NIBLACK MINING CORPORATION
QUALITY ASSURANCE PROJECT PLAN

Prepared for

Niblack Mining Corporation
615-800 West Pender Street
Vancouver, B.C. V6C2V6
Canada

Prepared by

integral consulting inc.

319 SW Washington Street
Suite 1150
Portland, OR 97204

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A. PROJECT MANAGEMENT ELEMENTS

A1. APPROVALS

ADEC Project Manager	Date
ADEC QA Officer	Date
NMC Project Manager	Date
Integral Project Manager	Date
Integral QA Manager	Date
Laboratory Project Manager	Date
Laboratory QA Manager	Date

A2. TABLE OF CONTENTS

A. PROJECT MANAGEMENT ELEMENTS 2
A1. APPROVALS 2
A2. TABLE OF CONTENTS 3
A2.1 LIST OF FIGURES 5
A2.2 LIST OF TABLES 5
A2.3 ACRONYMS AND ABBREVIATIONS 5
A3. DISTRIBUTION LIST 6
A4. PROJECT/TASK ORGANIZATION 7
A4.1 Alaska Department of Environmental Conservation (ADEC) Organization and Responsibilities 8
A4.2 Integral Organization and Responsibilities 8
A4.3 Analytical Laboratory Organization and Responsibilities 9
A5. PROBLEM DEFINITION/BACKGROUND 10
A6. PROJECT/TASK DESCRIPTION 10
A7. DATA QUALITY OBJECTIVIES AND CRITERIA FOR MEASUREMENTS OF DATA 12
A8. TRAINING AND CERTIFICATIONS 16
A9. DOCUMENTS AND RECORDS 16
A9.1 Field Documentation 16
A9.2 Laboratory Documentation 17
A9.3 Data Quality Documentation 18
B. DATA GENERATION AND ACQUISTION 19
B1 SAMPLING PROCESS DESIGN 19
B2 SAMPLING METHODS 20
B3 SAMPLE HANDLING AND CUSTODY 20
B3.1 Field to Laboratory Sample Handling and Custody 20
B3.2 Intra-Laboratory and Sub-Laboratory Sample Transfer 21
B4. ANALYTICAL METHODS 21
B4.1 Conventional Analyses 23
B4.2 Cations/Anions 23
B4.3 Total/Dissolved Metals 23
B5. QUALITY CONTROL 23
B5.1 Field Quality Control Samples 23

B5.2 Laboratory Quality Control 24
B6. INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE 26
B7. INSTRUMENT/EQUIPMENT CALIBRATION AND FREQUENCY 26
B8. INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES 27
B9. DATA MANAGEMENT 27
B9.1 Field Data 28
B9.2 Laboratory Data 28

C. ASSESSMENT AND OVERSIGHT 29
C1. ASSESSMENTS AND RESPONSE ACTIONS 29
C2. REPORTS TO MANAGEMENT 30

D. DATA VALIDATION AND USABILITY 32
D1. DATA REVIEW, VALIDATION & VERIFICATION REQUIREMENTS 32
D2. DATA EVALUATION 32
D3. VALIDATION AND VERIFICATION METHODS 34
D4. RECONCILIATION WITH USER REQUIREMENTS 34

E. REFERENCES 35

Appendix A. Columbia Analytical Services Quality Assurance Manual

A2.1 LIST OF FIGURES

Figure A4-1. Niblack Project Site
Figure A4-2. Niblack Mining Corporation QAPP Organization Chart
Figure A6-1. Surface Water and Groundwater Monitoring Locations
Figure B3-1. Example of Chain-of-Custody Form

A2.2 LIST OF TABLES

Table A6-1. Niblack Exploration Project Water Quality Monitoring Sites
Table B1-1. Water Quality Parameters to be Monitored in Surface Water, Groundwater, and Effluent
Table B4-1. Laboratory Methods for Water Quality Samples
Table B5-2. Laboratory Control Limits for Matrix Spike and Laboratory Control Samples

A2.3 ACRONYMS AND ABBREVIATIONS

AAS	atomic absorption spectrometry
ADEC	Alaska Department of Environmental Conservation
COC	chain of custody
CVAAS	cold vapor atomic absorption spectrometry
DQO	data quality objective
EDD	electronic data deliverable
EPA	U.S. Environmental Protection Agency
ICP/AES	inductively-coupled plasma/atomic emission spectrometry
ICP/MS	inductively-coupled plasma/mass spectrometry
LCS/LCSD	laboratory control sample/laboratory control sample duplicate
LIMS	Laboratory Information Management System
MS/MSD	matrix spike/matrix spike duplicate
NAG	non-acid generating
NMC	Niblack Mining Corporation
PAG	potentially acid generating
PARCC	precision, accuracy, representatives, completeness, and comparability
QA	quality assurance
QAPP	quality assurance project plan
QC	quality control
RPD	relative percent difference
SOP	standard operating procedure
STORET	Storage and Retrieval (EPA database)
TDS	total dissolved solids
TSS	total suspended solids

A3. DISTRIBUTION LIST

Darwin Green
Niblack Mining Corporation
615-800 West Pender Street
Vancouver, B.C. V6C2V6
Canada
Phone: (604) 484-5045
Email: dgreen@niblackmining.com

Jim Gendron, Project Quality Assurance Officer
Alaska Department of Environmental Conservation-Water Division
410 Willoughby Avenue, Suite 303
Juneau, AK 99801
Phone: (907) 465-5305
Email: james.gendron@alaska.gov

Laura Jones, Project Manager
Integral Consulting Inc.
319 SW Washington Street, Suite 1150
Portland, OR 97204
Phone: (503) 284-5545, ext. 11
Email: ljones@integral-corp.com

Manon Tanner, Quality Assurance Manager
Integral Consulting Inc.
319 SW Washington Street, Suite 1150
Portland, OR 97204
Phone: (503) 284-5545, ext. 16
Email: mtanner@integral-corp.com

Greg Salata, Project Manager
Columbia Analytical Services
1317 South 13th Avenue
Kelso, WA 98620
Phone: (360) 577-7222
Email: gsalata@caslab.com

Lee Wolf, Quality Assurance Manager
Columbia Analytical Services
1317 South 13th Avenue
Kelso, WA 98620
Phone: (360) 577-7222
Email: lwolf@caslab.com

A4. PROJECT/TASK ORGANIZATION

Niblack Mining Corporation (NMC) is proposing to construct an underground exploration project at the Niblack property located on southern Prince of Wales Island approximately 30 miles southwest of Ketchikan in southeast Alaska (Figure A4-1). The exploration site is located on the north slope of Lookout Mountain. The Lookout Mountain Geological Unit contains mineralization, which is the target of this exploration project (RTR 2007a).

NMC submitted an application for a Wastewater Treatment and Disposal authorization for this site (RTR 2007a). The application is for a wastewater treatment system designed to control the 24-hour, 25-year storm event and to discharge water from a water storage/treatment pond through a drip emitter system to a natural attenuation system. The permit application incorporated additional project documents by reference including the Application for an Industrial Solid Waste Landfill Permit (RTR 2007b), *Water Quality Baseline and Site Monitoring Plan* (Monitoring Plan) (Knight Piesold 2007a), the *Operational Characterization Plan* (Knight Piesold 2007b), and *Underground Exploration Plan of Operations* (Niblack 2007). The Alaska Department of Environmental Conservation (ADEC) issued Waste Management Permit 2006-DB0037 for the Niblack Exploration Project on June 29, 2007 (ADEC 2007). Permit 2006-DB0037 covers both the disposal of non-domestic waste water and the management and disposal of potentially acid-generating (PAG) solid waste material that are associated with project development activities, exploration adit dewatering, and underground drilling.

ADEC has determined that site-specific natural condition-based water quality criteria shall be established under the permit for surface water and groundwater and shall follow the procedures in ADEC's guidance (ADEC 2006). The permit requires specific procedures for establishing site specific water quality criteria to be specified in a project quality assurance project plan (QAPP). The permit requires submittal of a QAPP for ADEC approval within thirty (30) days of the effective date of this permit or before work commences on the access tunnel portal, whichever is sooner.

The following section presents the organizational structure for activities associated with the NMC exploration project, including project management and oversight, fieldwork, sample analysis, and data management. The organizational structure for the NMC project activities is illustrated in the organization chart provided below. Project responsibilities are also described below.

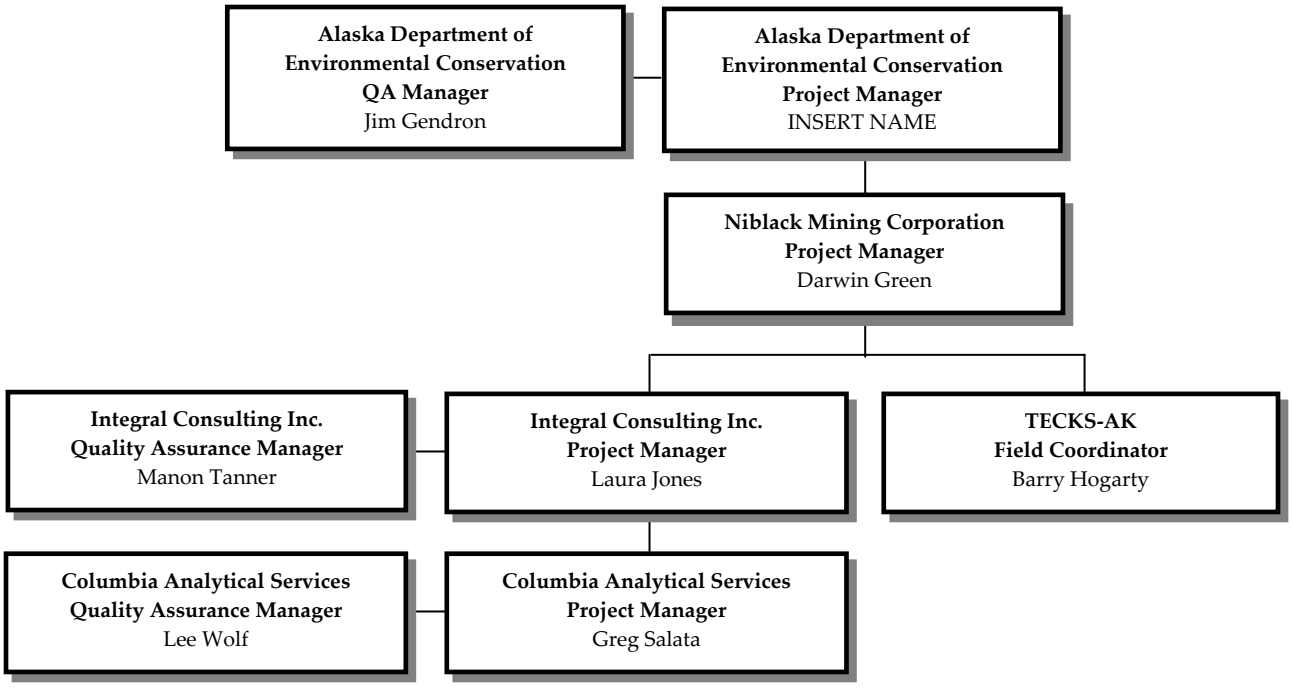

Figure A4-2Niblack Mining Corporation QAPP Organization Chart

A4.1 Alaska Department of Environmental Conservation (ADEC) Organization and Responsibilities

ADEC is the lead agency for all activities related to this project. ADEC will oversee the activities associated with the NMC exploration project, review quarterly and annual reports, and approve this QAPP. *Insert name here* is the ADEC project manager. Jim Gendron is the ADEC QA Officer.

A4.2 Niblack and Consultants Organization and Responsibilities

NMC Project Manager —Darwin Green is the NMC project manager. Mr. Green is responsible for final approval of the QAPP and all reports submitted to ADEC for this project.

Field Coordinator—Barry Hogarty is the field coordinator and will oversee all sample collection activities. The field coordinator will be responsible for collecting samples and field measurements, completing field sampling logs and chain-of-custody documentation, and shipping samples to the analytical laboratory. The field coordinator will document any deviations from the Monitoring Plan and QAPP, and report these deviations to the NMC and Integral project managers.

Project Manager—Laura Jones is the Integral Project Manager and will oversee development and implementation of the field program, including implementation of this QAPP.

Quality Assurance Manager—Manon Tanner is the Quality Assurance (QA) Manager for analytical chemistry. She will be responsible for the following activities:

- Coordinate the activities of the laboratories and track laboratory progress
- Ensure that method development is satisfactorily completed prior to analysis of project samples
- Verify that the laboratories implement the requirements of the Monitoring Plan and QAPP and address quality assurance issues related to laboratory analyses
- Ensure that laboratory capacity is sufficient to undertake the required analyses in a timely manner
- Address scheduling issues related to laboratory analyses
- Direct the validation of the chemical data
- Communicate data quality issues to the data users
- Work with data users and ADEC to address any data limitations.

A4.3 Analytical Laboratory Organization and Responsibilities

Laboratory Project Manger—The Laboratory Project Manager carries overall responsibility for the successful and timely completion of sample analyses for this project. The Laboratory Project Manager will be responsible for the following tasks:

- Ensure that samples are received and logged in correctly, that the correct methods and modifications are used, and that data are reported within specified turnaround times
- Review analytical data to ensure that procedures were followed as required in this QAPP, the cited methods, and laboratory standard operating procedures (SOPs)
- Keep the QA Manager apprised of the schedule and status of sample analyses and data package preparation
- Notify the QA Manager if problems occur in sample receiving, analysis, or scheduling, or if control limits cannot be met
- Take appropriate corrective action as necessary
- Report data and supporting QA information as specified in this QAPP.

Laboratory Quality Assurance Manager—the Laboratory QA Manager is responsible for overseeing the QA activities in the laboratory and ensuring the quality of the data for this project. Specific responsibilities include the following:

- Oversee and implement the laboratory's QA program

- Maintain QA records for each laboratory production unit
- Ensure that quality assurance/quality control (QA/QC) procedures are implemented as required for each method and provide oversight of QA/QC practices and procedures
- Review and address or approve non-conformity and corrective action reports
- Coordinate responses to any QC issues that affect this project with the Laboratory Project Manager.

A5. PROBLEM DEFINITION/BACKGROUND

The State of Alaska issued Waste Management Permit No. 2006-DB0037 for the Niblack Exploration Project on June 29, 2007. Among other provisions, the permit includes requirements for monitoring of surface water and groundwater quality to ensure compliance with site-specific natural conditions based water quality standards. This QAPP specifies procedures and criteria to ensure that data collected and analyzed for this project will satisfy the requirements of the waste management permit and be valid and verifiable.

A6. PROJECT/TASK DESCRIPTION

The project is located on the southern end of Prince of Wales Island about 30 miles southwest of Ketchikan in the Ketchikan Recording District on Craig A-1 USGS Map. The proposed project site is located within Section 34, T. 78 S., R. 88 E., Copper River Meridian; in Niblack Anchorage, off Moira Sound on Prince of Wales Island, Southeast Alaska. The Niblack operation comprises approximately 6000 ft of underground drift development to provide access for continued exploration drilling on the Lookout and Mammoth massive sulfide mineral zones. Metals in the massive sulfide mineralization include copper, zinc, gold and silver. Non-acid generating (NAG) rock will be disposed of on the hill slope adjacent to the portal. The estimated quantity of NAG rock is 46,600 cubic yards. PAG rock will be disposed of at a 25,000-ft^2 lined temporary site until ultimate disposal by placing it back underground at the termination of the exploratory phase. The estimated quantity of PAG rock is 14,300 cubic yards. Water from the adit and PAG waste rock site will be disposed of via a two-pond treatment system, each pond being 76ft x 76ft x 8ft deep, and then to a drip infiltration system in woodlands. The project is expected to last approximately 2 years.

Surface water and groundwater quality monitoring locations are shown on Figure A6-1. Pre-construction surface water quality monitoring has been occurring within the Niblack Project study area since 1996 to establish baseline water quality for the area. Samples were collected in October 1996, September 1997, April 2005 and on multiple occasions in 2006 and 2007. Pre-construction groundwater monitoring commenced late in 2006. Both surface water and

groundwater monitoring will continue in advance of construction activities to further establish natural background conditions.

The purpose of each monitoring site under the waste management permit is specified in Table A6-1. Generally, the compliance points for evaluating compliance with site-specific natural conditions standards are downstream surface water sites and downgradient wetland groundwater sites. Eight surface water locations are monitored under the waste management permit. Six of these sites monitor upstream and downstream water quality in three of the creeks and streams within the immediate project area. From south to north the names of these creek systems are Waterfall Creek, Camp Creek and Unnamed Creek 1 (adjacent to old camp facilities). A seventh site is located on Unnamed Creek 2, downgradient of the NAG waste rock storage site, and an eighth site is located at the base of the PAG waste rock storage site as part of a leak detection system. No upgradient site is located on Unnamed Creek 2 because of the short length of this stream system. Seven permanent groundwater locations are monitored under the waste management permit. Five of these are permanent wells installed in the wetlands in February 2007. Two upgradient wells, MW8 and MW9, were installed upgradient of project facilities in July 2007.

The monitoring schedule, parameters, protocol and reporting requirements are described in Section B1. Statistical procedures for evaluating compliance with the site-specific standards are provided in Section D2.

Table A6-1. Niblack Exploration Project Water Quality Monitoring Sites.

Monitoring point	Location	Purpose				
		Pre-project reference conditions	Concurrent reference conditions	Compliance location	Information only	Post-closure monitoring
Effluent						
EFF1	Discharge from the treatment ponds				X	
Surface waters						
WQ4	Waterfall Creek – downstream	X		X		X
WQ6	Camp Creek – downstream	X		X		
WQ7	Camp Creek – upstream	X	X			
WQ8	Waterfall Creek – upstream	X	X			
WQ10	Unnamed Creek 1 – downstream	X		X		
WQ12	Unnamed Creek 1 – upstream	X	X			
WQ13	Unnamed Creek 2	X				X
	PAG site under drain				See Note A1	
Ground water wells						
MW1	Wetlands below NAG site	X	See Note A2	See Note A2		X
MW2	Wetlands below settlement/treatment ponds	X	See Note A2	See Note A2		X
MW3	Wetlands below PAG site	X	See Note A2	See Note A2		X
MW4	Wetlands below infiltration system area	X	See Note A2	See Note A2		X
MW7	Wetlands – offsite and to the east of the project	X	X			
MW8	Up gradient of land application area and MW3				See Note A3	
MW9	Up gradient of land application area and MW4				See Note A3	

Note A1: Monitoring PAG site under drain is a component of the leak detection system, required to monitor potential degradation of ground waters should a breach in the liner occur.

Note A2: MW1, MW2, MW3 and MW4 will be used for monitoring for changes to natural water quality in wetlands water when compared to historic values and remote wetland wells.

Note A3: MW8 and MW9 will be used to determine background groundwater quality for information purposes only.

A7. DATA QUALITY OBJECTIVIES AND CRITERIA FOR MEASUREMENTS OF DATA

Project Data Quality Objectives

Data quality objectives (DQOs) are designed to ensure that the type, quality, and quantity of environmental data used in decision making are appropriate for their intended application. For this QAPP, the following two DQOs have been identified to ensure that data of adequate quantity and quality are generated to support the requirements of the State of Alaska's Waste Management Permit No. 2006-DB0037:

> ***DQO 1—Surface Water Quality Compliance.*** The DQO for surface water quality compliance is to ensure that data of sufficient quantity and quality are collected to determine whether concentrations of water quality parameters in designated surface water compliance monitoring locations comply with site-specific natural conditions based water quality standards. The site-specific standards for surface water will be established by the combined dataset from surface water monitoring conducted at pre-project reference locations before exploratory activity and from ongoing monitoring of upstream reference locations. Pre-project reference locations, concurrent reference locations, and compliance monitoring locations for surface water are presented in Table A6-1.
>
> ***DQO 2—Wetlands Groundwater Quality Compliance.*** The DQO for wetlands groundwater quality compliance is to ensure that data of sufficient quality and quantity are collected to determine whether concentrations of water quality parameters in designated wetlands compliance wells comply with site-specific natural conditions based water quality standards. The site-specific standards will be determined based on pre-construction monitoring of wetlands wells and concurrent monitoring of remote wetlands reference location(s). Pre-project reference locations, concurrent reference location, and compliance monitoring locations for wetlands groundwater are presented in Table A6-1.

Statistical procedures for defining the site-specific standards are described in Section D2 of this QAPP. For this project, a confidence level of 95 percent has been selected to evaluate whether concentrations of target analytes in compliance locations are within the range of naturally occurring background concentrations in reference locations.

In accordance with the project DQOs, field and laboratory procedures for water quality monitoring have been established to ensure that the quantity and quality of data generated by field and laboratory activities are sufficient to evaluate compliance with the site-specific water quality standards at an acceptable level of confidence. An overview of the sampling process design is presented in Section B1; detailed field procedures are described in the Monitoring

Plan. Field measurements and laboratory analyses to be performed on surface water, groundwater, and effluent samples are presented in Table B4-1. Laboratory analytical methods are presented in Table B4-1, and reporting limits are presented in Table B5-2.

Criteria for Measurement of Data

DQOs were developed to describe data and data quality needs for the project. Data quality indicators such as the precision, accuracy, respresentativeness, completeness, and comparability (PARCC) parameters (EPA 2002a) and analytical sensitivity will be used to assess conformance of data with quality control criteria. PARCC parameters are commonly used to assess the quality of environmental data.

Accuracy

Accuracy or bias represents the degree to which a measured concentration conforms to the reference value. The results for matrix spikes, laboratory control samples, field blanks, and method blanks will be reviewed to evaluate bias of the data. The following calculation is used to determine percent recovery for a matrix spike sample:

$$\%R = \frac{M - U}{C} x100$$

%R = Percent recovery
M = Measured concentration in the spiked sample
U = Measured concentration in the unspiked sample
C = Concentration of the added spike

The following calculation is used to determine percent recovery for a laboratory control sample or reference material:

$$\%R = \frac{M}{C} x100$$

%R = Percent recovery
M = Measured concentration in the reference material
C = Established reference concentration

Results for field and method blanks can reflect systematic bias that results from contamination of samples during collection or analysis. Any analytes detected in field or method blanks will be evaluated as potential indicators of bias.

Precision

Precision reflects the reproducibility between individual measurements of the same property. Precision will be evaluated using the results of matrix spike duplicates, laboratory duplicates, and field replicates. Precision is expressed in terms of the relative standard deviation for three or more measurements and the relative percent difference (RPD) for two measurements. The following equation is used to calculate the RPD between measurements:

$$RPD = \frac{|C_1 - C_2|}{(C_1 + C_2)/2} x100$$

RPD = Relative percent difference
C_1 = First measurement
C_2 = Second measurement

The relative standard deviation is the ratio of the standard deviation of three or more measurements to the average of the measurements, expressed as a percentage.

Representativeness

Representativeness is the degree to which data represent a characteristic of an environmental condition. In the field, representativeness will be addressed primarily in the sampling design by the selection of sampling sites and sample collection procedures. In the laboratories, representativeness will be ensured by the proper handling and storage of samples and initiation of analysis within holding times.

Comparability

Comparability is the qualitative similarity of one data set to another (i.e., the extent to which different data sets can be combined for use). Comparability will be addressed through the use of field and laboratory methods that are consistent with methods and procedures recommended by the U.S. Environmental Protection Agency (EPA) and by statistical evaluation of the data (Section D2).

Completeness

Completeness is the comparison between the amount of usable data collected versus the amount of data called for in the permit and/or certification. Completeness will be determined by comparing sampling and analyses completed with the requirements in the permit. The overall completeness goal is 95 percent.

A8. TRAINING AND CERTIFICATIONS

Integral has assembled a project team with the requisite experience and technical skills to successfully complete the NMC project. All consultant team personnel involved in sample collection have extensive environmental sampling experience. Minimum training and certification requirements for laboratory personnel are described in the laboratory QA manual (Appendix A of this QAPP).

A9. DOCUMENTS AND RECORDS

Records will be maintained documenting all activities and data related to sample collection and to laboratory analyses. Results of data verification and validation activities will also be documented. Procedures for documentation of these activities are described in this section.

A copy of the QAPP will be provided to every project participant listed in Section A3. Any revisions or amendments to any of these documents will also be provided to these individuals.

A9.1 Field Documentation

The following field records will be maintained throughout the duration of sampling activities:

- Field logbooks
- Field data forms
- Sample description forms
- Sample labels
- Sample chain-of-custody (COC) forms
- Custody labels
- Photographic documentation.

Field documentation related to sample collection will be maintained by the field coordinator in a designated project file. The laboratory will return original completed COC forms to Integral with the data packets; these will also be maintained in the project file.

The following reports will be completed, as necessary, to document an audit or a deviation from the QAPP:

- Corrective action reports will be used, as necessary, to document any problems encountered during field activities and corrective actions taken
- Field change request forms will be used, as necessary, to document the need for a procedural change or a station location change

- System and performance audit reports will be used, as necessary, to document review or audit of field sampling activities.

The Integral project manager will ensure that the field coordinator receives the final approved version of the QAPP prior to the initiation of field activities.

A9.2 Laboratory Documentation

All activities and results related to sample analysis will be documented at the analytical laboratory. Internal laboratory documentation procedures are described in the laboratory QA manual (Appendix A of this QAPP).

The analytical laboratory will provide a data package for each sample delivery group or analysis batch. Each data package will contain all information required for a completed QA review, including the following:

- A cover letter discussing analytical procedures and any difficulties that were encountered
- A case narrative referencing or describing the procedures used and discussing any analytical problems and deviations from SOPs and this QAPP
- COC and cooler receipt forms
- A summary of analyte concentrations (to two significant figures, unless otherwise justified), method reporting limits, and method detection limits
- Laboratory data qualifier codes appended to analyte concentrations, as appropriate, and a summary of code definitions
- Sample preparation and cleanup logs
- Instrument tuning check data
- Initial and continuing calibration data, including instrument printouts and quantification summaries, for all analytes
- Results for method and calibration blanks
- Results for all QA/QC checks, including but not limited to surrogate spikes, internal standards, laboratory control samples, matrix spike samples, matrix spike duplicate samples, and laboratory duplicate or triplicate samples provided on summary forms
- Instrument data quantification reports for all analyses and samples
- Copies of all laboratory worksheets and standards preparation logs.

Data will be delivered in both hard-copy and electronic format to the Integral QA Manager, who will be responsible for oversight of data verification and validation and for archiving the

final data and data quality reports in the project file. Electronic data deliverables will be compatible with EPA's Storage and Retrieval (STORET) database.

Laboratory data will be maintained by each laboratory for a period of at least 5 years. These data will include the original instrument data files, reduced and verified data stored in the laboratory information management system (LIMS), and final hard-copy and electronic data deliverables. The laboratory will obtain approval from Integral prior to discarding these data.

A9.3 Data Quality Documentation

Data validation reports will be prepared and provided to the QA manager. Results of the validation reports and any limitations to the usability of the data will be summarized in the project summary data report for each sampling event.

All electronic database entries provided by each laboratory will be verified against the validated hard-copy data in the data package. All changes to the database will be documented in an electronic log file that automatically enters a current time stamp when opened and allows the data editor to enter notes about changes to the database. Any data tables prepared from the database for data users will include all qualifiers that were applied by the laboratories and during data validation, unless otherwise requested.

B. DATA GENERATION AND ACQUISTION

B1 SAMPLING PROCESS DESIGN

Surface water, groundwater, and effluent water quality monitoring locations are shown on Figure A6-1 and Table A6-1. Water quality analyses to be performed are listed in Table B1-1. Detailed field procedures are described in the Monitoring Plan.

Surface water sites shall be sampled monthly until 20 valid pairs of upstream and downstream sample results are obtained, (or 20 valid samples for any surface water site that does not have an associated upstream site), then quarterly thereafter unless any parameter is greater at the downstream site than at the upstream site. If the downstream value is significantly greater (statistically) than the upstream value, then monitoring at those downstream and upstream locations shall be monthly until either the cause shall be shown to be natural, or corrected if caused by project activity, at which time the monitoring frequency shall revert to quarterly. For location WQ13 which does not have an upstream location for comparative purposes, if there is a significant upward trend in any parameter, then that location shall be monitored monthly until either the cause shall be shown to be natural, or corrected if caused by project activity, at which time the monitoring frequency shall revert to quarterly.

Ground water wells shall be sampled monthly until 20 valid sample results are obtained, then quarterly thereafter. Note that the results for some groundwater wells will be combined (see Note A2 to Table A6-1) for generation of 20 valid sample results. EFF1 will be sampled monthly with the other groundwater wells, once the waste treatment system is operational. Once the groundwater sampling frequency has shifted to quarterly sampling, EFF1 will be sampled quarterly, with the groundwater wells. For compliance wells, if any monitored parameter increases statistically significantly over natural levels, based on the combined dataset from pre-project baseline monitoring and monitoring during the project of a reference location located outside of the area of potential influence of project activities, or if there is a significant upward trend in any parameter, then that well shall be monitored monthly until either the cause shall be shown to be natural, or corrected if caused by project activity, at which time the monitoring frequency shall revert to quarterly.

Table B1-1. Water Quality Parameters to be Monitored in Surface Water, Groundwater, and Effluent.

Characteristic	Sample Type
Total Dissolved Solids (TDS)	Grab
Temperature	Grab
Conductivity	Grab
TDS cations/anions	Grab
pH	Grab
Nitrogen (nitrate/nitrite)	Grab
Aluminum	Grab
Arsenic	Grab
Cadmium	Grab
Copper	Grab
Lead	Grab
Mercury	Grab
Selenium	Grab
Zinc	Grab

B2 SAMPLING METHODS

Procedures for collection, preservation, and handling of surface water, groundwater, and effluent samples are specified in the Monitoring Plan

B3 SAMPLE HANDLING AND CUSTODY

B3.1 Field to Laboratory Sample Handling and Custody

Detailed descriptions of procedures for sample identification, handling, documentation, custody, and ultimate disposal are presented in the Monitoring Plan.

From the time of collection, all samples will be stored on ice or refrigerated at an approximate temperature of 4°C.

The field coordinator, or the designated field sample custodian, will be responsible for sample tracking in the field. Samples will remain in the field coordinator's custody until COC forms and final sample inventory are completed in the field or at the field sample processing facility. COC forms will be used for samples that are in transit from the field site to the analytical laboratory. The custodian will relinquish the samples prior to shipping to the analytical laboratory.

Samples are considered to be in custody if they are 1) in the custodian's possession or view, 2) in a secured location (under lock) with restricted access, or 3) in a container that is secured with an official seal(s) such that the sample cannot be reached without breaking the seal(s). The principal documents used to identify samples and to document possession are COC records,

field logbooks, and field tracking forms. COC procedures will be used for all samples at all stages in the analytical or transfer process and for all data and data documentation whether in hard-copy or electronic format. An example of a COC form is provided in Figure B3-1.

Sample packing and shipping procedures are detailed in Section 2.6 of the Monitoring Plan. Samples will be shipped to the laboratory in ice chests sealed with custody seals. Each ice chest will have three seals, one on the front of the chest and one on each side. The laboratory sample custodian will establish the integrity of the seals at the laboratory. The way bill of the carrier used to ship samples will provide additional custody and sample tracking information. The way bills will be maintained in the project file.

The sample custodian at the laboratory will accept custody and log samples into the LIMS. The sample custodian will check that the COC forms were properly completed and signed, that a sample receipt form is completed for each cooler, and that samples are stored under the required temperature conditions. The laboratory will deliver a copy of the COC and sample receipt form to the QA manager. Any breaks in the COC or non-conformances will be noted and reported in writing to the QA manager within 24 hours of receipt of samples. Specific laboratory COC procedures are described in the laboratory QA manual (Appendix A).

B3.2 Intra-laboratory and Sub-laboratory Sample Transfer

The laboratory project manager will ensure that a sample-tracking record is maintained that follows each sample through all stages of laboratory processing. The sample-tracking record must contain, at a minimum, the names of individuals responsible for performing the analysis; dates of sample extraction, preparation, and analysis; and the type of analysis being performed.

Any sample needing further analysis that is not performed by the initial contracted laboratory will be subject to all custody specifications provided in the previous section.

B4. ANALYTICAL METHODS

Laboratory methods to be used for the NMC project are consistent with requirements provided in EPA SW-846 methods (USEPA 2007). Samples will be analyzed for the following:

- Conventional analyses
- Cations/anions
- Total/dissolved metals

The total number of samples and the analyses that will be conducted on each sample are indicated in the Monitoring Plan. The laboratory methods for sample preparation and analysis are summarized in Table B4-1.

Table B4-1. Laboratory Methods for Water Quality Samples.

Analysis	Sample Preparation		Quantitative Analysis	
	Protocol	Procedure	Protocol	Procedure
Conventional Analyses				
Hardness as $CaCO_3$	--	--	EPA 130.2	Titrimetric
Total dissolved solids	--	--	EPA 160.1	Gravimetric
Total suspended solids	--	--	EPA 160.2	Gravimetric
Cations/Anions				
Alkalinity as $CaCO_3$	--	--	SM 2320	Titrimetric
Bromide, chloride, fluoride, sulfate	--	--	EPA 300.0	Ion chromatography
Nitrate/nitrite as N	EPA 353.2	Cadmium reduction	EPA 353.2	Colorimetric
Ammonia as N	EPA 350.1	Buffered to pH 9.5	EPA 350.1	Colorimetric
o-Phosphate as P, dissolved	EPA 365.3	Persulfate digestion	EPA 365.3	Colorimetric
Phosphate as P, dissolved		Persulfate digestion & 0.45-mm filtration		
Phosphate as P, total		Persulfate digestion		
Total/Dissolved Metals				
Aluminum, antimony, arsenic, barium, beryllium, bismuth, cadmium, chromium, cobalt, copper, lead, manganese, molybdenum, nickel, selenium, silver, thallium, uranium, vanadium, zinc	EPA 3020A	Nitric acid digestion	EPA 6020	ICP/MS
Boron, calcium, iron, lithium, magnesium, phosphorus, potassium, silicon, sodium, strontium, tin, titanium	EPA 3010A	Nitric/hydrochloric acid digestion	EPA 6010	ICP/AES
Mercury	EPA 7470A	Acid digestion/oxidation	EPA 7470A	CVAAS

Notes:

-- Not applicable
Field measurements collected for each event include dissolved oxygen, pH, temperature, conductivity, and turbidity.

CVAAS - cold vapor atomic absorption spectrometry
ICP/AES - inductively-coupled plasma/atomic emission spectrometry
ICP/MS - inductively-coupled plasma/mass spectrometry
SM - Standard Method
EPA - U.S. Environmental Protection Agency

B4.1 Conventional Analyses

Conventional analyses will include hardness as $CaCO_3$, total dissolved solids (TDS), and total suspended solids (TSS). EPA methods will be used as shown in Table B4-1.

Hardness as $CaCO_3$ will be determined titrimetrically according to EPA Method 130.2. TDS and TSS will be determined gravimetrically according to EPA Methods 160.1 and 160.2, respectively.

B4.2 Cations/Anions

Alkalinity as $CaCO_3$ will be determined titrimetrically according to Standard Method 2320. Bromide, chloride, fluoride, and sulfate will be determined by ion chromatography according to EPA Method 300.0. Nitrate/nitrite as nitrogen and ammonia as nitrogen will be determined colorimetrically according to EPA Methods 353.2 and 350.1, respectively. Ortho-phosphate, total phosphate as phosphorus, and dissolved phosphate as phosphorus will be determined colorimetrically according to EPA Method 365.3.

B4.3 Total/Dissolved Metals

Three methods will be used to analyze samples for total and dissolved metals (Table B4-1). Digestion with nitric and hydrochloric acids will be used to prepare samples for analysis of metals other than mercury. Analysis for these metals other than mercury and selenium will be completed by inductively coupled plasma/mass spectrometry (ICP/MS) and inductively coupled plasma/atomic emission spectrometry (ICP/AES), according to EPA Methods 6020 and 6010.

Mercury samples will be extracted with aqua regia and oxidized using potassium permanganate. Analyses will be completed by cold vapor atomic absorption spectrometry (CVAAS), according to EPA Method 7470A.

B5. QUALITY CONTROL

Quality control samples will be prepared in the field and at the laboratories to monitor the bias and precision of the sample collection and analysis procedures.

B5.1 Field Quality Control Samples

Field QC samples for this study will include field replicates and field blanks. A summary of field QC samples that will be collected for each sampling event is provided below.

Field replicates are samples collected at the same station, but using different deployments of the sampling equipment. The data for field replicates are used to evaluate variability at the

sampling site. One field replicate will be collected for each event for the surface water quality stations and the groundwater monitoring stations.

Equipment rinse blanks are used to monitor equipment decontamination procedures. Equipment rinse blanks will not be collected for this project because disposable equipment (e.g. tubing for groundwater wells) is being used for each sampling event, and the equipment is not being decontaminated between stations.

A field blank (a sample collection bottle filled with laboratory distilled/deionized water) is included with each sampling event. This blank is used to ensure that the sample containers and laboratory water do not contain analytes of interest at concentrations that impact the project samples.

B5.2 Laboratory Quality Control

Extensive and detailed requirements for laboratory QC procedures are provided in the method protocols that will be used for this study (Table B4-1). Every method protocol includes descriptions of QC procedures, and many incorporate additional QC requirements by reference to separate QC chapters. QC requirements include control limits and, in many cases, requirements for corrective action. QC procedures will be completed by the laboratories, as required in each method protocol and as indicated in this QAPP.

The frequency of analysis for laboratory control samples, matrix spike samples, matrix spike duplicates or laboratory duplicates, and method blanks will be one for every 20 samples, or one per extraction batch, whichever is more frequent. Surrogate spikes and internal standards will be added to every field sample and QC sample, as required by the method. Calibration procedures will be completed at the frequency specified in each method description. As required for EPA SW-846 methods (USEPA 2007), performance-based control limits have been established by each laboratory. These and all other control limits specified in the method descriptions will be used by the laboratories to establish the acceptability of the data or the need for reanalysis of the samples. Control limits for laboratory control sample/laboratory control sample duplicates (LCS/LCSDs), and matrix spike/matrix spike duplicates (MS/MSDs) are provided in Table B5-2.

Where discrepancies exist between this QAPP and laboratory SOPs and QA Manuals, this QAPP will take precedence.

Table B5-2. Laboratory Control Limits for Matrix Spike and Laboratory Control Samples.

Analysis [a]	Units	MRL	MDL	Matrix Spike Recovery (percent)	Laboratory Control Sample Recovery (percent)	Type of Duplicate	Control Limit Relative Percent Difference
Conventional Analyses							
Hardness as $CaCO_3$	mg/L	2	0.6	75-125	85-115	LD	20
Total dissolved solids	mg/L	5	--	--	85-115	LD	20
Total suspended solids	mg/L	5	--	--	85-115	LD	20
Cations/Anions							
Alkalinity as $CaCO_3$	mg/L	2	0.8	--	85-115	LD	20
Bromide	mg/L	0.2	0.02	80-120	90-110	LD	20
Chloride	mg/L	0.2	0.03	80-120	90-110	LD	20
Fluoride	mg/L	0.2	0.005	80-120	90-110	LD	20
Sulfate	mg/L	0.2	0.03	80-120	90-110	LD	20
Nitrate/nitrite as N	mg/L	0.2	0.02	90-110	90-110	LD	20
Ammonia as N	mg/L	0.05	0.006	90-110	90-110	LD	20
o-Phosphate as P, dissolved	mg/L	0.01	0.003	75-125	85-115	LD	20
Phosphate as P, dissolved	mg/L	0.01	0.003	75-125	85-115	LD	20
Phosphate as P, total	mg/L	0.01	0.003	75-125	85-115	LD	20
Total/Dissolved Metals							
EPA Method 6020							
Aluminum	ug/L	2	0.8	73-123	88-117	LD	20
Antimony	ug/L	0.05	0.03	63-131	93-106	LD	20
Arsenic	ug/L	0.5	0.07	68-128	88-110	LD	20
Barium	ug/L	0.05	0.02	77-126	93-106	LD	20
Beryllium	ug/L	0.02	0.006	56-134	87-124	LD	20
Bismuth	ug/L	0.1	0.02	75-125	85-115	LD	20
Cadmium	ug/L	0.017	0.017	82-114	90-109	LD	20
Chromium	ug/L	0.2	0.05	62-121	87-114	LD	20
Cobalt	ug/L	0.02	0.005	80-119	87-115	LD	20
Copper	ug/L	0.1	0.07	52-129	87-114	LD	20
Lead	ug/L	0.05	0.02	72-116	90-110	LD	20
Manganese	ug/L	0.05	0.01	73-121	87-117	LD	20
Molybdenum	ug/L	0.05	0.03	78-125	86-111	LD	20
Nickel	ug/L	0.2	0.05	66-121	87-114	LD	20
Silver	ug/L	0.02	0.009	46-136	85-111	LD	20
Thallium	ug/L	0.02	0.004	79-112	88-110	LD	20
Tin	ug/L	0.1	0.04	75-125	85-115	LD	20
Uranium	ug/L	0.02	0.004	75-125	85-115	LD	20
Vanadium	ug/L	0.2	0.05	75-120	86-115	LD	20
Zinc	ug/L	0.5	0.1	57-126	87-116	LD	20
EPA Method 6010B							
Boron	ug/L	50	9	76-132	89-112	LD	20
Calcium	ug/L	50	30	75-125	94-111	LD	20

Table B5-2. Laboratory Control Limits for Matrix Spike and Laboratory Control Samples.

Analysis [a]	Units	MRL	MDL	Matrix Spike Recovery (percent)	Laboratory Control Sample Recovery (percent)	Type of Duplicate	Control Limit Relative Percent Difference
Iron	ug/L	20	3	58-142	94-113	LD	20
Lithium	ug/L	10	6	70-130	85-115	LD	20
Magnesium	ug/L	5	0.6	75-125	91-112	LD	20
Phosphorus	ug/L	200	200	70-130	85-115	LD	20
Potassium	ug/L	2000	900	75-125	89-117	LD	20
Silicon	ug/L	400	40	70-130	85-115	LD	20
Sodium	ug/L	100	50	75-125	92-116	LD	20
Strontium	ug/L	10	0.5	70-130	85-115	LD	20
Titanium	ug/L	10	3	70-130	85-115	LD	20
EPA Method 7742							
Selenium	ug/L	1	0.2	70-121	87-113	LD	20
EPA Method 7470A							
Mercury	ug/L	0.2	0.03	79-118	83-112	LD	20

Notes:

[a] The analytes listed in this table reflect the full analyte list from the Water Quality Baseline and Site Monitoring Plan; the Waste Management Permit requires the collection of TDS, temperature, conductivity, pH, nitrate/nitrite as nitrogen, aluminum, arsenic, cadmium, copper, lead, mercury, selenium, zinc for groundwater and surface water samples.

-- not applicable

LD - laboratory duplicate
MDL - method detection limit
MRL - method reporting limit

B6. INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

Analytical instrument testing, inspection, maintenance, setup, and calibration will be conducted by each laboratory in accordance with the requirements identified in the laboratory SOPs and manufacturer instructions. Instrument maintenance and repair will be documented in maintenance logs or record books.

B7. INSTRUMENT/EQUIPMENT CALIBRATION AND FREQUENCY

Laboratory instruments will be properly calibrated, and the calibration will be verified with appropriate check standards and calibration blanks for each parameter before beginning each analysis. Instrument calibration procedures and schedules will conform to analytical protocol requirements and descriptions provided in the laboratories' QA manuals and SOPs.

All calibration standards will be obtained from either the EPA repository or a commercial vendor, and the laboratory will maintain traceability to the National Institute of Standards and Technology. Stock standards will be used to make intermediate standards and calibration standards. Special attention will be given to expiration dating, proper labeling, proper refrigeration, and prevention of contamination. Documentation relating to the receipt, mixing, and use of standards will be recorded in a laboratory logbook. All calibration and spiking standards will be checked against standards from another source.

B8. INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES

The quality of supplies and consumables used during sample collection and laboratory analysis can affect the quality of the project data. All equipment that comes into contact with the samples and extracts must be sufficiently clean to prevent detectable contamination, and the analyte concentrations must be accurate in all standards used for calibration and quality control purposes.

During sample collection, solvents of appropriate, documented purity will be used for decontamination. Solvent containers will be dated and initialed when they are opened. The quality of laboratory water used for decontamination will be documented at the laboratory that provides that water. Cleaned and documented sample containers will be provided by the laboratory. All containers will be visually inspected prior to use, and any suspect containers will be discarded.

Reagents of appropriate purity and suitably cleaned laboratory equipment will also be used for all stages of laboratory analyses. Details for acceptance requirements for supplies and consumables at the laboratories are provided in the laboratory SOPs and QA manuals (Appendix A). All supplies will be obtained from reputable suppliers with appropriate documentation or certification. Supplies will be inspected to confirm that they meet use requirements, and certification records will be retained by Integral (i.e., for supplies used in the field) or the laboratories.

B9. DATA MANAGEMENT

Data for this project will be generated in the field and at the laboratories. The final repository for all sample information will be a STORET database. Procedures to be used to transfer data from the point of generation to the STORET database are described in this section.

B9.1 Field Data

Daily field records (a combination of field logbooks and field data sheets) will make up the main documentation for field activities. The records and procedures most applicable to field activities are summarized in field logbooks, field data sheets, and field data management.

Data that are generated during sample collection and sample preparation will be manually entered into the field logbook and field data sheets. Data from these sources will be entered into an Excel® workbook template directly from the field logbook. These data include station location coordinates, station names, sampling dates, sample identification codes, and additional station and sample information (e.g., water depth, sample type). A second individual will review all entries for accuracy and completeness, and any errors will be corrected before the data are uploaded to the STORET database and approved for release to data users.

B9.2 Laboratory Data

A wide variety of manually entered and electronic instrument data are generated at the laboratories. Data are manually entered into:

- Standard logbooks
- Storage temperature logs
- Balance calibration log
- Instrument logs
- Sample preparation and analysis worksheets
- Maintenance logs
- Individual laboratory notebooks
- Results tables for conventional analyses.

The LIMS is the central data management tool for each laboratory. All manual data entry into the LIMS is proofed at the laboratory. All data collected from each laboratory instrument, either manually or electronically, are reviewed and confirmed by analysts before reporting. The LIMS is used for every aspect of sample processing, including sample log-in and tracing, instrument data storage and processing, generation of data reports for sample and QC results, and preparation of electronic data deliverables (EDDs).

Laboratory data will be entered directly into the STORET database from the EDD. A database printout will be used to verify database entries against the hard-copy laboratory data packages.

C. ASSESSMENT AND OVERSIGHT

C1. ASSESSMENTS AND RESPONSE ACTIONS

Readiness reviews are conducted to ensure that all necessary preparations have been made for efficient and effective completion of each critical phase of project work. Field readiness reviews will be conducted prior to initiation of each field sampling event. The field coordinator will verify that all field equipment is ready for transfer to the site. Any deficiencies noted during this readiness review will be corrected prior to initiation of sampling activities.

A readiness review will also be completed before final data are released for use for each annual report. The data manager will verify that all results have been received from the laboratory, data validation and data quality assessment have been completed for all of the data, and data qualifiers have been entered into the database and verified. Any deficiencies noted during this review will be corrected by the data manager, the QA manager, or their designee. Data will not be released for final use until all data have been verified and validated. No report will be prepared in conjunction with the readiness reviews. However, the project manager and data users will be notified when the data are ready for use.

Technical review of intermediate and final work products generated for this project will be completed throughout the course of all sample collection, laboratory analysis, data validation, and data management activities to ensure that every phase of work is accurate and complete and follows the QA procedures outlined in this QAPP. Any problems that are encountered will be resolved between the reviewer and the person completing the work. Any problems that cannot be easily resolved or that affect the final quality of the work product will be brought to the attention of the ADEC project managers. ADEC will be notified of any problems that may affect the final outcome of the project.

The laboratory has implemented a review system that serves as a formal surveillance mechanism for all laboratory activities. The analyst initially verifies the accuracy of the data and conformance of calibrations and QC results to control limits. A second review of sample, calibration, and QC results is conducted by the section supervisor, a senior chemist, or other qualified personnel, as designated by the laboratory. Details are provided in the laboratory QA manual (Appendix A).

Any project team member who discovers or suspects a non-conformance is responsible for reporting the non-conformance to the project manager or the QA manager, as applicable. The project manager will ensure that no additional work dependent on the non-conforming activity is completed until a confirmed non-conformance is corrected.

When a non-conformance is identified, a corrective action plan will be prepared. The plan will include identifying the corrective action, the person or organization responsible for

implementing the corrective action, and procedures for confirming that the desired results are produced. The corrective measures will be appropriate to the severity of the non-conformance and realistic in terms of the resources required for implementation.

C2. REPORTS

Laboratory data packages and EDDs will be prepared by the laboratory upon completion of analyses for each sample delivery group. The case narrative will include a description of any problems encountered, control limit exceedances, and a description and rationale for any deviations from protocol. Copies of corrective action reports generated at the laboratory will also be included with the data package.

Monthly monitoring reports with the water quality analytical results and field notes from the previous month's sampling event will be submitted electronically to ADEC. Quarterly reports submitted to ADEC electronically will include the analytical results from the surface water quality monitoring sites, compliance wells, and other groundwater quality monitoring well sites. The quarterly reports will also include tabulated summaries of the analytical results, field parameter measurements, and visual observations. An annual report will be submitted as a hard copy and electronically to Alaska Department of Natural Resources and ADEC. The annual report will include:

- Summary of analytical results
- Summary explanation of water quality results
- Statistical comparison of surface water and groundwater results
- Summary of monitoring results
- Water quality trends, if present, in graphical form
- Volumes of NAG and PAG rock
- Project progress
- Work proposed for the next year
- Any foreseen changes to the *Plan of Operations*.

NMC will notify ADEC within 30 days if site-specific water quality standards are exceeded during a sampling event. The notification will include evaluation of the magnitude and extent of the exceedance, and assessment of whether migration from the disposal or treatment facilites was the cause of the change in water quality.

All records and information resulting from monitoring activities required in the permit including all records of analyses, calibration records, quality control documentation, field sampling logs, and recordings from continuous monitoring instrumentation, shall be retained in NMC's project office in Alaska for three years for review by ADEC.

D. DATA VALIDATION AND USABILITY

Data generated in the field and at the laboratories will be verified and validated according to criteria and procedures described in this section. Data quality and usability will be evaluated, and a discussion will be included in the applicable quarterly and annual reports.

D1. DATA REVIEW, VALIDATION AND VERIFICATION REQUIREMENTS

Field and laboratory data for this project will undergo a formal verification and validation process. All entries into the database will be verified. All errors found during the verification of field data, laboratory data, and the database will be corrected prior to release of the final data.

Data verification and validation will be conducted in accordance with *Guidance on Environmental Data Verification and Validation* (USEPA 2002a). Data verification and validation for conventional and inorganic analyses will be completed according to methods described in the EPA guidance for data review (USEPA 2002b). Performance-based control limits established by the laboratory and control limits provided in the method protocols will be used to evaluate data quality and determine the need for data qualification. Laboratory control limits for LCS/LCSDs and MS/MSDs are provided in Table B5-2 and will be used for data validation.

Results for field splits and replicates will be evaluated against a control limit of 50 RPD. Data will not be qualified as estimated if this control limit is exceeded, but RPD results will be tabulated, and any exceedances will be discussed in the annual report. Equipment rinse blanks will be evaluated and data qualifiers will be applied in the same manner as method blanks, as described in the applicable EPA guidance documents for data review (USEPA 2002a,b).

Data will be rejected if control limits for acceptance of data are not met, as described in USEPA (2002a,b).

D2. DATA EVALUATION

Monitoring data will be evaluated to determine whether concentrations of metals and other constituents in downgradient groundwater and surface water are equivalent to natural conditions. Compliance samples will be taken from groundwater wells and surface water locations downgradient from the treated wastewater land application area. Natural conditions for groundwater will be defined by the combined dataset from wetland groundwater monitoring conducted before commencement of any exploratory activity and from monitoring during the project of a reference location located outside of the area of potential influence of project activities. Natural conditions for surface water will be defined by the combined dataset from surface water monitoring conducted before exploratory activity and from ongoing

monitoring of upstream reference locations. The data sets defining natural conditions will therefore be based on six months or more of monthly pre-construction groundwater and surface water sampling, and will grow in size and seasonal coverage as the project proceeds. Groundwater and surface water compliance data will be analyzed separately, and in comparison to the appropriate reference data set. These data sets will provide a sound basis for statistical comparisons between natural conditions and compliance monitoring samples.

Systematic variation in natural conditions (i.e., seasonality) will be distinguished from random (non-systematic) variation. Seasonality will be evaluated after monthly samples have been collected for at least six months from monitoring locations sampled before construction or from reference locations. Both statistical methods (e.g., regression) and visual methods (e.g., examination of scatter plots) will be used to evaluate whether seasonality may be present. Evaluation of the potential influence of seasonality will be carried out after each sampling event throughout the first year when at least six monthly samples are available. If seasonality appears to be present, then data from each compliance sample will be compared to data from reference samples (including pre-construction samples as appropriate) that were collected during a three-month period representative of the season in which the compliance well is sampled. Data from the same season and multiple years will be pooled after the first year of data has been collected. If seasonality is not apparent, then data from each compliance sample will be compared to all pooled data from reference and pre-construction sample.

Data from compliance and reference samples will be compared using a one-sided Student's *t*-test or a non-parametric equivalent, using a false positive rate (alpha) of 0.05, which is equivalent to a 95 percent confidence level. The pool of data from reference samples (and pre-construction samples, if appropriate) will be used to establish the statistical distribution of each monitoring parameter, and data from each compliance sample will be tested to determine if it is statistically likely to have come from that distribution or from some alternate distribution with a mean value (higher or lower, depending on the parameter) that is likely to indicate changes in wetlands water quality due to effluent discharge. Outliers will be identified using the methods described in ADEC (2006), and eliminated only if they are attributable to unusual conditions during sample collection or analysis. If a parameter is not detected in some of the reference samples, the mean and standard deviation of the reference distribution will be determined using the methods described by Helsel (2005) to account for the non-detects. If reference data do not conform to a normal (Gaussian) distribution, either an appropriate data transformation will be made before the *t*-test is applied, or a non-parametric test will be applied (e.g., a Wilcoxon one-sample signed rank test). The use of data transformations and parametric tests will be favored over the use of nonparametric tests, because the former have greater statistical power to detect differences. These steps will provide an assessment of whether or not each parameter in each compliance sample is statistically different from the set of reference samples.

Because the chosen confidence level allows a 5 percent false positive rate for each test, and many tests will be conducted (considering both analytes and sampling events), the statistical

tests for individual analytes will be augmented by additional decision rules to account for the effect of false positives. For example, statistical differences for a single analyte in three successive samples (ADEC 2006), or statistically significant differences for three analytes in the same compliance sample are likely to be strong evidence for systematic deviation from natural conditions.

D3. VALIDATION AND VERIFICATION METHODS

Field data will be verified during preparation of samples and COCs. Field data and COCs will be reviewed on a daily basis. After field data are entered into the project database, 100 percent verification of the entries will be completed to ensure the accuracy and completeness of the database. Any discrepancies will be resolved before the final database is release for use.

In addition to verification of field and laboratory data and information, data qualifier entries into the database will be verified. Any discrepancies will be resolved before the final database is released for use. The accuracy and completeness of the database will be verified at the laboratory and again as part of data validation. All entries to the database from the laboratory EDDs will be checked against the hard-copy data packages.

D4. RECONCILIATION WITH USER REQUIREMENTS

The goal of data validation is to determine the quality of each data point and to identify data points that do not meet the project criteria. Non-conforming data may be qualified as undetected, estimated, or rejected as unusable during data validation if criteria for data quality are not met. Rejected data will not be used for any purpose. An explanation of the rejected data will be included in the quarterly and annual reports.

Data qualified as estimated will be used for all intended purposes and will be appropriately qualified in the final project database. These data may be less precise or less accurate than unqualified data. The data users will evaluate the effect of the inaccuracy or imprecision of the qualified data.

E. REFERENCES

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