048
23-Jun	0.045
30-Jun	0.047
04-Jul	0.042
05-Jul	0.045
17-Jul	0.050
19-Jul	0.048
27-Jul	0.048
11-Aug	0.046
20-Aug	0.046
23-Aug	0.048
26-Aug	0.050
02-Sep	0.045
12-Sep	0.047

**QUALITY CONTROL CHART**



**USEPA STATISTICAL DATA**

TRUE VALUE: 0.046 mg/L  
UPPER LIMIT: 0.055 mg/L  
LOWER LIMIT: 0.035 mg/L

**EBM STATISTICAL DATA**

MEAN: 0.047 mg/L  
STANDARD DEVIATION: 0.002 mg/L  
PERCENT ERROR: 4.34 %

**COMMENTS**

\* Slight modification to analysis method.(Arsenic-Continuous Gaseous Hydride Generation).

[illegible]

ECHO BAY MINES LTD.  
LUPIN OPERATION, NORTHWEST TERRITORIES