Quality Assurance Project Plan for

Water Quality Monitoring of Surface Waters Within the Pyramid Lake Indian Reservation, Nevada

Prepared for:
U.S. Environmental Protection Agency, Region IX
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Date:
January 20, 2011

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Wayne Burke, PLPT Tribal Chairman	**Date**
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John Mosley, Environmental Department Director	**Date**
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Daniel Mosley, Pyramid Lake Fisheries Director	**Date**
_____	_____
Fannie Ely, Project Manager/QA Officer	**Date**
_____	_____
Donna Noel, QA Manger	**Date**
_____	_____
Tiffany Eastman, USEPA Project Officer	**Date**
_____	_____
Eugenia McNaughton, USEPA QA Manager	**Date**

Contents

1.0 Project Management

This Quality Assurance (QA) Project Plan has been prepared for the monitoring of surface water by the Pyramid Lake Paiute Tribe (PLPT) on the Pyramid Lake Indian Reservation (PLIR). The surface water monitoring program is part of the PLPTs water quality management program developed under Section 106 of the Clean Water Act. This section of the QA Project Plan describes how the project will be managed, organized and implemented.

1.1 Title and Approval Page- See page 1.

1.2 Table of Contents- See page 2.

1.3 Document Distribution List

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1.4 Project Organization

The Pyramid Lake Paiute Tribe (PLPT) is the responsible agency for the surface water quality monitoring program. The participating agency is the U.S. Environmental Protection Agency, Region 9 (USEPA). The Pyramid Lake Paiute Tribe's Water Quality Laboratory is the laboratory that will be performing the chemical and microbiological analyses for the monitoring program following commonly used analytical methods. The PLPT Water Quality Laboratory is located in Sutcliffe, Nevada. If, in the future, the PLPT WQ Laboratory is no longer the laboratory and a new laboratory is selected, this Quality Assurance Project Plan (QAPP) will be amended accordingly.

The roles and responsibilities are described below. All contact information can be found in the Document Distribution List above. An organizational chart for the project, shown in Figure 1, illustrates relationships and lines of communication among participants and data users.

Tiffany Eastman, US EPA Project Officer
Will provide overall grant oversight, and work with the Project Manager to ensure grant adherence, reviewing and approving the work plan and reports for this project.

Eugenia McNaughton, US EPA QA Program Manager
The US EPA QA Program Manager is responsible for providing QAPP review and guidance for the PLPT and final document approval.

Fannie Ely, Project Manager/QA Officer
The project manager/QA officer is the responsible official who will oversee the WQ monitoring program and budget for this project. She will provide guidance, ensuring technical quality/adherence/compliance, and overall development of the sampling design and protocols. She will oversee training of personnel on QC requirements and procedures. She will be responsible for ensuring that any amended versions of the QA Project Plan are distributed to the organizations and individuals on the Distribution List. As QA Reviewer, she will provide review and guidance, reviewing QA/QC plans for completeness and noting errors and inconsistencies. Since the PLPT is not large enough to support a full-time QA Officer, she will function in the dual role as Project Manager and QA Reviewer. Fannie has a BS degree in Geography, and experience conducting sampling and analysis of water samples for the PLPT since 2004.

WQ Laboratory Manager
The Laboratory Manager is the responsible official who will serve as the primary contact and provide oversight for all laboratory-related activities. The Laboratory Manager will coordinate annually with Pyramid Lake Fisheries and PLPT Specialists/Technicians to review laboratory roles and responsibilities, sampling and field measurement requirements, analytical requirements, sampling schedule, and requirements for field and laboratory documentation to minimize potential problems that could occur during the sampling season. The Laboratory Manager will provide oversight for water sample

preparation and analytical activities within the WQ laboratory, and review analytical data to ensure it is consistent with the QA/QC program defined in this QA Project Plan. The Laboratory Manager is responsible for the QA/QC Quality Assurance/Quality Control (QA/QC) review of all data generated for the samples collected.

This position is currently empty; please contact Project Manager/QA Officer with questions regarding Laboratory Manager responsibilities.

Donna Noel, QA Manager
She will receive all data reports from the analytical laboratory and will be their main contact regarding data quality/control issues and concerns. She will notify the Project and/or Laboratory Manager if there is any knowledge that standard operating procedures are not being followed. Donna has a BS in Bacteriology and Chemistry and a MS in Metallurgical Engineering. She has 10 years of laboratory supervisor and QA/QC experience.

Field Samplers
Beverly Harry, Environmental Manager
Chris Katopothis, Environmental Specialist
Fannie Ely, Environmental Specialist
Bonita Natonabah, Environmental Technician
Sarah Mandell, Resource Technician
Nancy Vucinich, Environmental Technician

The individuals listed above have the primary responsibility for performing sample collection and field measurement activities. They will enter data and conduct data/QC analysis. Each person has at least three years experience in collecting water quality data.

1.5 Problem Definition/Background

1.5.1 Background

The Pyramid Lake Paiute Indian Reservation (PLIR) is located 35 miles northeast of Reno - Nevada, in rural Washoe County, Nevada. The PLIR encompasses approximately 475,000 acres or 742 sq. miles, about 25% is surface water. The PLIR land is held in trust by the United States government, less than 1% remains in “fee” status. The PLPT is comprised of approximately 2,300 tribal members, 1,600 of whom live on the Reservation. The PLPT is a federally recognized Tribe.

The Pyramid Lake Paiute Tribe has a 56% employment rate, the rest being unemployed, retired, or ranchers. The majority of the reservation resident population is young, comprised of individuals under age thirty-five (35) years. The median age is twenty-two (22) years. Much of the economy on the Pyramid Lake Reservation is centered on fishing and recreational activities at Pyramid Lake. In addition to permit fees for fishing, day use and overnight camping; the Tribe also receives lease revenue, and tax revenue. Several tribal members belong to the Pyramid Lake Cattleman's Cooperative Association and the Association utilizes the reservation under a range management plan to operate and manage livestock owned by individual tribal members.

The Truckee River, which originates in the Truckee River Watershed (Figure 2), flows east for approximately 125 miles from the spill-way at Lake Tahoe, through the cities of Reno and Sparks, to its

terminus in Pyramid Lake. Pyramid Lake is 114,000 surface acres and classified as a mesotrophic, monomictic terminal lake. Pyramid Lake and 31 miles of the lower Truckee River is located within the exterior boundaries of the PLIR in Northwest Nevada.

Surface water resources within the Reservation include 31 miles of the lower Truckee River, all of Pyramid Lake, 12 perennial streams, and many intermittent creeks, ephemeral washes, seeps, springs and wetlands (Figure 3).

The Paiute people (Numa) of Pyramid Lake were traditionally fishermen, hunters, and gatherers. The original name of the Pyramid Lake Paiute is "Cui-ui Dicutta," meaning Cui-ui eater, which speaks of the Tribe's dependence upon Cui-ui for food. Quality of life for the Numa has decreased over the years due to the affects of growth and urbanization. Upstream water diversions and subsequent low flows have affected recovery efforts of two Pyramid Lake fish species important to the Tribe's culture and traditional way of life. The Lahontan Cutthroat Trout (LCT) and Cui-ui (chasmistes cujus) are listed as threatened and endangered by the USFWS.

The Cui-ui (a lake sucker) is a long-lived fish, living up to 50 years. Historically, Cui-ui have been known to spawn farther upstream but now spawn primarily in the Truckee River portion of the PLIR. The PLPT maintains a Cui-ui hatchery in Sutcliffe, Nevada and the Cui-ui successfully spawn in the river when flows are greater than 1000 cubic feet per second from March through June.

Blocked by dams and confined to the main stem of the lower Truckee River, the few LCT who manage to migrate out of the lake have very limited spawning habitat and no fry-hatch success in the river. Therefore, all Pyramid Lake LCT are maintained by three hatcheries and hand-spawned by fishery technicians. Historically LCT migrated up the Truckee River in great numbers on their way to upper Truckee River watershed tributaries. Today there is very little evidence of spawning LCT even in the best of years in the lower Truckee River.

1.5.2 Problem Definition

The Truckee River and Pyramid Lake are important cultural resources to the PLPT. These two water bodies are integral to the Tribe's cultural and economic life, therefore any current or potential future impairment to aquatic life needs to be identified.

The Cities of Reno/ Sparks, Nevada currently have a permit to discharge up to 40 million gallons per day of tertiary-treated effluent into Steamboat Creek by Reno's wastewater treatment plant, which then flows into the Truckee River. The North Truckee Drain (from agricultural return flows) enters the Truckee River just upstream of the confluence of the Steamboat Creek and the Truckee River. Urban Storm runoff is another nonpoint source of pollution (NPS). These point & nonpoint sources of pollution enter the Truckee River about 25 -30 miles upstream of the PLIR boundary, and all have negative impacts on aquatic life, especially the threatened and endangered fish.

Washoe County is one of the fastest growing counties in the U.S., and the cities of Reno/ Sparks are currently looking into increasing their discharge of effluent into the Truckee River. More Industrial companies are building along the Truckee River corridor, adding the potential of NPS pollution.

Derby Dam is located about 10 miles upstream of the PLIR boundary. Built in 1906, it is operated by the U.S. Bureau of Reclamation to divert Truckee River water out of basin to Churchill County for agricultural

use. Diversions in drought years results in low flows, compounded by upstream point and nonpoint sources of pollution, take their toll on aquatic life. Channelization and cutting down of mature cottonwood trees along the lower Truckee River 1960's by U.S. ACE, extending to the PLIR have contributed to a degraded spawning habitat and living conditions for salmonids in the lower Truckee River.

In 1994, the PLPT developed a Nonpoint Source Management plan identifying NPS concerns within the PLIR. Most of these concerns have been mitigated by fencing the lower river from livestock, laser leveling agricultural fields to reduce runoff, and by taking many homes off septic systems. The current plan is under revision and should be completed in 2011.

The PLPT is concerned about the effects of current land use (e.g., septic systems, livestock, agriculture, upstream urbanization and point/ nonpoint sources of pollution, etc.) may have on aquatic life in the Truckee River and Pyramid Lake. To date, however, there has not been an adequate biological assessment of the quality of the Reservation's surface waters or evaluation of potential sources of contamination.

Section 101(a) of the CWA states the "objective of the act is to restore and maintain the chemical, physical, and biological integrity of the nation's waters…" States and Tribes have a fairly good understanding of the chemical and physical conditions, but have just recently began developing bioassessment programs to assess biological conditions of their surface waters.

For this reason, the PLPT through the Pyramid Lake Fisheries WQ laboratory and Resource Management program began conducting nutrient analysis of waters within the PLIR in 1985.

The PLPT's goal is to 'maintain' the biological, chemical and physical integrity of surface waters and riparian areas within the PLIR, with a long-term goal of improving WQ conditions for the benefit of aquatic life, and especially endangered and threatened fish dependent upon Pyramid Lake and the Lower Truckee River.

The purpose for this QAPP is to adopt standardized protocols for conducting long-term water quality monitoring of surface waters within the PLIR. Data generated from this study will be used to assess aquatic health/ conditions, and monitor/ evaluate trends. Data generated will also be used to establish or determine if water quality standards are being achieved. Best management practices will be incorporated to improve the biological integrity, riparian health, and water quality of a water body. These decisions will be the role of the Project Manager who receives direction from the Environmental, Pyramid Lake Fisheries, and Water Resources directors, managers, specialists and Tribal Council representatives during monthly Tribal interdisciplinary team (TIDT) meetings.

Biological monitoring of surface waters will also be conducted under a separate QA Plan. Long-term biological monitoring data will provide information to help the PLPT establish an Index of Biological Integrity (IBI) and biocriteria standards, which can later be implemented into Tribal regulations and ordinances for the Pyramid Lake Paiute Indian Reservation.

All water quality data including previously collected field and laboratory data is stored within the PLPT Environmental Department. This data has been collected since 1985 and includes chemical, physical, and biological data. Continuing surface water monitoring is needed to provide information of current conditions, as well as to track changes in water quality over time.

1.6 Project/Task Description and Schedule

Water quality monitoring is conducted by Tribal Environmental Water Quality staff and Pyramid Lake Fisheries staff.

There are five monthly sampling sites along the Truckee River and three non-point source sites that are included in the Truckee River monthly monitoring events, Figure 4. There are eleven annual sampling sites along the Truckee River. These sites correspond with the bioassessment monitoring activities described in the Bioassessment Monitoring in Surface Waters of the Pyramid Lake Indian Reservation, Nevada: Pyramid Lake Paiute Tribe Stream Bioassessment Procedure (QA EPA Office Document Control Number: WATR0568QV3), Figure 5.

There are thirteen perennial stream sample sites, Figure 6 - Figure 8, and two monitoring stations for Pyramid Lake, Figure 9.

Sampling locations are accessible either by boat, truck (2 or 4-wheel drive), or hiking. All sampling locations have been previously recorded using global positioning system equipment. See Table 14 for GPS coordinates for each sampling location. Other water bodies may be sampled when time permits.

Water samples will be taken immediately to the Tribe's laboratory for preservation after collection. Analysis will be conducted for the following parameters: total ammonia, nitrate + nitrite, total kjeldahl nitrogen, total phosphate and orthophosphate. A YSI Model 6920V2 will be used to measure temperature, pH, specific conductivity, total dissolved solids, and dissolved oxygen or a SEACAT Profiler SBE 19plus, for lake sampling, will be used to measure the following in-situ parameters: temperature (C°), pH, conductivity, dissolved oxygen (DO), and chlorophyll.

Monitoring of Truckee River/NPS will be conducted monthly. Monitoring of Pyramid Lake station 96 will be conducted monthly and both Pyramid Lake monitoring stations 96 and 93 will be sampled quarterly (February, May, August, and November). Annual monitoring of the physical habitat and bioassessment sites along the Truckee River will be conducted in late August to early September; timing depends on the flows of the river and corresponds with low flow. Monitoring of the mountain streams will be conducted annually along with the physical habitat and bioassessment activities, this occurs in spring (April/May) depending on water flows due to winter precipitation.

It is expected to take one day a month to monitor Pyramid Lake and the Truckee River each, one week to complete the mountain stream sampling, and one week to complete the monitoring of the bioassessment sample sites.

The monthly monitoring schedule for the Truckee River/NPS and Pyramid Lake is as follows:

First week:	Collect/preserve water samples.
	Analyze water samples at WQ laboratory.
Third week:	Evaluate WQ data, and then enter results into database.
Fourth week:	Summarize & tabulate data, and include in monthly and Quarterly Reports.
	Submit monthly reports to Tribal Council and annual report to EPA.

The annual monitoring schedule for the Mountain Streams is as follows:

First week:	Collect/ preserve water samples from mountain stream sites. Analyze water samples at WQ laboratory.
Second week:	Evaluate WQ data, and then enter results into database.
Fourth week:	Summarize & tabulate data, and include in monthly and Quarterly Reports. Submit monthly reports to Tribal Council and annual report to EPA.

The annual monitoring schedule for the Truckee River Bioassessment is as follows:

First week:	Collect/ preserve water samples from BA sampling sites. Analyze water samples at WQ laboratory.
Third week:	Evaluate WQ data, and then enter results into database.
Fourth week:	Summarize & tabulate data, and include in monthly and Quarterly Reports. Submit monthly reports to Tribal Council and annual report to EPA.

1.7 Quality Objectives and Criteria for Measurement Data

This section describes the objectives of the project (i.e., decision or study questions to be answered), identifies the targeted action limits/levels, and defines the measurement performance or acceptance criteria deemed necessary to meet these objectives.

1.7.1 Objectives and Project Decisions

The surface water monitoring program is designed to characterize the surface water resources of the PLPT. The continued monitoring will provide valuable information about the current condition of the waters within the PLIR. Ongoing monitoring will allow the PLPT to continue to track changes in water quality over time and help to assess potential future environmental impacts to the PLIR surface waters.

The Pyramid Lake Paiute Tribal Council adopted numeric and narrative Water Quality Standards (WQS) for Pyramid Lake and the Truckee River, and narrative standards for tributaries to those water bodies by resolution on September 19, 2008. The PLPT received final approval on the Water Quality Control Plan (WQCP) on December 19, 2008, from EPA. These water quality standards will be used to evaluate the quality of water and serve as the Project Action Limits (PALs). Data collected will be compared to these approved standards presented as PALs in Table 1 and Table 2. Data collected from the mountain streams will be compared to the WQS for the Truckee River as a guideline.

Decisions to be made with the data include:

- If data for an analyte or field parameter (from an individual location or single sampling event) are found to exceed the PLPT WQS, then the Tribal IDT will be notified.
- If data are found to exceed the PLPT WQS for three consecutive sampling events and/or appear to be increasing with time, then the Tribal IDT and Tribal Council will be notified and a plan for future investigations of potential sources will be discussed.
- If waters flowing onto the reservation are impaired the issue will be brought to the attention of the Tribal IDT and Tribal Council for possible discussion with the US EPA Project Officer.

1.7.2 Action Limits/Levels

Table 1 and Table 2 provide a listing of the parameters to be sampled and the associated PALs. The information demonstrates that the analytical methods selected for this project are capable of providing data with quantitation limits (QL) reported to concentrations lower than the PLPT WQS for the majority of the parameters of interest, and therefore, the data generated will be able to support sound decisions at the PALs. In addition, Table 1 and Table 2, provide a summary of the laboratory's analytical detection limits (DL), those minimum concentrations that can be detected above instrumental background or baseline/signal noise, providing further assurance that the analytical methods are capable of meeting the data needs of the project in terms of sensitivity.

Table 1 and Table 2 provide additional information related to the field measurements to be conducted. The QLs listed, as well as the measurement ranges associated with each field parameter, based on information provided in the respective equipment manufacturers identified in Table 3, are deemed acceptable to meet the project objectives.

1.7.3 Measurement Performance Criteria/Acceptance Criteria

In order to support project decisions, data generated must be of known and acceptable quality. To define acceptable data quality for this project data quality indicators (DQIs) were identified for each analytical parameter, and decisions were made regarding how each DQI would be assessed. The DQIs include: precision, accuracy/bias (as related to percent recovery and contamination), representativeness, comparability, completeness and sensitivity.

The general approach to assessing each DQI is described below. Some DQIs will be assessed quantitatively, while others will be assessed qualitatively. For quantitative assessments, example calculations have been provided and the QA samples (to assess each DQI) have been identified.

The frequency of the QC samples and the measurement performance criteria for each QC sample for each type of analysis are provided in Table 5. For quantitative assessment of laboratory methodology, the laboratory's Data Quality Indicator Tables (Appendix D), and analytical SOPs have been reviewed by the Tribe's project team, and the associated laboratory QC (types & frequencies of QC samples and QC acceptance limits) have been determined to be adequate to meet the data quality needs of the project. As such, the laboratory QC has been accepted as the project's measurement performance criteria for the analytical component, while project specific criteria have been defined to assess the field-sampling component.

For field measurement, the associated acceptance criteria (types and frequencies of QC checks and acceptance limits) for the project are summarized in Table 4.

General Approach:

Precision- Precision will be assessed quantitatively with duplicate samples and expressed as relative percent difference (RPD) by the following equation:

$$RPD\ (\%) = \frac{|X_1 - X_2|}{(X_1 + X_2)/2} \times 100$$

where,

RPD (%) = relative percent difference
X_1= Original sample concentration
X_2= Duplicate sample concentration
$|X_1 – X_2|$ = Absolute value of $X_1 – X_2$

To assess precision associated with all steps of the project (from sample collection through analysis) field duplicates will be collected and analyzed. Field duplicates will be collected at a frequency of 10% (1 duplicate/10 field samples) for each analytical parameter and 5% (1 duplicate each of 2 days/10 field samples) for each field measurement parameter. To assess laboratory precision alone, laboratory duplicates will be prepared and analyzed at a 5% frequency.

Accuracy/Bias- Accuracy/bias will be assessed as related to recovery, as well as in regards to potential contamination sources. Both of these terms will be evaluated quantitatively.

Accuracy/bias related to recovery is an assessment of the laboratory analytical methods alone. For laboratory control samples (LCS), it will be expressed as % Recovery from the expected concentration by the following equation.

$$\% \text{ Recovery} = \frac{X}{T} \times 100$$

where,

X = Measured concentration
T= Expected (True) concentration

or, for Matrix Spike (MS) samples, by the following equation:

$$\% \text{ Recovery} = \frac{(B-A)}{T} \times 100$$

where,

B= Measured concentration of spiked sample
A= Measured concentration of unspiked sample
T= Expected (True) spiked concentration

The frequency of the LCS and/or MS samples associated with the analytical parameters will be one for every 20 samples or 5%. No LCS or MS samples will be analyzed as part of the field measurements.

Accuracy/bias as related to contamination involves both a field sampling and laboratory component. To assess all steps of the project (from sample collection through analysis), field blanks will be collected and analyzed. Field blanks are planned to be collected at a frequency of 5% (or 1 blank/20 field samples) for analysis. To assess potential laboratory contaminant sources alone, laboratory blanks will be prepared and analyzed at one per batch or 5% frequency. No blanks will be analyzed as part of the field measurements.

Another way to measure accuracy is through the use of performance evaluation samples. These are samples containing analytes whose concentration is known to the tribe, but not to the laboratory.

However, submission of performance evaluation samples to the laboratory is presently outside the scope and budget of the tribe's water monitoring program. It is also felt that, given the planned use of the data by the tribe for its internal purposes, that performance evaluation samples are not warranted at this time. If performance evaluation samples are deemed necessary in the future, the Tribe would acquire the samples from commercial sources and would rely on the preparer of the samples to establish acceptance criteria, whether that were EPA, the state, or a commercial supplier.

Representativeness - Representativeness, or the ability of a sample to represent the environmental conditions at the time of collection, will be assessed both quantitatively and qualitatively.

To assess this term quantitatively, an overall evaluation will be made of how well the precision and accuracy/bias assessments met their associated measurement performance criteria. An additional assessment will involve ensuring that a temperature blank sample has accompanied each cooler of samples that has a temperature requirement associated with its preservation (Table 6) and that the temperature of these temperature blank samples are 4°C ± 2°C when received at the laboratory.

To assess this term qualitatively, no actual QC samples are involved. Instead, the evaluation will involve verifying that documented sample collection and analytical methods (including sample handling and chain-of-custody procedures, sample preservation, and sample holding time protocols) were followed.

The procedures identified throughout this QA Project Plan were chosen to optimize the potential for obtaining samples that reflect the true state of the environment, within practical limits. In addition, efforts were made to ensure samples would be collected so that the overall condition of all the Tribe's water can be assessed. Long-term monitoring will increase the representativeness of the project in that it would enable an assessment of changes over time. Basically, the more sampling events, the more statistically representative the collected data will be of the area.

Comparability- Comparability, or the degree to which data from different studies or methodologies agree, will be assessed qualitatively.

Comparability expresses the confidence with which one data set can be compared to another. It describes the ability and appropriateness of making collective decisions with two or more data sets. Many variables may affect the descriptive value of the data. These include:

- Variables of interest in each data set
- Use of common units
- Similarity of methods and QA
- Time frames
- Season
- Weather
- Equipment

These variables are addressed by describing the project objectives and activities planned under the project.

The analytical methods to be used by the PLPT WQ Laboratory will be EPA or Standard Methods, both well- documented and published methods for surface water analyses. In addition, the analytical reports will be in consistent units of measure, such as milligrams per liter (mg/l) or micrograms per liter (µg/l).

Table 6 lists the analytical parameters to be sampled and the methods to be used for the analysis, as well as the field measurements.

Completeness- Completeness, the amount of valid data obtained compared to the planned amount, may be assessed quantitatively and/or qualitatively. To assess the term quantitatively, % Completeness will be expressed by the following equation:

$$\% \text{ Completeness} = \frac{N}{T} \times 100$$

where,

N= Number of usable results

T= Total targeted number of samples planned to be collected

All data collected in this project will be used to determine the quality of surface water within the PLIR. Due to a variety of circumstances, sometimes not all samples scheduled to be collected can be collected (e.g., a creek may be dry, etc.) or the data from the samples cannot be used (e.g., samples bottles are broken in transit, sample holding times are grossly exceeded, etc.). For this surface water sampling project, the overall completeness goal has been set at 90% for each analytical parameter and field measurement type. If the completeness goal is not met, re-sampling and/or re-analyzing will be conducted.

At this point in time, no sampling locations have been deemed more critical to the overall project goal than any other. As such, there will be no qualitative assessment of completeness to ensure that samples from critical locations have been collected and their associated data has been deemed usable to support the project objectives.

Sensitivity- Sensitivity, or the ability of a method to detect and quantify an analytical parameter of concern at the concentration level of interest, will be assessed semi-quantitatively. No actual QC samples are involved. Instead, the laboratory to perform the analyses has provided their QLs and DLs and demonstrated that these are lower than the respective WQS serving as the project action limits, for the majority of the analytical parameter. For field measurements, the sensitivity is defined by the instrument manufacturers.

1.8 Special Training Requirements/Certification

1.8.1 Field Sampling and Measurement Personnel

No special training of field personnel is required for this project. The PLPT field personnel conducting the field activities are experienced staff members who have been supporting similar activities for many years.

1.8.2 Laboratory Personnel

No special training of laboratory personnel is required for this project. Laboratory training ensures that personnel performing designated tasks have participated in ongoing training associated with those tasks.

1.9 Documents and Records

1.9.1 QA Project Plan Distribution

It is the responsibility of the PLPT Project Manager to prepare and maintain amended versions of the QA Project Plan and to distribute the amended QA Project Plan to the individuals listed in Section 1.3.

1.9.2 Field Documentation and Records

In the field, records will be documented in several ways, including field logbooks, photographs, pre-printed forms, corrective action reports, and field audit checklists and reports. Field activities must be conducted according to the appropriate SOPs (Appendix A). It is the responsibility of the PLPT Project Manager to maintain updated revisions of SOPs at all times and to distribute updated SOPs to the PLPT field personnel, as appropriate. All documentation generated by the sampling program will be kept on file in the office of the PLPT Environmental Department.

1.9.2.1 Field Notebooks

Bound field logbooks will be used to record field observations, sampling site conditions, and on-site field measurements. These books will be kept in a permanent file in the office of the PLPT Environmental Department. At a minimum, information to be recorded in the field logbooks at each sample collection/measurement location includes:

- Sample location and description,
- Sampler's names,
- Date and time of sample collection,
- Designation of sample as composite or grab,
- Type of sampling equipment used,
- Type of field measurement instruments used, along with equipment model and serial number,
- Field measurement instrument readings,
- Field observations and details related to analysis or integrity of samples,
- Preliminary sample descriptions,
- Sample preservation,
- Lot numbers of the sample containers, sample identification numbers and any explanatory codes, and
- Name of recipient laboratory(ies).

In addition to the sample information, the following specific information will also be recorded in the field logbook for each day of sampling:

- Team members and their responsibilities,
- Time of arrival/entry on site and time of site departure,
- Other personnel on site,
- Deviations from the QAPP or SOPs required in the field, and
- Summary of any meetings or discussions with tribal, contractor, or federal agency personnel.

Separate instrument/equipment notebooks or logbooks will be maintained for each piece of equipment or instrument. These logbooks will be used to record field instrument calibration and maintenance information. Each logbook will include the name, manufacturer, and serial number of the instrument/equipment, as well as dates and details of all maintenance and calibration activities.

1.9.2.2 Photographs

Digital photographs will be taken at each sampling location and at other areas of interest near sampling area for every sampling event, except monthly monitoring sites; these will be photographed at least quarterly. The photographs will serve to verify information entered into the field logbook. Digital photographs will be archived in a permanent digital file to be kept in the office of the PLPT Environmental Program.

For each photograph taken, the following information will be written in the field logbook or recorded in a separate field photography logbook:

- Time, date, location, and weather conditions,
- Description of the subject photographed,
- Direction in which the picture was taken, and
- Name and affiliation of the photographer.

1.9.2.3 Labels

All samples collected will be labeled in a clear and precise way for proper identification in the field and for tracking in the laboratory. The samples will have identifiable and unique numbers. At a minimum, the sample label will contain the following information

- Sampling location or name,
- Unique sample number,
- Sample description (e.g., grab, composite),
- Date and time of collection,
- Initials/signature of sampler,
- Analytical parameter(s), and
- Method of preservation.

Each sample location will have a unique sample identification number. The unique sample ID will be two to three letters to represent the site followed by the date without spaces or for lake samples the station number and depth followed by the date. For example a sample taken at Pierson Dam on January 1, 2010 would be PD-01012010 and a sample taken at Station 96 at a depth of 10 m on January 01, 2010 would be ST9610-01012010. The two to three letter codes for each sample site can be found in Tables 8 - Table 11.

1.9.2.4 Field Quality Control Sample Records

Field QC samples (duplicates and blanks) will be labeled as such in the field logbooks. They will be given unique (fictitious) sample identification numbers and will be submitted “blind” to the laboratory (i.e., only the field logbook entry will document their identification and the laboratory will not know these are QC samples). The frequency of QC sample collection will also be recorded in the field logbook.

1.9.2.5 Sample Chain-of-Custody Forms and Custody Seals

Chain-of-custody forms will be provided by the laboratory (Figure 10). The forms will be used to document collection and shipment of samples for laboratory analysis. All samples will be accompanied by a chain-of-custody form. The forms will be completed and sent with each shipment of samples to the laboratory. If multiple coolers are sent to a laboratory on a single day, forms will be completed and sent with the samples for each cooler. The original form will be included with the samples and sent to the laboratory. Copies will be sent to the PLPT Project Manager.

The chain-of-custody form will identify the contents of each shipment and maintain the custodial integrity of the samples. Generally, a sample is considered to be in someone's custody if it is either in someone's physical possession, in someone's view, locked up, or kept in a secured area that is restricted to authorized personnel. Until the samples are shipped the custody of the samples will be the responsibility of the field personnel, who will sign the chain-of-custody form in the "relinquished by" box and note the date, time, and air bill number, if applicable.

Custody seals will not need to be used for this project, as the field personnel will transport the samples directly to the PLPT WQ Laboratory immediately after sampling.

Procedures for completion and distribution of the chain-of-custody forms are included in Appendix A.

1.9.3 Laboratory Documentation and Records

The analytical laboratory will keep a sample receiving log and all completed chain-of-custody forms submitted with the samples collected for this project. The analytical laboratory will also keep records of all analyses performed, as well as associated QC information, including: laboratory blanks, matrix spikes, laboratory control samples, and laboratory duplicates. Hard copy data of the analytical results will be maintained for six years by the laboratory.

The data generated by the laboratory for each sampling event will be compiled into individual data packages/reports. The data packages will include the following information:

- Project narrative including a discussion of problems or unusual events (including but not limited to the topics such as: receipt of samples in incorrect, broken, or leaking containers, with improperly or incompletely filled out chair-of-custody forms; receipts and/or analysis of samples after the holding times have expired; summary of QC results exceeding acceptance criteria; etc.),
- Sample results and associated QLs,
- Copies of completed sample receiving logs and chain-of-custody forms, and
- QC check sample records and acceptance criteria.

All data packages will be reviewed by the Laboratory Manager to ensure the accurate documentation of any deviations from sample preparation, analysis, and/or QA/QC procedures; highlights of any excursions from the QC acceptance limits; and pertinent sample data. Once finalized, the Laboratory Manager will submit them to the PLPT Project Manager. Any problems identified by the Laboratory Manager will be documented in the narrative of the Tribe's report.

1.9.4 Technical Reviews and Evaluations

As part of the QA efforts for the project, on-going technical reviews will be conducted and documented. These reviews are associated with both field activities and the data generated by the WQ Laboratory.

The PLPT Project Manager will observe selected sampling events to ensure that sample collection and field measurements are going according to plan. The results of the observations will be documented in a designated QA Audit Logbook. Once back in the office, the PLPT Project Manager will formalize the audit in a Field Audit Report to be forwarded to the PLPT Environmental Department Director and PLPT Field Personnel.

1.9.4.1 Corrective Action Reports (following Field Audits)

Corrective action reports will be prepared by the Field Personnel in response to findings identified by the PLPT Project Manager during field visits and audits. The reports will focus on plans to resolve any identified deficiencies and non-compliance issues that relate to on-going activities and problems of a systematic nature, rather than on one time mistakes. Corrective Action reports do not have a specific format, but will be handled as an internal memorandum.

1.9.4.2 Field Activities Review Checklist

At the end of each sampling event, a technical review will be conducted of field sampling and field measurement documentation to ensure that all information is complete and any deviations from planned methodologies are documented. This review is described in Section 3.1.1.3. The review, as well as comments associated with potential impacts on field samples and field measurement integrity, will be documented on a Field Activities Review Checklist (Figure 11).

1.9.4.3 Laboratory Data Review Checklist

Following receipt of the WQ laboratory's data package for each sampling event, the PLPT Project Manager will conduct a technical review of the data to ensure all information is complete, as well as to determine if all planned methodologies were followed and QA/QC objectives were met. The results of this review, as well as comments associated with potential impacts on data integrity to support project decisions, will be documented on a Laboratory Data Review Checklist (Figure 12).

1.9.5 Quarterly and Annual Reports

The PLPT Project Manager is responsible for the preparation of quarterly reports and annual reports to be submitted to the US EPA Grants Project Officer.

The quarterly report should include, at a minimum:

- Table summarizing the results (including both laboratory data and field measurements),
- Description of data submissions to WQX,
- Final laboratory data package (including QC sample results),
- Brief discussion of the field and laboratory activities as well as any deviations or modifications to the plans,
- Copies of Field Audit Reports and any associated Corrective Action Reports,

- Copies of Field Activities Review Checklists and Data Review Checklists,
- Discussion of any problems noted with the data, either from laboratory or field measurements,
- Discussion of any data points showing exceedences of Action Levels, and
- Recommendations/changes for the next sampling event.

The annual reports should include, at a minimum:

- Description of the project,
- Table summarizing the results (of all project data collected to date, including both laboratory data and field measurements),
- Description of data submissions to WQX,
- Final laboratory data package for the fourth quarter (including QC sample results),
- Discussion of the field and laboratory activities, as well as any deviations or modifications to the plans,
- Trends observed as a result of the year's monitoring efforts,
- Copies of Field Audit Reports and any associated Corrective Action Reports (for the fourth quarter),
- Copies of Field Activities Review Checklists and Data Review Checklists (for fourth quarter),
- Evaluation of the data in meeting the project objectives, including data exceeding Action Levels,
- Recommendations to the Tribal Council regarding exceedences which are occurring on an on-going basis, and
- Recommendations/changes for future project activities (e.g., adding/deleting sampling locations and/or analyses, modifications to SOPs, amendments to the QA Project Plans, etc.).

2.0 Data Generation and Acquisition

This section of the QA Project Plan describes how the samples will be collected, shipped, and analyzed.

2.1 Sampling Design

In 1989 the PLPT approached the University of California, Davis - Limnological Research Group (Dr. John E. Reuter and Dr. Charles R. Goldman) to help undertake the task of developing a reasonable and scientifically sound set of water quality standards, which when implemented would help protect the beneficial uses of Pyramid Lake and that portion of the Truckee River on Tribal land. This task included: evaluation of historical data, detailed limnological monitoring, field and laboratory experiments, limnological research, and modeling. Examples of topics investigated included, but were not limited to: measurement and evaluation of physical and chemical parameters, evaluation of nutrient and particulate matter, phytoplankton and zooplankton ecology, algal growth bioassays and nutrient limitation, measurement of surficial sediment composition, paleolimnology, measurement of primary productivity and algal biomass, internal and external loading of nutrients, development of nutrient budgets for carbon, nitrogen and phosphorus, estimates of sedimentation rates, evaluating susceptibility of Lake to anoxia, primary productivity and dissolved oxygen modeling, modeling of total dissolved solids concentration, nonpoint source management and assessment, and Lake and watershed management. The results of these studies have been published in a series of technical reports and peer review scientific publications.

The volumes entitled, Pyramid Lake, Nevada, Water Quality Study 1989-1993, Volume I - Limnological Data, Volume II - Limnological Description, Volume III - Nutrient Budgets, and Volume IV - Modeling Studies, have been widely distributed regionally and contain much of the information used for developing the Pyramid Lake standards. The first WQ Monitoring QA Project Plan and sampling design was developed as a result of this study.

The data collected will be used to: (1) assess water quality conditions; (2) determine whether water quality standards are being achieved; (3) provide a basis for evaluating watershed management strategies; and (4) improve the overall understanding processes which control water quality conditions.

Currently, the Tribe is conducting an extensive water quality and biological monitoring program through combined efforts of the Pyramid Lake Fisheries and Environmental Departments. At the same time, there is a large, regional effort to monitor the lower Truckee River lead by the State of Nevada and the Truckee Meadows Water Reclamation Facility (Cities of Reno and Sparks) with contributions by the University of Nevada – Reno, Desert Research Institute, and the US Geological Survey. All monitoring will be re-assessed as part of the triennial review of water quality standards.

2.1.1 Pyramid Lake

A total of two sampling stations have been previously identified for on-going monitoring activities. Sample locations, names, and rationale for selecting each sampling location are included in Table 7. Samples to be collected are summarized in Table 1.

All lake sampling locations are accessible using a boat powered by twin 160hp engines. All sampling locations were previously recorded using global positioning system (GPS) equipment.

Water quality monitoring for Pyramid Lake consists of monthly sampling at the deep index station (Way Point 96) as well as quarterly synoptic sampling at the shallow index station (Way Point 93), which is in the shallower south basin. Quarterly synoptic samplings will be conducted during winter mixing (February), the spring phytoplankton bloom (April-May), summer (August), and in the fall (November); the exact timing may vary from year to year.

Field measurements at both stations will include water column profiles using a SEACAT SBE19 plus which will record: temperature (C°), pH, conductivity, dissolved oxygen (DO), and chlorophyll. A profile of light intensity, Secchi depth, and zooplankton samples will also be taken.

Water samples will be collected from discrete depths for nutrient analyses. Nutrient analyses include: ammonia, nitrate+nitrite, total Kjeldahl nitrogen, orthophosphate, and total phosphorous.

2.1.2 Truckee River

A total of five monthly sampling stations along the Truckee River have been previously identified for on-going monitoring activities. Sample locations, names, and rationale for selecting each sampling location are included in Table 8. In addition there are eleven annual sampling stations along the Truckee River that have been identified. These sample locations, names, and rationale for selecting each sampling location are included in Table 9. Samples to be collected are summarized in Table 2.

All Truckee River sampling locations are accessible using a 4-wheel drive vehicle. All sampling locations were previously recorded using global positioning system (GPS) equipment.

Water quality monitoring for the Truckee River consists of monthly sampling at the five identified sample sites shown in Figure 4 and annual sampling of eleven sites identified in Figure 5.

Field measurements will be taken using an YSI Model 6920V2 instrument at all sites which measures: temperature, dissolved oxygen, pH, and specific conductivity, and turbidity.

Water samples will be collected at each site and analyzed for nutrients. Nutrient analyses include: ammonia, nitrate+nitrite, total Kjeldahl nitrogen, orthophosphate, and total phosphorus.

2.1.3 Non-Point Source

A total of four non-point source sampling sites have been previously identified for on-going monitoring activities, all sites occur along the Truckee River. Sample locations, names, and rational for selecting each location is included in 10. Samples to be collected are summarized in Table 2.

Non-point source monitoring consists of monthly sampling, in conjunction with the Truckee River sampling events. These sites are shown in Figure 4.

Field measurements will be taken using an YSI Model 6920V2 instrument at all sites which measures: temperature, dissolved oxygen, pH, and specific conductivity, and turbidity.

Water samples will be collected at each site and analyzed for nutrients. Nutrient analyses include: ammonia, nitrate+nitrite, total Kjeldahl nitrogen, orthophosphate, and total phosphorus.

2.1.4 Streams

A total of thirteen perennial stream sampling sites have been previously identified for on-going monitoring activities. Sample locations, names, and rational for selecting each location is included in Table 11. Samples to be collected are summarized in Table 2. If time permits, other streams located within the PLIR may also be sampled.

Stream monitoring consists of annual sampling, in conjunction with physical habitat surveys and bioassessment sampling events. This usually occurs in spring (April/May) depending on the annual snow pack and spring runoff rates. These sites are shown in Figure 6 - Figure 8.

Field measurements will be taken using an YSI Model 6920V2 instrument at all sites which measures: temperature, dissolved oxygen, pH, and specific conductivity, and turbidity.

Water samples will be collected at each site and analyzed for nutrients. Nutrient analyses include: ammonia, nitrate+nitrite, total Kjeldahl nitrogen, orthophosphate, and total phosphorous.

2.2 Sampling Methods

2.2.1 Surface Water Sampling

All samples will be collected using the field SOPs included in Appendix A. If an SOP is updated or revised, the updated or revised SOP will be used for the subsequent sampling event(s). Any revisions/updates to SOPs will be documented in an amendment to the QA Project Plan.

Water samples will be collected 6 - 12 inches below the water's surface, except Lake samples which will be collected at discrete depths. At each sampling location or discrete depth at lake monitoring stations, one-liter sample bottles will be filled for nutrient analysis. If a QC sample is to be collected at a given location, all containers designated for a particular analysis for both the sample and QC sample will be filled sequentially before containers for another analysis are filled.

All collected surface water samples will be placed in sample bottles/containers appropriate for nutrient analysis. Preservatives will be added at the laboratory, if required. Once the samples are collected and preserved, they will be stored at 4°C at the PLPT WQ Laboratory.

Care will be taken to not touch the lip of the sample bottle during the sample collection and preservation, so as not to potentially contaminate the sample. Table 6 summarizes the sample bottle/containers, volumes, and preservation requirements for each analysis and field measurement.

2.2.2 Zooplankton Sampling

All samples will be collected using the field SOPs included in Appendix A. If an SOP is updated or revised, the updated or revised SOP will be used for the subsequent sampling event(s). Any revisions/updates to SOPs will be documented in an amendment to the QA Project Plan.

Zooplankton samples will be collected at both the Pyramid Lake sampling stations at specified depths. All collected zooplankton samples will be placed in appropriate sample bottles/containers. Preservatives will be added at the laboratory. Once the samples are collected and preserved, they will be stored at 4°C at the PLPT WQ Laboratory.

Table 6 summarizes the sample bottle/containers and preservation requirements for zooplankton samples.

2.2.3 Field Health and Safety Procedures

A brief tailgate safety meeting will be held the first day of each sampling event to discuss emergency procedures (e.g., location of the nearest hospital or medical treatment facility), local contact information (e.g., names and telephone numbers of local personnel, fire department, police department), as well as to review the Tribe's Workplace Safety Program, Appendix B.

All field sampling activities will be conducted with a buddy system (i.e., two field personnel will constitute the sampling team. This will allow for the presence of a second person to provide assistance and/or call in an emergency or accident for the other field person, if/when needed.

Level D personal protective equipment (PPE) will be used when needed when collecting the surface water samples. At a minimum, safety glasses, plastic gloves, and sole-felt waders will be worn to avoid slipping on rocks and algae. Also, due to weather conditions during the sampling events and the possibility of health concerns (e.g., heat stress) from working in high temperatures, field personnel will

be advised to drink plenty of water and wear clothing (e.g., hat, long-sleeved shirt) that will cover and shade the body.

Potential routes of exposure related to field sampling and measurement activities are through the skin (e.g., from direct contact from the surface water) and/or by ingestion (e.g., from not washing up prior to eating). The use of Level D PPE, good hygiene, and following proper sampling procedures will minimize these potential exposures.

2.2.4 Field Measurements

Surface water samples will be analyzed at each sample collection location, except for Pyramid Lake, for the following field measurement parameters: pH, dissolved oxygen, conductivity, turbidity, and temperature. At the Pyramid Lake monitoring stations the following field measurement parameters will be analyzed: temperature (C°), pH, conductivity, dissolved oxygen (DO), and chlorophyll. Secchi disc reading and zooplankton samples will also be collected.

The measurement procedures are described in the SOPs included in Appendix A. Field measurements will be taken at each location prior to sample collection laboratory analysis. All field instruments will be calibrated (according to the manufacturer's instructions) at the beginning of each date of sampling and checked at the end of each day. Field instrument calibration and sample measurement data will be recorded in the field logbook.

2.2.5 Field Variances

As conditions in the field vary, it may become necessary to implement minor modifications to the sampling procedures and protocols described in this QA Project Plan. If/when this is necessary; the field personnel will notify the Project Manager of the situation to obtain a verbal approval prior to implementing any changes. The approval will be recorded in the field logbook. Modifications will be documented in the Quarterly Reports to the US EPA Grants Project Officer.

2.2.6 Decontamination Procedures

For the currently planned sample collection activities, samples will be collected directly into sample bottles/containers provided from the laboratory. As such, no field decontamination of these bottles (used as the sampling equipment) is necessary. The bottles will be provided and certified clean by the PLPT WQ Laboratory.

In the case that there is a need to collect surface water samples by one of the alternative methods (as discussed in the sampling SOP provided in Appendix A), decontamination of reusable sampling equipment coming in direct contact with the samples will be necessary. Decontamination will occur prior to each use of a piece of equipment and after use at each sampling location. Disposable equipment (intended for one-time use) will not be decontaminated but will be packaged for appropriate disposal. All reusable/non-disposable sampling devices will be decontaminated according to US EPA Region 9 recommended procedures using the following washing fluids in sequence:

- Non-phosphate detergent and tap water wash (using a brush, if necessary),
- Tap-water rinse, and
- Deionized/ distilled water rinse (twice).

Equipment will be decontaminated in a pre-designated area on plastic sheeting. Cleaned small equipment will be stored in plastic bags. Materials to be stored more than a few hours will also be covered.

2.2.7 Disposal of Residual Materials

In the process of collecting water samples for this project, various types of potentially contaminated wastes will be generated which may include the following:

- Used PPE,
- Disposable sampling bottles/containers or equipment,
- Decontamination fluids, and
- Excess water collected for sample container filling.

The USEPA's National Contingency Plan requires that management of the wastes generated during sampling comply with all applicable or relevant and appropriate requirements to the extent practicable. (Note: Although the National Contingency Plan does not strictly apply on tribal land, the PLPT feels that its requirements are reasonable and has adopted its policies.) Residuals generated for this project will be handled in a manner consistent with the Office of Emergency and Remedial Response (OERR) Directive 9345.3-02 (May 1991), which provides the guidance for the management of wastes. In addition, other legal and practical considerations that may affect the handling of the wastes will be considered, as follows:

- Used personal protective equipment (PPE) and disposable containers/equipment will be double bagged and placed in a municipal refuse dumpster. These wastes are not considered hazardous and can be sent to a municipal landfill. Any used PPE and disposable containers or equipment (even if it appears to be reusable) will be rendered inoperable before disposal in the refuse dumpster.
- Decontamination fluids generated in the sampling event could consist of deionized water, residual contaminants, and water with non-phosphate detergent. The volume and concentration of the decontamination fluid will be sufficiently low to allow disposal at the sampling area. The water (and water with detergent) will be poured onto the ground.
- Decontamination fluids generated in the sampling event could consist of deionized water, residual contaminants, and water with non-phosphate detergent. The volume and concentration of the decontamination fluid will be sufficiently low to allow disposal at the sampling area. The water (and water with detergent) will be poured onto the ground.
- Excess water collected for sample container filling will be poured onto the ground.

2.2.8 Quality Assurance for Sampling

Documentation of deviations from this QA Project Plan or applicable SOPs is the responsibility of the PLPT QA Officer. Deviations noted during the field audit will be documented in the QA Audit Logbook, recorded in the Field Audit Reports, and discussed in the Quarterly Reports.

Additional deviations from the QA Project Plan and/or SOPs may be implemented as field variances or modifications, as discussed in Section 2.2.4. These deviations will be communicated to the PLPT Project Manager by field personnel for approval. The approval will be recorded in the field logbook, and the modifications will be documented in the Quarterly Report.

2.3 Sample Handling and Custody

This section will describe the sample handling and custody procedures from sample collection through transport and laboratory analysis. It also includes procedures for the ultimate disposal of the samples.

2.3.1 Sample Containers and Preservatives

The PLPT Project Manager has worked directly with the PLPT Laboratory Manager to determine the number of sample containers, and associated sizes/volumes and materials, needed for this monitoring project. The containers will be provided pre-cleaned from the laboratory directly and require no washing or rinsing by the field personnel prior to sample collection. Sample bottles will not be pre-preserved. Preservatives (i.e., sulfuric acid for nutrient analysis) will be added at the laboratory if needed. Filtration is required for some samples; these samples will be filtered before preservatives are added.

2.3.2 Sample Packaging and Shipping

Water samples collected in the field will be immediately placed in a cooler. Water samples will be transported directly to the PLPT WQ Laboratory by the field team immediately following sample collection.

Daily, the field personnel will notify the Laboratory Manager of their sample transport schedule. The PLPT WQ Laboratory will be provided with the following information:

- Sampling department's name,
- Name and location of the site or sampling area,
- Name of project,
- Total number(s) and matrix of samples transported to the laboratory,
- Filtering and preservation requirements,
- Date/time when samples will arrive at the laboratory,
- Irregularities or anticipated problems associated with the samples, and
- Whether additional samples will be transported.

2.3.3 Sample Custody

The WQ field personnel are responsible for custody of the samples until they are delivered to the laboratory or picked up for transport. (Note: As few people as possible will handle the samples to ensure sample custody.) Chain-of-custody forms must be completed in the field. Each time one person relinquishes control of the samples to another person, both individuals must completed the appropriate portions of the chain-of-custody form by filling in their signature as well as the appropriate date and time of the custody transfer.

Once at the laboratory, the sample receipt coordinator will open the coolers and sign and date the chain-of-custody form. The laboratory personnel are then responsible for the care and custody of samples. The PLPT WQ Laboratory will track sample custody through their facility using separate tracking forms. In some cases the sample receipt coordinator will be the field personnel. This will be noted on the chain-of-custody forms and in field notebooks.

A sample is considered to be in one's custody if:

- The sample is in the sampler's physical possession,

- The sample has been in the sampler's physical possession and is within sight of the samplers,
- The sample is in a designated, secure area, and/or
- The sample has been in the sampler's physical possession and is locked up.

2.3.4 Surface Water Sample Filtration

Samples collected in the field and to be analyzed for ortho-phosphate, nitrate + nitrite, and ammonia are to be filtered. Filtering of these samples will take place immediately after the samples arrive at the laboratory. Filtering duties are the responsibility of the laboratory personnel after the samples are received at the laboratory. In some instances the receiving laboratory personnel will be the field personnel who collected the sample. This will be noted in laboratory notebooks and laboratory tracking forms.

Filtration equipment includes vacuum pump, vacuum flasks, and 0.45 micron filters. Once the filtration equipment is assembled the filters are pre-washed with about 150 ml of deionized water prior to contact with the sample water. Once the samples are filtered they are ready for chemical preservation if needed.

2.3.5 Sample Disposal

Following sample analysis, the laboratory will store the unused portions for 6 months. At that time, the laboratory will properly dispose of all the samples. Sample disposal procedures at the PLPT WQ Laboratory are described in the Laboratory QA Manual.

2.4 Analytical Methods

The field measurement and off-site laboratory analytical methods are listed in Table 6 and discussed below.

2.4.1 Field Measurements

See section 2.2.3.

2.4.2 Laboratory Analysis

All samples will be analyzed at the PLPT WQ Laboratory. Analyses will be performed following either EPA-approved methods or methods from *Standard Methods for the Examination of Water and Wastewater, 20th Edition,* as summarized in Table 6. The Laboratory personnel must notify the Project Manager and/or Laboratory Manager if there is any knowledge of the SOPs not being followed, see QA Manual- Appendix C. Laboratory SOPs for all water quality analyses can be found in Appendix E. Zooplankton samples will also be analyzed at the PLPT WQ Laboratory, the analysis method can also be found in Appendix E.

The Project manager and/or the Laboratory Manager will summarize the data and associated QC results in a data report, and provide this report to the QA Officer within 4 weeks of sample receipt. The content of the data report is described in Section 1.9.3. The QA Officer will review the data reports and

associated QC results to make decisions on data quality and usability in addressing the project objectives.

2.5 Quality Control Requirements

This section identifies the QC checks that are in place for the sample collection, field measurement, and laboratory analysis activities that will be used to access the quality of the data generated from this project.

2.5.1 Quality Control Requirements

Field sampling QC consists of collecting field QC samples to help evaluate conditions resulting from field activities. Field QC is intended to support a number of data quality goals:

- Combined contamination from field sampling through sample receipt at the laboratory (to assess potential contamination from field sampling equipment, ambient conditions, sample containers, sample transport, and laboratory analysis) - assessed using field blanks;
- Sample shipment/transport temperature (to ensure sample integrity and representativeness that the sample arriving at the laboratory has not degraded during transport) - assessed using temperature blanks; and
- Combined sampling and analysis technique variability, as well as sample heterogeneity - assessed using field duplicates.

For the current project, the types and frequencies of field QC samples to be collected for each field measurement and onsite laboratory analysis are listed in Table 5. These include field blanks, temperature blanks, and field duplicates.

Field Blanks - Field blanks will be collected to evaluate whether contaminants have been introduced into the samples during the sample collection due to exposure from ambient conditions or from the sample containers themselves. Field blank samples will be obtained by pouring deionized water into a sample container at the sampling location. Field blanks will not be collected if equipment blanks have been collected during the sampling event. If no equipment blanks are collected (and none are planned because samples will be collected directly into sample containers), one field blank will be collected for every 10 samples or a frequency of 10%.

Field blanks will be preserved, packaged, and sealed in the same manner described for the surface water samples. A separate sample number and station number will be assigned to each blank. Field blanks will be submitted blind to the laboratory for analysis of nutrients.

If target analytes are found in field blanks, sampling and handling procedures will be reevaluated and corrective actions taken. These may consist of, but are not limited to, obtaining sampling containers from new sources, training of personnel, discussions with the laboratory, invalidation of results, greater attention to detail during the next sampling event, or other procedures felt appropriate.

Temperature Blanks - For each cooler of samples that is transported to the analytical laboratory, a 40-ml VOA vial (prepared by the laboratory) will be included that is marked “temperature blank.” This blank will be used by the laboratory’s sample custodian to check the temperature of samples upon receipt to ensure that samples were maintained at the temperature appropriate for the particular analysis.

For the current project, temperature blanks will be included in all coolers containing samples requiring temperature preservation, as identified in Table 6.

Field Duplicate Samples - Field duplicate samples will be collected to evaluate the precision of sample collection through analysis. Field duplicates will be collected at designated sample locations by alternately filling two distinct sample containers for each analysis.

Field duplicate samples will be preserved, packaged, and sealed in the same manner described for the surface water samples. A separate sample number and station number will be assigned to each duplicate. The samples will be submitted as "blind" (i.e., not identified as field duplicates) samples to the laboratory for analysis.

For the current project, field duplicates will be collected for each analytical parameter, and field measurement parameter, at the frequencies shown in Table 5. The duplicate samples will be collected at random locations for each sampling event. Criteria for field duplicates for the analytical and field measurement parameters are provided in Table 5. If criteria are exceeded, field sampling and handling procedures will be evaluated, and problems corrected through greater attention to detail, additional training, revised sampling techniques, or other procedures that are deemed appropriate.

2.5.2 Field Measurement Quality Control

Quality control requirements for field measurements are provided in Tables 3 - 4.

2.5.3 Laboratory Analyses Quality Control

Laboratory QC is the responsibility of the personnel and QA/QC department of the PLPT WQ Laboratory. The laboratory Quality Assurance Manual details the QA/QC procedures. The following elements are part of the standard laboratory quality control practices:

- Analysis of method blanks,
- Analysis of laboratory control samples
- Instrument calibration (including initial calibration, calibration blanks, and calibration verification),
- Analysis of matrix spikes, and
- Analysis of duplicates.

The data quality objectives for the PLPT WQ Laboratory (including frequency, QC acceptance limits, and corrective actions if the acceptance limits are exceeded) are detailed in its QA Manual (Appendix C) and DQI Tables (Appendix D) in this QA Project Plan (Table 5). Any excursions from these objectives must be documented by the laboratory and reported to the Project Manager.

The PLPT WQ Laboratory's control limits and corrective action procedures have been reviewed, and these will satisfactorily meet the project data quality needs. A summary of this information is included in Table 5 and Appendix D. These include laboratory (or method) blanks, laboratory control samples, matrix spikes, and laboratory duplicates.

Method Blanks - A method blank is an analyte-free matrix, analyzed as a normal sample by the laboratory using normal sample preparation and analytical procedures. A method blank is used for monitoring and documenting background contamination in the analytical environment. Method blanks will be analyzed at a frequency of one per sample batch (or group of up to 20 samples analyzed in sequence using the same method).

Corrective actions associated with exceeding acceptable method blank concentrations (Appendix D) include isolating the source of contamination and re-digesting and/or re-analyzing the associated samples. Sample results will not be corrected for blank contamination, as this is not required by the specific analytical methods. Corrective actions will be documented in the laboratory report's narrative statement.

Laboratory Control Samples - Laboratory control samples (LCS) are laboratory-generated samples used to monitor the day-to-day performance (accuracy) of routine analytical methods. An LCS is an aliquot of clean water spiked with the analytes of known concentrations corresponding to the analytical method. LCS is used to verify that the laboratory can perform the analysis on a clean matrix within QC acceptance limits. Results are expressed as percent recovery of the known amount of the spiked analytical parameter.

One LCS is analyzed per sample batch. Acceptance criteria (control limits) for the LCS are defined by the laboratory and summarized in Table 5 and Appendix D. In general, the LCS acceptance criteria recovery range is 80 to 120 percent of the known amount of the spiked analytical parameter. Corrective action, consisting of a rerunning of all samples in the affected batch, will be performed if LCS recoveries fall outside of control limits. Such problems will be documented in the laboratory report's narrative statement.

Matrix Spikes - Matrix spikes (MS) are prepared by adding a known amount of the analyte of interest to a sample. MS are used as a similar function as the LCS, except that the sample matrix is a real-time sample rather than a clean matrix. Results are expressed as percent recovery of the known amount of the spiked analytical parameter. Matrix spikes are used to verify that the laboratory can determine if the matrix is causing either a positive or negative influence on sample results.

One matrix spike is analyzed per sample batch. Acceptance criteria are the MS are defined by the laboratory and summarized in Table 5 and Appendix D. In general, the MS acceptance criteria recovery range is of 75 to 125 percent of the known amount of the spiked analytical parameter. Generally, no corrective action is taken for matrix spike results exceeding the control limits, as long as the LCS recoveries are acceptable. However, the matrix effect will be noted in laboratory report's narrative statement and documented in the tribe's reports for each sampling event.

Laboratory Duplicates - A laboratory duplicate is a laboratory-generated split sample used to document the precision of the analytical method. Results are expressed as relative percent difference between the laboratory duplicate pair.

One laboratory duplicate will be run for each laboratory batch or every 20 samples, whichever is more frequent. Acceptance criteria (control limits) for laboratory duplicates are specified in the laboratory QA Manual and SOPs and are summarized in Table 5 and Appendix D. If laboratory duplicates exceed criteria, the corrective action will be to repeat the analyses. If results remain unacceptable, the batch

will be rerun. The discrepancy will be noted in the laboratory report's narrative statement and documented in the reports for each sampling event.

2.5.4 Background Samples

Background samples are collected because there is a possibility that there are native or ambient levels of one or more target analytes present, and because one objective of the sampling event is to differentiate between on-site and off-site contributions to a parameter's concentration. The background location for this monitoring program will be the most upstream (and thus assumed to be least impacted) sample collected. The analyses to be conducted on the background samples will be the same as that for the other surface water samples.

2.6 Instrument/Equipment Testing, Inspection, and Maintenance

2.6.1 Field Measurement Instrument/Equipment

Sampling equipment under the care of the PLPT WQ Monitoring Program will be maintained according to the manufacturer's instructions. Maintenance logs of sampling equipment under the care of the Environmental Department will be kept in the office of the Project Manger. Maintenance logs of sampling equipment under the care of the PLF Resource Department will be kept in the administration office of the PLF. Each piece of equipment will have its own maintenance log. The log will document any maintenance and service of the equipment. A log entry will include the following information:

- Name of person maintaining the instrument/equipment,
- Date and description of the maintenance procedure,
- Date and description of any instrument/equipment problem(s),
- Date and description of action to correct problem(s),
- List of follow-up activities after maintenance (i.e., system checks) and
- Date the next maintenance will be needed.

2.6.2 Laboratory Analysis Instruments/Equipment

Inspection and maintenance of laboratory equipment is the responsibility of the PLPT WQ Laboratory and is described in the QA Manual included as Appendix C.

2.7 Instrument/Equipment Calibration and Frequency

2.7.1 Field Measurement Instrument/Equipment

Calibration and maintenance of field equipment/instruments will be performed according to the associated SOP (see Appendix A) and recorded in an instrument/equipment logbook. Each piece of equipment/instrument will have its own logbook.

The project-specific criteria for calibration (frequency, acceptance criteria, and corrective actions associated with exceeding the acceptance criteria) are provided in Tables 12 - 13.

2.7.2 Laboratory Analysis Instruments/Equipment

Laboratory instruments will be calibrated according to the appropriate analytical methods. Calibration acceptance criteria are found in the PLPT WQ QA Manual included as Appendix C.

2.8 Inspection and Acceptance of Supplies and Consumables

2.8.1 Field Sampling Supplies and Consumables

Sample containers and preservatives will be provided by the analytical laboratory. Containers will be inspected for breakage and proper sealing of caps. Other equipment such as sample coolers and safety equipment will be acquired by the Tribe. If reusable sampling equipment is acquired in the future, materials/supplies necessary for equipment decontamination will be purchased by the tribe; however, this is not necessary for the present study. Any equipment deemed to be in unacceptable condition will be replaced.

2.8.2 Field Measurement Supplies and Consumables

Field measurement supplies, such as calibration solutions, will be acquired from standard sources, such as the instrument manufacturer or reputable suppliers. Chemical supplies will be American Chemical Society reagent grade or higher. The lot number and expiration date on standards and reagents will be checked prior to use. Expired solutions will be discarded and replaced. The source, lot number, and expiration dates of all standards and reagents will be recorded in the field log books.

2.8.3 Laboratory Analyses Supplies and Consumables

The laboratory's requirements for supplies and consumables are described in its QA Manual which is provided in Appendix C.

2.9 Data Acquisition Requirements (Non-Direct Measurements)

To supplement field measurements and laboratory analytical activities conducted under this project, other potential "external" data sources will be researched. These sources include, but are not limited to, the U.S. Geological Survey, the Desert Research Institute, the Washoe County Department of Water Resources, the Cities of Reno/Sparks, the U.S. Environmental Protection Agency, and the Bureau of Reclamation. The primary use of this external data will be to help focus the Tribe's data collection efforts (for example, the information may be used to identify new sites in the Truckee River/Winnemucca Lake watersheds for future sampling).

If it appears that the "external" data might facilitate water body evaluation, the data will first be reviewed to verify that they are of sufficient quality to meet the needs of the project by examining: (1) the sample collection and location information; (2) the data to see whether they are consistent with known tribally-collected data from the same general vicinity; and (3) the QA/QC information associated with the data. If the data are of insufficient or unknown quality, limitations will be placed on its use in supporting project decisions. In general, it is anticipated that decisions for the current project will be based on data collected by the tribe following this current QA Project Plan.

2.10 Data Management

All data recorded on physical forms/notebooks and data reports from the laboratory will be stored in the PLPT Environmental Department. All electronic versions of data will be stored on the Environmental Department Server which is accessible by all Environmental Department staff.

After field and laboratory activities are reviewed for the sampling event, the water quality data gathered will be entered into spreadsheets. Spreadsheets have been created for each water body type and are centrally stored on the Environmental Department server.

The Environmental Database Specialist uses the water quality data spreadsheets to enter the data into the WQX Template for submission to the data warehouse. The WQX spreadsheets are stored on the Environmental Department server and data is submitted quarterly to WQX. The following is the list of water quality characteristics that are submitted to WQX:

- Orthophosphate (mg/l)
- Phosphate-phosphorus (Total Phosphorus) (mg/l)
- Ammonia (mg/l)
- Inorganic nitrogen (nitrate and nitrite as N) (mg/l)
- Temperature, water (°C)
- Specific conductance (mS/cm)
- Total dissolved solids (g/l)
- Salinity (ppt)
- Dissolved oxygen (mg/l)
- pH
- Turbidity (NTU)

The data gathered is also used to determine whether a water body is meeting/not meeting the PLPT Water Quality Standards. This information is entered throughout the year and reported annually. The analysis methods are described in the PLPT Water Quality Control Plan. The analysis is done in a spreadsheet format and a copy is stored on the Environmental Department Server.

All monitoring sites coordinates have been collected and entered in a spreadsheet that is stored on the Environmental Department Server. The data submitted to WQX also includes site coordinates. The Environmental Database Specialist can produce maps for monitoring purposes when needed.

3.0 Assessment and Oversight

This section describes how to check that all activities are completed correctly and according to procedures outlined in this QA Project Plan.

3.1 Assessment/Oversight and Response Actions

During the course of the project, it is important to assess the project's activities to ensure that the QA Project Plan is being implemented as planned. This helps to ensure that everything is on track and serves to minimize learning about critical deviations toward the end of the project when it may be too late to remedy the situation. For the current project, the ongoing assessments will include:

Field Oversight -

- Readiness review of the field team prior to starting field efforts,
- Field activity audits, and
- Review of field sampling and measurement activities methodologies and documentation at the end of each of event, and

Laboratory Oversight -

- Evaluation of laboratory data generated for each sampling event.

Details regarding these assessments are included below.

3.1.1 Field Oversight

3.1.1.1 Readiness Reviews

Sampling personnel will be properly trained by qualified personnel before any sampling begins and will be given a brief review of sampling procedures and equipment operation by the PLPT Project Manager before each sampling event. Equipment maintenance records will be checked to ensure all field instruments are in proper working order. Adequate supplies of all preservatives and bottles will be obtained and stored appropriately before heading to the field. Sampling devices will be checked to ensure that they have been properly cleaned (for devices which might be reused) or are available in sufficient quantity (for devices which are disposable). Proper paperwork, logbooks, chain of custody forms, etc. will be assembled by the sampling personnel. The Project Manger will review all field equipment, instruments, containers, and paperwork to ensure that all is in readiness prior to the first day of each sampling event. Any problems that are noted will be corrected before the sampling team is permitted to depart the Tribe's facilities.

3.1.1.2 Field Activity Audits

During at least two of the sampling events, the PLPT Project Manager will assess the sample collection methodologies, field measurement procedures, and record keeping of the field team to ensure activities are being conducted as planned (and as documented in this QA Project Plan). Any deviations that are noted will be corrected immediately to ensure all subsequent samples and field measurements collected are valid. (Note: If the deviations are associated with technical changes and/or improvements made to the procedures, the QA Officer will verify that the changes have been documented by the Field Sampler in the Field Log Books and addressed in an amendment to this QA Project Plan). The QA Officer may stop any sampling activity that could potentially compromise data quality.

The PLPT QA Officer will document any noted issues or concerns in a QA Audit Logbook and discuss these items informally and openly with the Field Personnel while on site. Once back in the office, the QA Officer will formalize the audit findings (for each event) in a Field Audit report, which will be submitted to the PLPT Environmental Program Director and the Field Sampler.

The Field Sampler will prepare a Corrective Action Report to address any audit findings discussed in the Field Audit Report. The Corrective Action Report will be issued as an internal memorandum the PLPT Environmental Program Director and Project Manager/QA Officer in response to problems noted during on-site audits and will document steps taken to reduce future problems prior to the next sampling event.

3.1.1.3 Post Sampling Event Review

Following each sampling event, the Field Sampler will complete the Field Activities Review Checklist (Figure 11). This review of field sampling and field measurement documentation will help ensure that all information is complete and any deviations from planned methodologies are documented. This review will be conducted in the office, not in the field. (Note: This function is typically performed by a third party not directly involved in the activities. However, due to the small size of the staff, the field technician will attempt to "wear a new hat" and self-evaluate his/her activities). The results of this review, as well as comments associated with potential impacts on field samples and field measurement integrity will be forwarded to the Project Manager to be used in preparing the reports for each event and also to be used as a guide to identify areas requiring improvement prior to the next sampling event.

3.1.2 Laboratory Oversight

Following receipt of the laboratory's data package for each sampling event, the QA Officer will review the data package for completeness, as well as to ensure that all planned methodologies were followed and that QA/QC objectives were met. The results of the review will be documented on the Laboratory Data Review Checklist (Figure 12). (Note: The Project Manager/QA Officer has the authority to request re-testing or other corrective measures if the laboratory has not met the project's QA/QC objectives and/or has not provided a complete data package.)

Due to the scope and objectives of the current project, the Tribe is not planning any laboratory audits at this time. The laboratory's QA Manual (Appendix C) describes the policies and procedures for assessment and response in the laboratory.

3.2 Reports to Management

Once each quarter, the Project Manager will prepare and submit a report on that quarter's monitoring and data management activities. Contents of this report have been described previously in Section 1.9.5. This report will be submitted to the PLPT - Environmental Director for approval. After approval, the report will be submitted to the US EPA Grants Project Officer.

Once a year a report summarizing the year's reports will be prepared which will show any data trends that have occurred. The report will also discuss how any actions taken during the year may have affected the trends. This report will also be submitted to the PLPT – Environmental Director for approval. After approval, the report will be submitted to the US EPA Grants Project Officer.

Additional (less formal) internal reports are described in Sections 1.9.2 through 1.9.4.

3.3 Programmatic Evaluation

An annual review and update of the monitoring strategy has been identified as an important component of implementing the strategy. An annual review and update of the monitoring strategy between the PLPT Environmental Department staff and regional EPA staff will be conducted. This annual review will address programmatic coordination and evaluate the effectiveness of the monitoring and assessment program. Resource limitations, new and emerging issues and changing program objectives will be evaluated and any data gaps or needs will be addressed.

4.0 Data Review and Usability

Prior to utilizing data to make project decisions, the quality of the data needs to be reviewed and evaluated to determine whether the data satisfy the project's objectives. This process involves technical evaluation of the laboratory data, as well as review of the data in conjunction with the information collected during the field sampling and field measurement activities. This later, more qualitative review provides for a clearer understanding of the overall usability of the project's data and potential limitations on their use. This section describes the criteria and procedures for conducting these reviews and interpreting the project's data.

4.1 Data Review, Verification, and Validation Requirements

Setting data review, verification, and validation requirements ensures that project data are evaluated in an objective and consistent manner. For the current project, such requirements have been defined for information gathered and documented as part of field sampling and field measurement activities, as well as for data generated by the PLPT WQ Laboratory.

4.1.1 Field Sampling and Measurement Data

Any information collected during sample collection and field measurements is considered "field data." This includes field sampling and measurement information documented in field logbooks (as listed in Section 1.9.2.1), photographs, and chain of custody forms.

Once the Field Sampler returns to the office following a field event, he/she is responsible for conducting a technical review of the field data to ensure that all information is complete and any deviations from the planned methodologies are documented. (Note: This function is typically performed by a third party not directly involved in the activities. However, due to the small size of the staff, the field technician will attempt to self-evaluate his activities). For the purpose of this project, the review will be documented using the Field Activities Review Checklist provided in Figure 11. This checklist comprehensively covers the items to be reviewed and leaves room to capture any comments associated with potential impacts on field samples and field measurement integrity based on the items listed.

4.1.2 Laboratory Data

The PLPT WQ Laboratory is responsible for its own internal data review and verification prior to submitting the associated data results package to the Project Manager/QA Officer. The details of the review (including checking calculations, reviewing for transcription errors, ensuring the data package is complete, etc.) are discussed in the laboratory's QA Manual included as Appendix C. Details of the information that will be included in each data package are listed in Section 1.9.3 of this QA Project Plan.

Once the laboratory data is received by the Project Manager/QA Officer, each data package will be further reviewed for validation. For the purpose of this project, data review and validation will be conducted using the Data Review Checklist provided in Figure 12 in conjunction with the QC criteria (i.e., frequency, acceptance limits, and corrective actions) defined in Tables 5 and Appendix D. This review will include evaluation of the field and laboratory duplicate results, field and laboratory blank data, matrix spike recovery data, and laboratory control sample data pertinent to each analysis. The review will also include ensuring data are reported in compliance with the project action limits and quantitation limits defined in Tables 1 - 2 ; the sample preparation/analytical procedures were performed by the

methods listed in Table 6; sample container, preservation, and holding times met the requirements listed in Table 6; the integrity of the sample (ensuring proper chain of custody and correct sample storage temperatures) is documented from sample collection through shipment and ultimate analysis, and the data packages. The Data Review Checklist comprehensively covers the review of all these items. (Note: Calibration data will not be requested for the project at this time.)

The Project Manager/QA Officer will further evaluate each data package's narrative report and summary tables to see whether the laboratory "flagged" any sample results based on poor or questionable data quality and to ensure that any exceedances of the laboratory's QC criteria (as listed in Tables 5 and Appendix D) are documented. If a problem was noted by the laboratory, the Project Manager/QA Officer will evaluate whether the appropriate prescribed corrective action was taken by the laboratory, the action successfully resolved the problem, and the process and its resolution were accurately documented.

An effort will be made to identify whether any data quality problem is the result of laboratory issues and/or if it may be traced to some field sampling activity. If the laboratory is determined to be responsible, the Project Manager/QA Officer will request information from the laboratory documenting that the problem has been resolved prior to submitting future samples. If some aspect of the field operation (e.g., sample collection, sample containers and/or preservation, chain-of-custody, sample shipment, paperwork, etc.) is identified as the possible problem, efforts will be made to retrain the Tribe's field staff to minimize the potential of the problem recurring. If the problem is believed to be due to the sample matrix, the Project Manager/QA Officer will discuss the use of alternative analytical methods with the laboratory; and, if an alternative method is available that might minimize the problem, the QA Project Plan will be modified and/or amended accordingly.

If any of the QC criteria and/or the project requirements (as discussed above) is exceeded, the associated data will be qualified as estimated and flagged with a "J". If grossly exceeded, the associated data will be rejected and the need for re-sampling will be considered. However, since there are no plans to use the data for enforcement or other legal applications, it is generally felt that paying special attention to some troublesome sample collection or analytical concern during the next sampling event will be sufficient and re-sampling will not be necessary.

4.2 Verification and Validation Methods

Defining the data verification and validation methods help to ensure that project data are evaluated in an objective and consistent manner. For the current project, such methods have been described for information gathered and documented as part of the field sampling and field measurement activities, as well as the data generated by the PLPT WQ laboratory.

4.2.1 Field Sampling and Measurement Data

The methods associated with verification and validation of the field sampling and measurement data are included within the discussion provided in Section 4.1.1.

4.2.2 Laboratory Data

The methods associated with verification and validation of the laboratory data are included within the discussion provided in Section 4.1.2.

4.3 Reconciliation with User Requirements

The purpose of the continued monitoring of surface waters within the PLIR is to protect the biological, chemical, and physical integrity of the PLPT's water resources. Data must fulfill the requirements of this QA Project Plan to be useful for the overall project. Information needed to support decision making under the surface water monitoring program is contained in this QA Project Plan, field documentation, the laboratory "data package" report, the Field Activities Review Checklist, the Laboratory Data Review Checklist, and the Field Audit Report and associated Corrective Action Report. This section describes the steps to be taken to ensure data usability (after all the data have been assembled, reviewed, verified, and validated) prior to summarizing the information in the Quarterly and Annual Reports.

Once all the data from the field and laboratory have been evaluated (as described in Sections 4.1 and 4.2), the Project Manager/QA Officer will make an overall assessment concerning the final usability of the data (and any limitations on its use) in meeting the project's needs. The initial steps of this assessment will include, but not necessarily be limited to:

- Discussions with the PLPT's Field Samplers,
- Review of deviations from the QA Project Plan or associated SOPs to determine whether these deviations may have impacted data quality (and determining whether any impacts are widespread or single incidents, related to a few random samples or a batch of samples, and/or affecting a single or multiple analyses),
- Evaluation of the field and laboratory results and QC information,
- Review of any other external information which might influence the results, such as off-reservation activities up stream, meteorological conditions (such as storm events preceding sampling that might contribute to high turbidity readings), and data from other sources,
- Evaluation of whether the completeness goals defined in this QA Project Plan have been met,
- Examination of any assumptions made when the study was planned, if those assumptions were met, and, if not, how the project's conclusions are affected.

After all this information has been reviewed, the Project Manager/QA Officer will incorporate his/her perspective on the critical nature of any problems noted and, ultimately, identify data usability and/or limitations in supporting project objectives and decision making. All usable data will then be compared to the Project Action Limits (as listed in Table 1 and Table 2) to identify whether these limits have been exceeded. Any result over these limits for two consecutive sampling events will be referred to the Tribal Council for consideration of possible action.

In addition, the PLPT Project Manager/QA Officer will assess the effectiveness of the monitoring program and data collection at the end of each calendar year. Sampling locations, frequency, list of analytical parameters, field measurement protocols, choice of the analytical laboratory, etc. will be modified as needed to reflect the changing needs and project objectives of the Pyramid Lake Paiute Tribe. This QA Project Plan will be revised and/or amended accordingly.

5.0 References

Pyramid Lake Paiute Tribe, 2008. *Pyramid Lake Paiute Tribe Water Quality Control Plan*, Environmental Department.

U.S. Environmental Protection Agency, 1991. *Office of Emergency and Remedial Response (OERR) Directive 9345.302*, May.

U.S. Environmental Protection Agency, 2000. *Guidance for the Data Quality Objectives Process*, EPA QA/G-4, EPA/600/R-96/005, August.

U.S. Environmental Protection Agency, 2001. *EPA Requirements for Quality Assurance Project Plans*, EPA QA/R-5, EPA/240/B-01/003, March.

U.S. Environmental Protection Agency, 2002, *Guidance for Quality Assurance Project Plans*, EPA QA/G-5, EPA/240/R-02/009, December.

U.S. Environmental Protection Agency, 2002. *Guidance on Choosing a Sampling Design for Environmental Data Collection for Use in Developing a Quality Assurance Project Plan*, QA/G-5sS, EPA/240/R-02/005, December.

U.S. Environmental Protection Agency, 2002. *Guidance on Environmental Data Verification and Data Validation*, EPA QA/G-8, EPA/240/R-02/004, November.

Figures

Figure 1: Project Organizational Charts

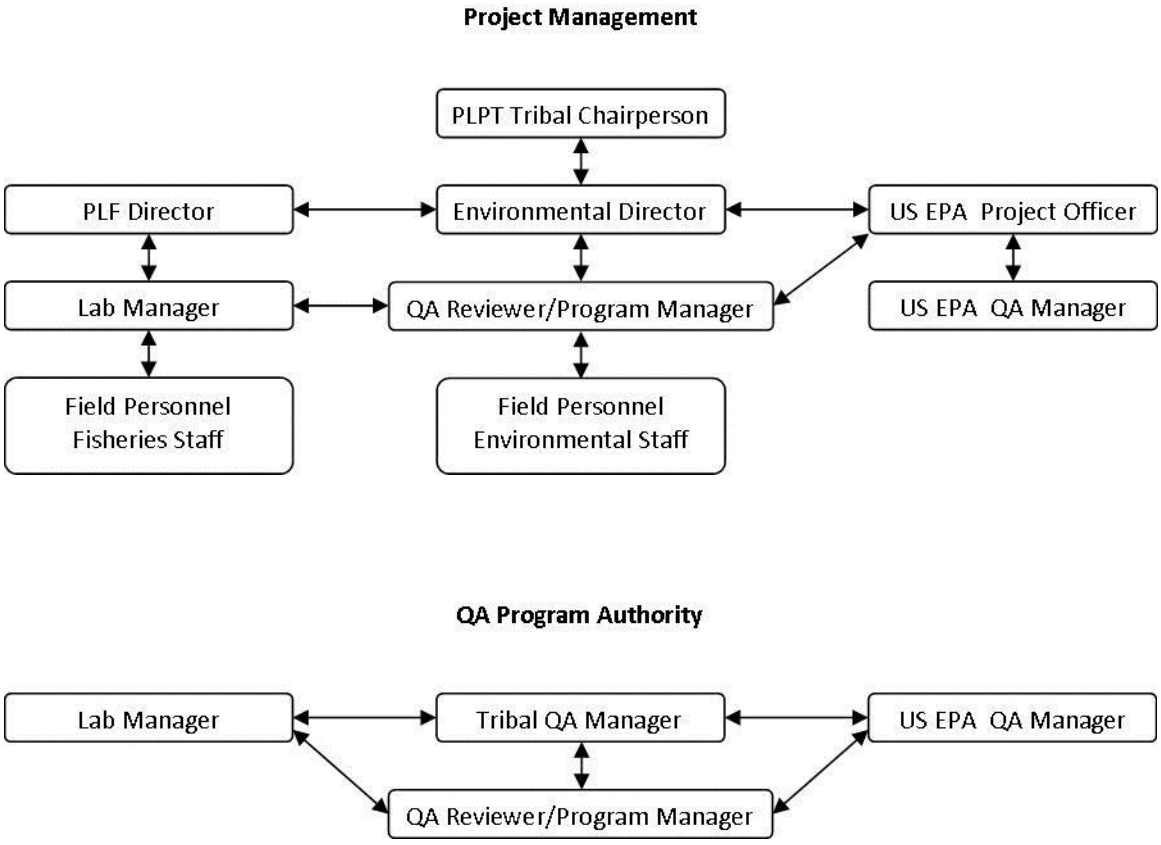


Figure 2: Lake Tahoe/Truckee River Watershed

Figure 3: Perennial, Intermittent, and Ephemeral Surface Waters within the PLIR.
The red line designates the Reservation Boundary line.

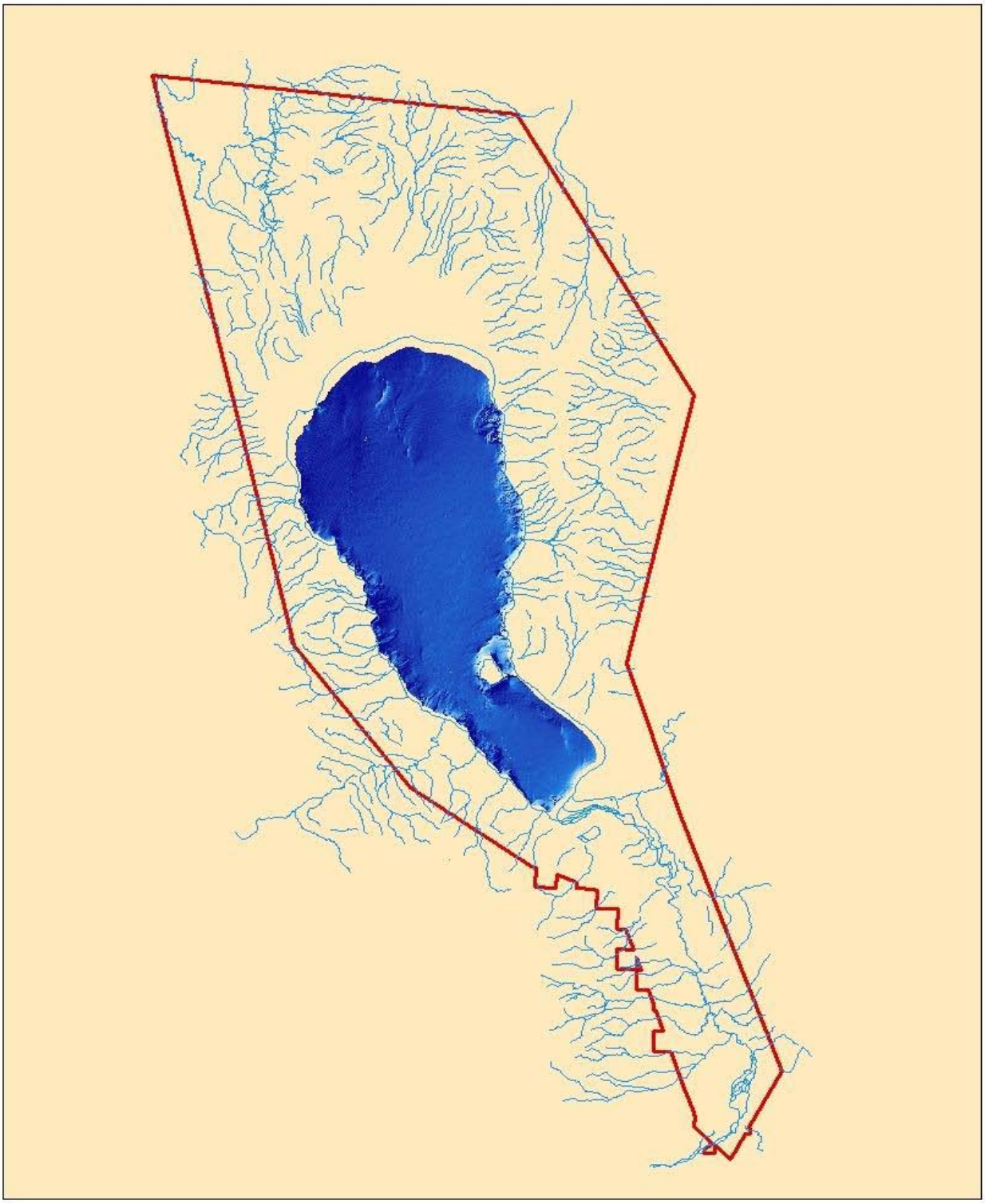


Figure 4: Lower Truckee River Water Quality Sampling Sites

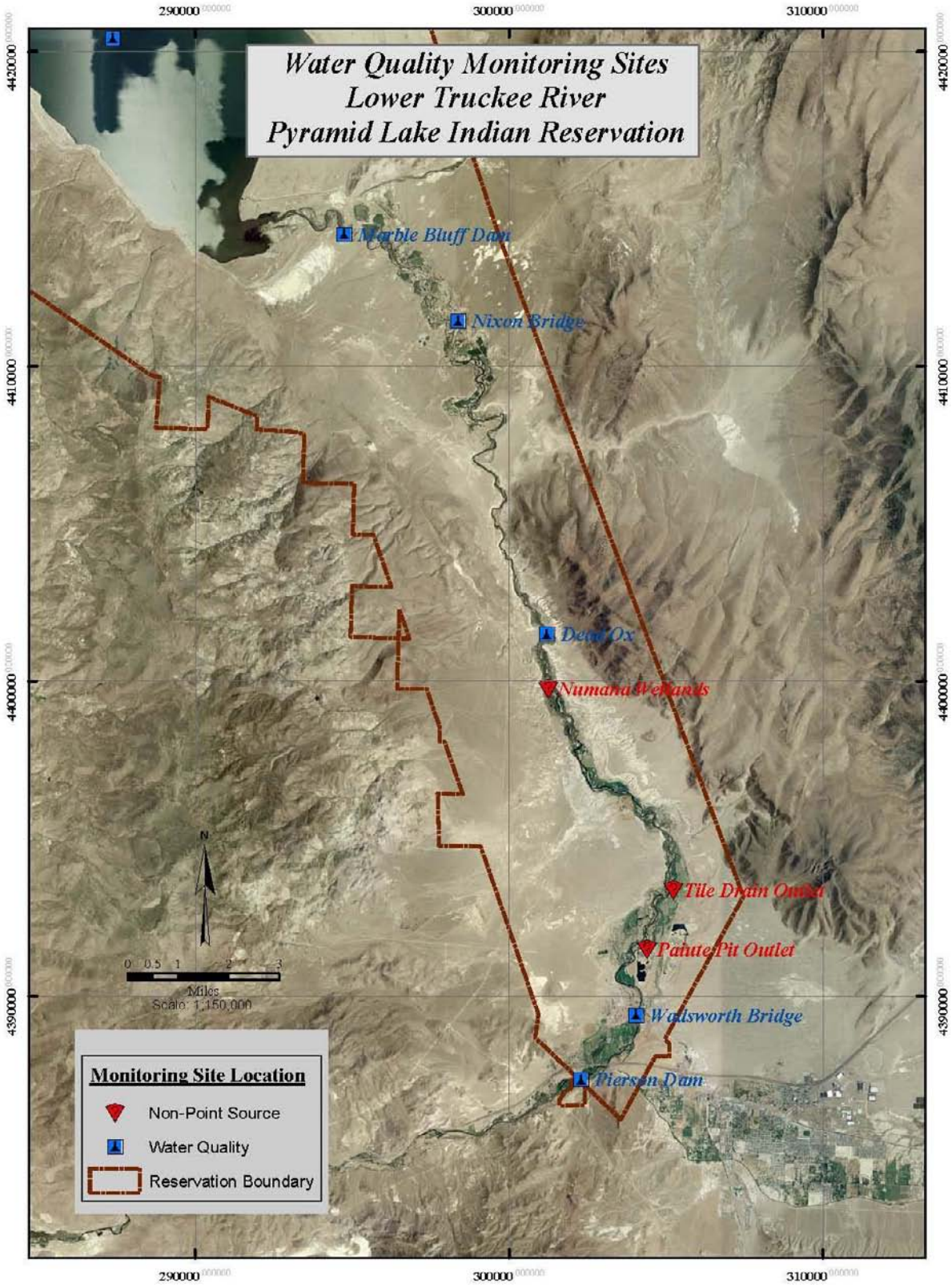


Figure 5: Truckee River Physical Habitat & Bioassessment Sampling Sites

Figure 6: Virginia Mountain Range Stream WQ Monitoring Sampling Sites

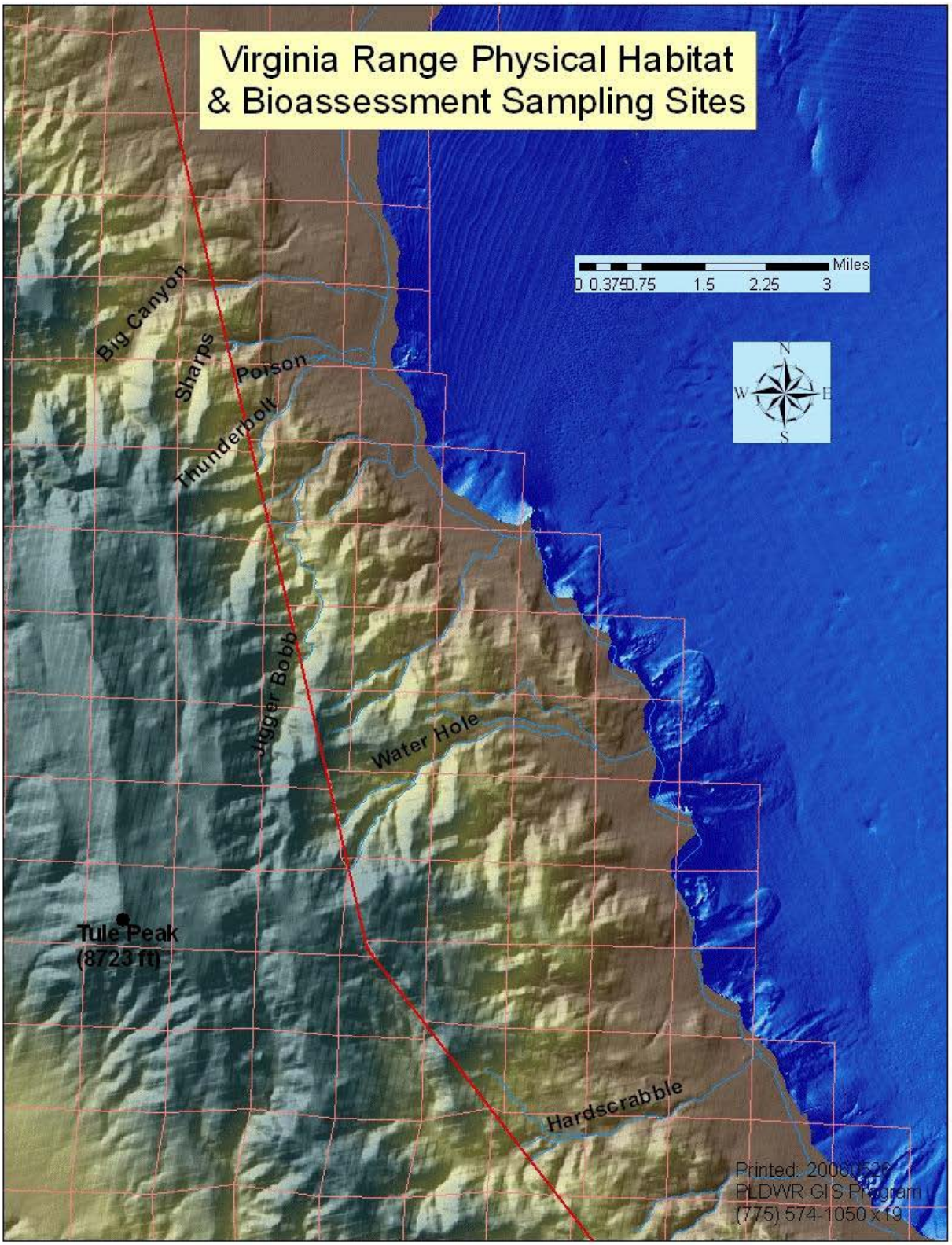


Figure 7: Pah Rah Mountain Range Stream WQ Monitoring Sampling Sites

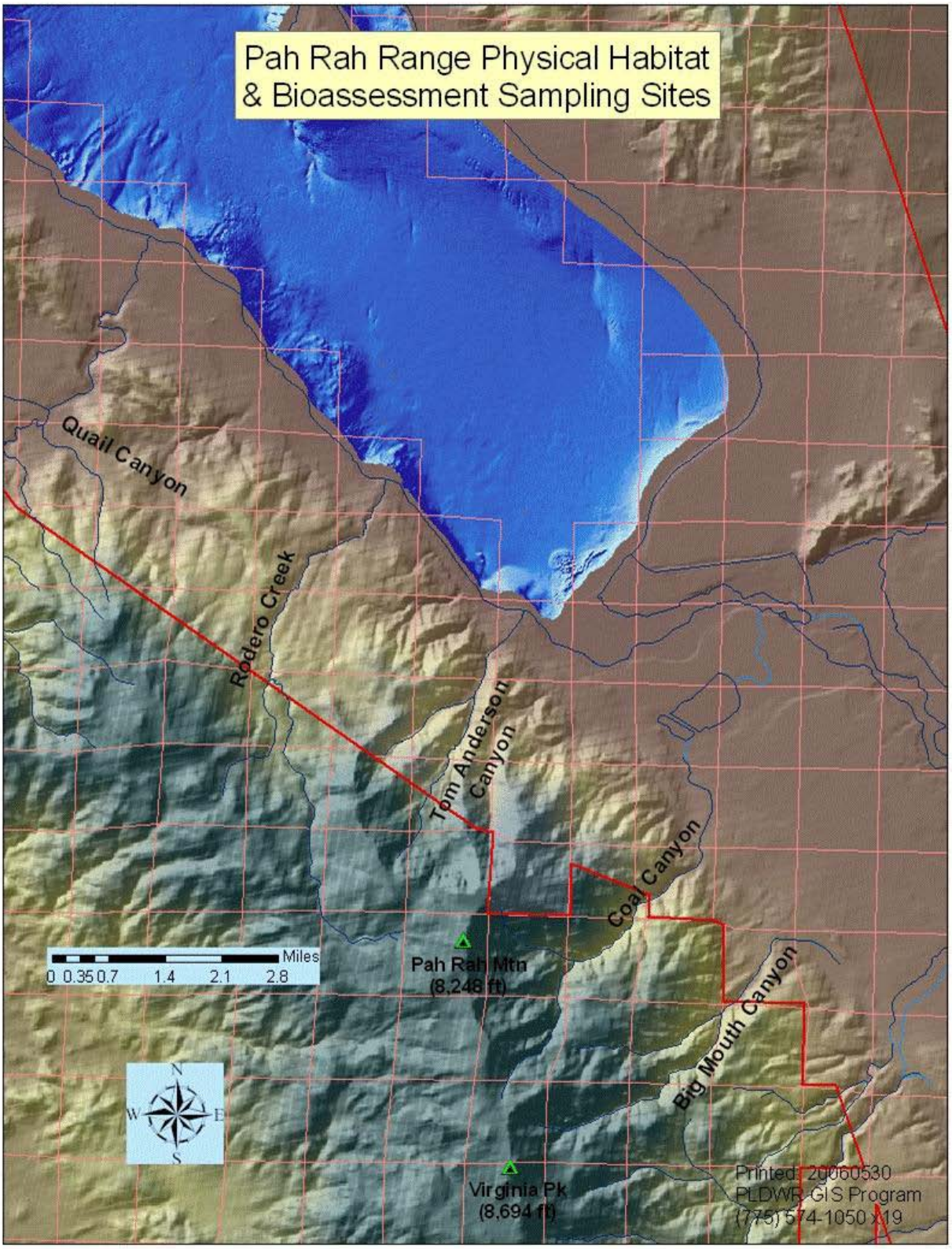


Figure 8: Lake Mountain Range Stream WQ Monitoring Sampling Sites

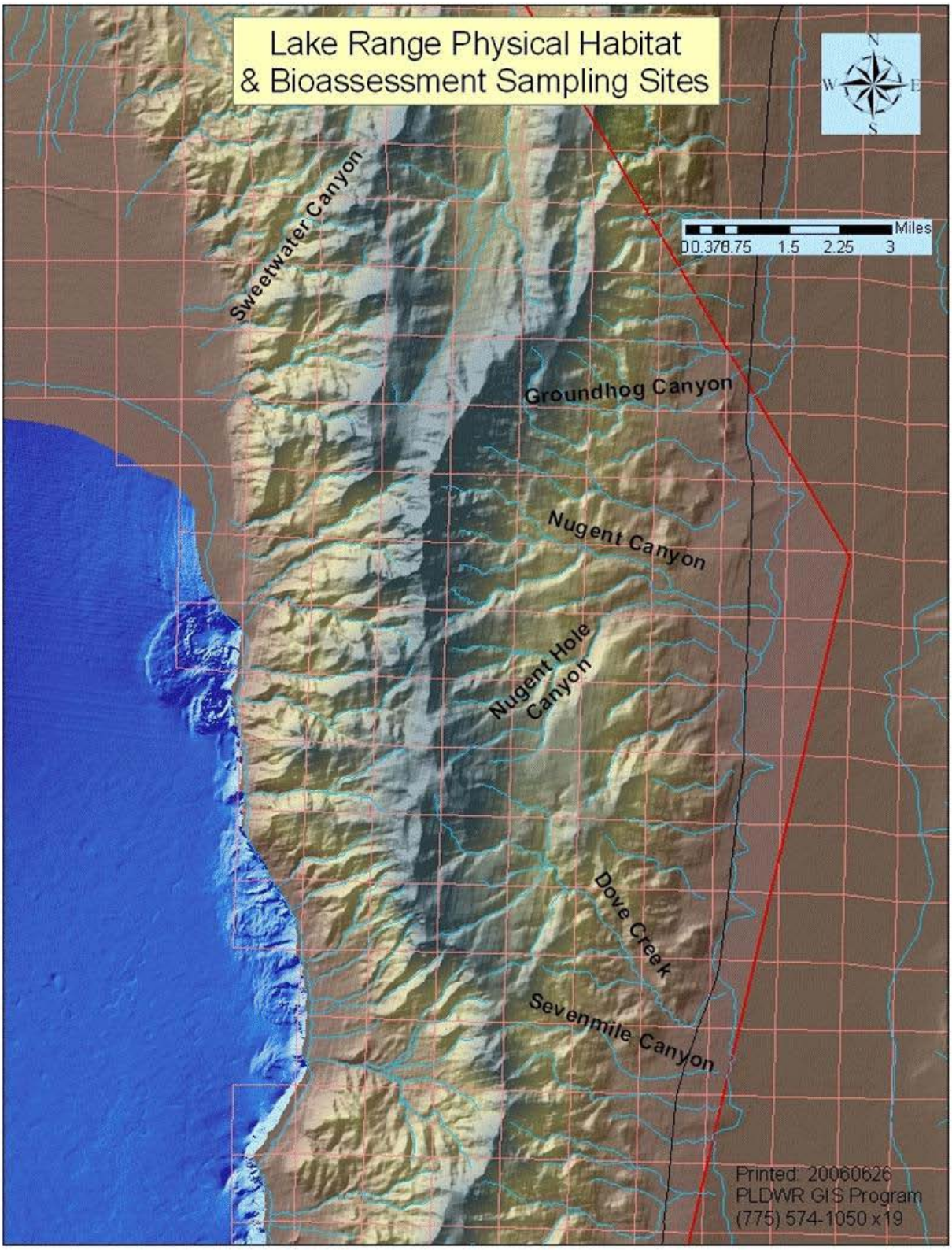


Figure 9: Pyramid Lake WQ Monitoring Sampling Sites

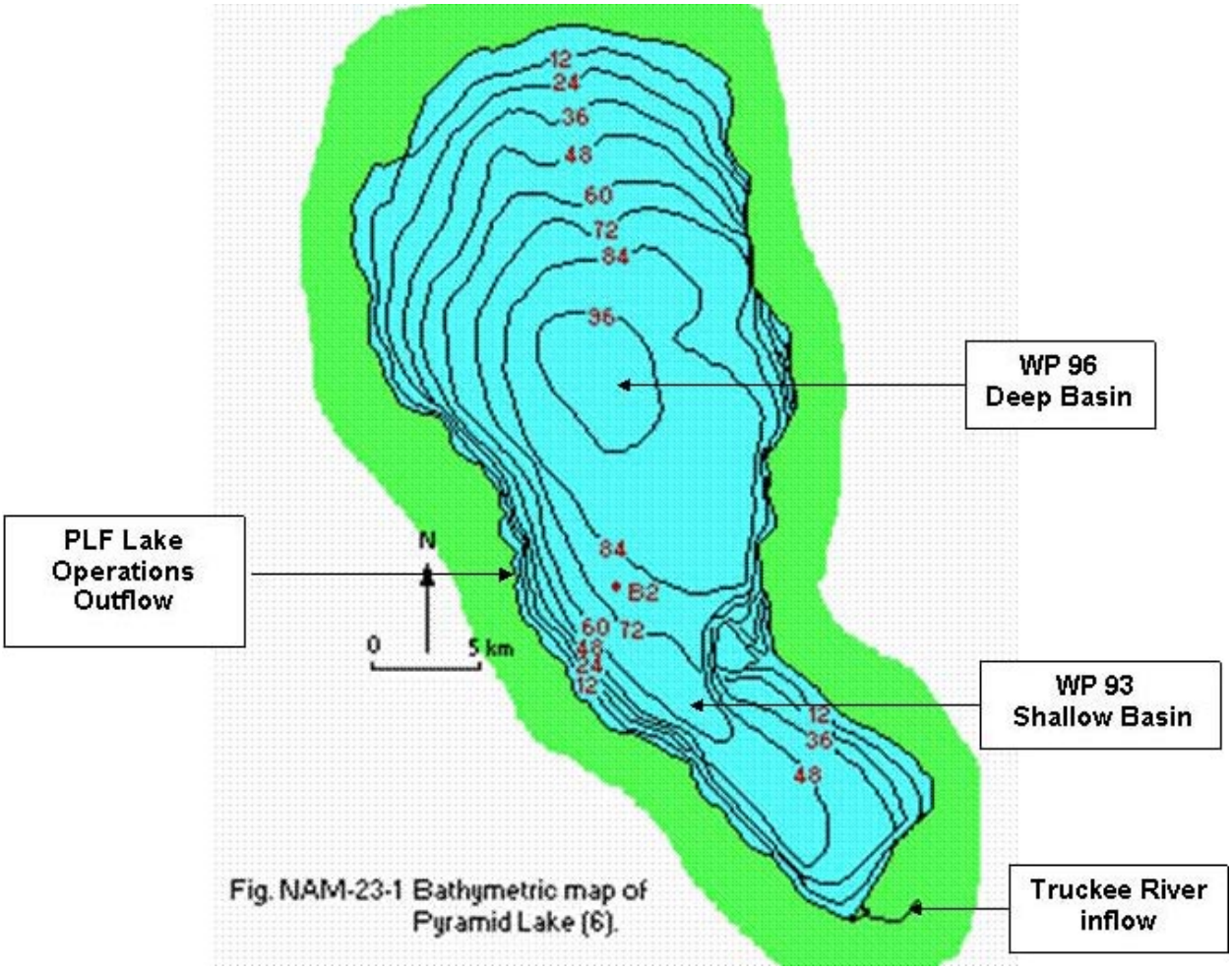


Figure 10: Sample Chain-of-Custody Form

Pyramid Lake Paiute Tribe Water Quality Laboratory
Chain of Custody and Test Request Form

Project: ☐ PL-ST 96 ☐ PL-ST 93 ☐ LTR/NPS ☐ RBA ☐ Streams ☐ NPS ☐ Wetlands ☐ Other[1]

Test(s) Requested: ☐ All ☐ Ortho-Phosphate ☐ Total Phosphate ☐ Nitrate+Nitrite ☐ Ammonia-N ☐ Total Kjeldahl Nitrogen

Date Sampled	Time Sampled	Sample ID	Sample Description	Remarks

Sampled By:

Print Name Signature Date

Additional Instructions:

Signature	Print Name	Date	Time
Relinquished by:			
Received by:			
Relinquished by:			
Received by:			
Relinquished by:			
Received by:			

[1]Sampler to provide information about water body, sample sites and any field measurements collected.

Only listed tests are offered at the PLPT WQ Laboratory at this time. Water samples are only received from departments within the PLPT organization. All sample bottles provided by the PLPT WQ Laboratory are 1 Liter Polyethylene bottles. Sample preservation is completed at the laboratory.

Figure 11: Field Activities Review Checklist

Field Activities Review Checklist

Sampling Location(s): _____

Date(s) of Sampling: _____

Mark each topic "Yes", "No", or "NA", and comment as appropriate.

____ All required information was entered into field logbooks in ink, and logbook pages were signed and dated.
Comment: _____

____ Deviations from SOPs, along with any pertinent verbal approval authorizations and dates, were documented in field logbooks.
Comment: _____

____ Samples that may be affected by deviations from SOPs were flagged appropriately.
Comment: _____

____ Field measurement calibration standards were not expired and were in the correct concentrations.
Comment: _____

____ Field calibrations were performed and results were within QAPP-specified limits for all parameters.
Comment: _____

____ Field measurement QA samples were within the QAPP-specified limits for all parameters.
Comment: _____

____ Field measurement data were recorded in the appropriate logbook(s).
Comment: _____

____ Samples were collected at the correct sites.
Comment: _____

____ The correct number of samples for each type of analysis and the correct volume was collected.
Comment: _____

____ Certified clean sample containers, appropriate for the intended analysis, were used.
Comment: _____

____ Requested/required field quality control (QC) samples were collected and at the correct frequency.
Comment: _____

____ Samples were preserved with the correct chemicals, if required.
Comment: _____

____ Samples were stored and/or shipped at the proper temperature.
Comment: _____

____ Chain-of-custody documents were completed properly.
Comment: _____

____ Custody seals were applied and intact when relinquishing custody of the samples.
Comment: _____

____ Sample holding times were not exceeded during field operations.
Comment: _____

Reviewer's Name (print): _____

Reviewer's Signature: _____

Reviewer's Title: _____

Address & Phone Number: _____

Figure 12: Laboratory Data Review Checklist

Laboratory Data Review Checklist

Sampling Project: _____

Date of Sampling: _____

Analytical Laboratory: _____

Mark each topic "Yes", "No", or "NA" and comment as appropriate:

____ Final data package includes chain-of-custody forms.
Comment: _____

____ Chain-of-custody forms were properly completed and signed by everyone involved in transporting the samples.
Comment: _____

____ Laboratory records indicate sample custody seals were intact upon receipt.
Comment: _____

____ Samples arrived at the laboratory at the proper temperature.
Comment: _____

____ All requested analyses were performed and were documented in the analytical report.
Comment: _____

____ Analyses were performed according to the methods specified in the approved QA Project Plan.
Comment: _____

____ Holding times for extraction and analysis were not exceeded.
Comment: _____

____ Method detection and/or quantitation limits were included in the report.
Comment: _____

____ A narrative summarizing the analyses and description of analysis problems was included in the final report.
Comment: _____

____ Method (laboratory) blank results were included for all analyses, at the appropriate frequency, and showed no laboratory contamination.
Comment: _____

____ Initial calibration data (if requested from the laboratory) were within QAPP method, or laboratory SOP defined acceptance criteria for all analyses.
Comment: _____

____ Continuing calibration data (if requested from the laboratory) were within QAPP, method, or laboratory SOP defined acceptance criteria for all analyses.
Comment: _____

____ Matrix spike data were included for all pertinent analyses for every 20 samples.
Comment: _____

____ Laboratory control sample data were included for all analyses for every 20 samples.
Comment: _____

____ Laboratory duplicate data were included for all analyses for every 20 samples.
Comment: _____

____ Field blanks do not contain analytes of interest or interfering compounds and included for all pertinent analyses for every 20 samples.
Comment: _____

Figure 12: Continued

____ Field duplicates are within QAPP-defined acceptance criteria and included for all analyses for every 10 samples.
Comment: _____

____ Matrix spike results were listed and within QAPP or laboratory defined acceptance criteria.
Comment: _____

____ Matrix interferences were definitively identified either through a second analysis or use of laboratory control sample results.
Comment: _____

____ Laboratory control sample results were within the QAPP or laboratory defined acceptance criteria.
Comment: _____

____ Laboratory duplicate results were within QAPP or laboratory defined acceptance criteria.
Comment: _____

____ Reported results were within method detection or quantitation limits.
Comment: _____

Reviewer's Name (print): _____

Reviewer's Signature: _____

Reviewer's Title: _____

Tables

Table 1: Pyramid Lake Analytical Parameters and Target Limits

Analytical Parameter/Field Measurement	**Project Action Limit[1]**	**Analytical Laboratory Limits (mg/L)**	
		Quantitation Limits[2]	**Detection Limits**
Laboratory Analyses			
Total Ammonia, (as nitrogen)	≤ .015 mg/L (0-20m depth)	0.015	0.010
Nitrate + Nitrite (as nitrogen)	NRL [3]	0.020	0.010
Total Kjeldahl Nitrogen (as nitrogen)	NRL	0.020	0.010
Total Nitrogen (as nitrogen)	≤ 0.900 mg/L (0-20m depth) ≤ 1.000 mg/L (full column)	N/A	N/A
Total Phosphate	≤ 0.120 mg/L (0-20m depth) ≤ 0.140 mg/L (full column)	0.020	0.010
Orthophosphate	≤ 0.095 (0-20m depth) ≤ 0.115 mg/L (full column)	0.020	0.010
Field Measurements	**Project Action Limit[1]**	**Measurement Range[6]**	**Detection Limits**
Temperature	Single value: ≤ 20°C	-5 to +35°C	N/A
pH	Single Value: ≤9.7	0.0 – 14.0 pH units	N/A
Dissolved Oxygen	Single Value: ≥80% Saturation A-Avg[5]: ≥90% Saturation (0-20m depth)	120% of surface saturation in all natural waters, fresh and salt	N/A
Specific Conductivity	NRL	0.0 to 7.0 S/m	N/A
Total Dissolved Solids	A-Avg[5].: ≤5,900 mg/L	N/A	N/A
Clarity[4]	A-Avg.: ≤0.45 m (0-20m depth)	N/A	N/A
Chlorophyll *a*	Depth Avg: ≤ 5 µg/L (0-20 m, April – October)	0 – 5 µg/L	N/A
PAR (Photosynthetic Active Radiation)	NRL	400 – 700 nm	N/A

Notes:

[1] Listed are numeric WQS for Pyramid Lake as identified in Pyramid Lake Paiute Tribe, Water Quality Control Plan: 2001.

[2] All "ANALYSES" values are in mg/l and based on information provided by PLPT Analytical Laboratory. All "FIELD MEASUREMENTS" values are in the units noted and based on information provided in the manufacturers' manuals for the equipment.

[3] NRL- No regulatory limit. Laboratory Quantitation Limit is acceptable for this project.

[4] Secchi Disk (Clarity) readings for Pyramid Lake are 'estimated' by sight.

[5] A-Average = Annual Average

[6] Values indicate the measurement ranges of field instruments and bracket the project action limits. The ranges are supported by calibration procedures.

Table 2: Truckee River Analytical Parameters and Target Limits

Analytical Parameter/Field Measurement	**Project Action Limit[1]**	**Analytical Laboratory Limits (mg/L)**	
		Quantitation Limits[2]	**Detection Limits**
Laboratory Analyses			
Total Ammonia, (as nitrogen)	≤ 2.8 mg/L (no aquatic life &0-20m depth) ≤ 2.1 mg/L (w/ aquatic life &0-20m depth)	0.015	0.010
Nitrate + Nitrite (as nitrogen)	Single Value: ≤ 2.04 mg/L	0.020	0.010
Total Kjeldahl Nitrogen (as nitrogen)	NRL	0.020	0.010
Total Nitrogen (as nitrogen)	A-Average[4]: ≤ 0.0.75 mg/L Single Value: ≤ 1.200 mg/L	N/A	N/A
Total Phosphate	NRL[3]	0.020	0.010
Orthophosphate	A-Average: ≤ 0.05 mg/L	0.020	0.010
Field Measurements	**Project Action Limit[1]**	**Measurement Range[5]**	**Detection Limits**
Temperature	Average Daily Temperature Nov-Mar: ≤ 13°C Apr-Jun: ≤ 14°C Jul-Oct: ≤ 21°C	-5.0 – 45.0°C	N/A
pH	Single Value: 6.5 – 9.0	0.0 – 14.0 pH units	N/A
Dissolved Oxygen	Nov-Jun: ≥6.0 mg/L Jul-Oct: ≥5.0 mg/L	0.0 – 50 mg/L	N/A
Specific Conductivity	NRL	0.0 - 100 mS/cm	N/A
Turbidity	Single Value: ≤ 10.0	0.0 – 1000 NTU	N/A
Total Dissolved Solids	Single Value: ≤ 310 mg/L A-Average: ≤ 245 mg/L	N/A	N/A

Notes:

[1] Listed are numeric WQS for Pyramid Lake as identified in Pyramid Lake Paiute Tribe, Water Quality Control Plan: 2001.

[2] All "ANALYSES" values are in mg/l and based on information provided by PLPT Analytical Laboratory. All "FIELD MEASUREMENTS" values are in the units noted and based on information provided in the manufacturers' manuals for the equipment.

[3] NRL- No regulatory limit. Laboratory Quantitation Limit is acceptable for this project.

[4] A-Average = Annual Average

[5] Values indicate the measurement ranges of field instruments and bracket the project action limits. The ranges are supported by calibration procedures.

Table 3: Quantitation Limits (Measurement Range) of Field Equipment

Parameter	**Method/Instrument**	**Measurement Range**
YSI 6920V2 Multi-Probe Water Quality Instrument		
Temperature	6560 Temperature/Conductivity Sensor	-5.0 to 45.0°C
pH	6561 pH Sensor	0.0 to 14.0 standard pH units
Dissolved Oxygen	6150 ROX Optical Dissolved Oxygen Sensor DO Sensor	0.0 to 50 mg/L
Turbidity	6136 Turbidity Sensor	0.0 to 1000 Nephelometric Unit (NTU)
Conductivity/ Specific Conductance	6560 Temperature/ Conductivity Sensor	0.0 to 100 mS/cm
SBE 19*plus* SEACAT Profiler		
Temperature	SBE 19*plus*	-5 to +35°C
pH	SBE 18 pH Sensor	0.0 to 14.0 standard pH units
Dissolved Oxygen	SBE 43 Dissolved Oxygen Sensor	120% of surface saturation in all natural waters, fresh and salt
Conductivity/ Specific Conductance	SBE 19*plus*	0.0 - 7.0 Siemens/meter (S/m)
Chlorophyll *a*	SBE 19*plus*	0 to 5 µg/L

Table 4: Quality Control Requirements for Field Measurements Collected with a YSI 6920V2 Environmental Monitoring System

Field Parameters: Temperature, pH, Dissolved Oxygen, Turbidity, Conductivity/Specific Conductance[1]

QC Sample:	Data Quality Indicator (DQI)[2]	Frequency/ Number	Method/SOP QC Acceptance Limits[3]	Acceptance Criteria/ Measurement Performance Criteria[4]	Corrective Action
Temperature					
Field Duplicate	Precision (S & A)	1/5 field samples	NA	±0.5°C	Collect & analyze 3rd sample. Qualify data, if still exceeding criteria.
QC Check Sample[5]	Accuracy	NA	NA	NA	None. Sensor not used if didn't meet calibration criteria.
pH					
Field Duplicate	Precision (S & A)	1/5 field samples	NA	±0.3 pH units	Collect & analyze 3rd sample. Qualify data, if still exceeding criteria.
QC Check Sample[6]	Accuracy	1/batch (each day)	±0.5 units of true value for both calibration check standards	±0.5 units of true value	Qualify associated field data.
Dissolved Oxygen					
Field Duplicate	Precision (S & A)	1/5 field samples	NA	±20% RPD	Collect & analyze 3rd sample. Qualify data, if still exceeding criteria.
QC Check Sample[6]	Accuracy	1/batch (each day)	±0.5 mg/L of true value of full saturation standard	±0.5 mg/L of true value	Qualify associated field data.
Turbidity					
Field Duplicate	Precision (S & A)	1/5 field samples	NA	±20% RPD	Collect & analyze 3rd sample. Qualify data, if still exceeding criteria.
QC Check Sample[6]	Accuracy	1/batch (each day)	±20% or ±2 NTU of 20 NTU standard (whichever is greater) and ±1 NTU for 0 NTU standard	±20% of true value	Qualify associated field data.
Conductivity/Specific Conductance					
Field Duplicate	Precision (S & A)	1/5 field samples	NA	±20% RPD	Collect & analyze 3rd sample. Qualify data, if still exceeding criteria.
QC Check Sample[6]	Accuracy	1/batch (each day)	±10% of true value or ±0.2 mS/cm (whichever is greater) for calibration check standard	±10% of true value	Qualify associated field data.

Notes:
[1] Methods are provided in Appendix A.
[2] Data Quality Indicators may be related to sampling (S) and/or analysis (A) activities.
[3] For field duplicate samples, there are no method-specific QC acceptance limits (NA- Not applicable.)
[4] The information in this column supports acceptance criteria/measurement performance criteria introduced in Section 1.7.3. For this study, the field measurement's QC acceptance limits (as determined from a calibration check sample analyzed half-way through the field day) were reviewed and found acceptable to meet the current data quality needs. As such, the field measurement's QC acceptance limits and the project's measurement performance criteria are equivalent.
[5] Accuracy is not ensured through the analysis of a QC check. If the temperature sensor meets the annual calibration procedures and criteria presented in Table 12, the measurements are considered accurate enough to meet the needs of the current project.
[6] Accuracy is ensured through the calibration and calibration check process presented in Table 12. The post calibration check sample(s) will be considered as QC check samples for the field measurements.

ALL SAMPLES ARE SURFACE WATER MATRIX. ALL SAMPLES ARE COLLECTED BY THE SAME PROCEDURE, AS PRESENTED IN APPENDIX A. NO ADDITIONAL QC CHECKS ARE PLANNED BEYOND THOSE IDENTIFIED ABOVE FOR ACCURACY AND PRECISION.

Table 5: Quality Control Requirements for Laboratory Analyses

See Appendix D for Laboratory Data Quality Indicator Tables.

QC Sample:	Data Quality Indicator (DQI)[1]	Frequency/Number	Acceptance Criteria/Measurement Performance Criteria[2]	Corrective Action[3]
Field:				
Field Duplicate	Precision (S & A)	1/10 field samples	RPD</= 20% for concentrations >5 x QL	Qualify associated field data and/or resample.
Field Blank	Accuracy/ Bias as Contamination (S & A)	1/20 field samples	Concentration < QL	Qualify associated field data and/or resample.
Temperature Blank	Representative-ness	1/cooler of samples	4°C ± 2°C	Contact Tribe's Project Manager
Laboratory:				
Duplicate Samples	Precision (A)	35% of samples	# of Duplicates ≥ 35% of Samples	Review with lab manager. Reanalyze or justify in data report.
Duplicate Sample Variability	Precision (A)	N/A	<20% of Duplicates exceeds maximum variability[4]	Review with lab manager. Reanalyze or justify in data report.
Matrix Spike	Accuracy/Bias as Recovery (S & A)	With each set of samples analyzed	± 15% from expected value	Reprep and reanalyze. If problem recurs, justify in data report.

Notes:

[1] Data quality indicators may be related to sampling (S) and/or analysis (A) activities.

[2] The information in this column supports the acceptance criteria/measurement performance criteria introduced in Section 1.7.3. For this study, the laboratory's QC acceptance limits were reviewed and found acceptable to meet the current data quality needs. As such, the laboratory's QC acceptance limits and the project's measurement performance criteria are equivalent.

[3] Tribe's project manager will make decision on how to proceed on a case-by-case basis. At a minimum, a note will be included with the data report from the laboratory.

[4] Variability Limits

DL = Detection Limit

Calculation of Limits

Interval	RSD%
<2 · DL	100%
2-3 · DL	80%
3-4 · DL	60%
4-5 · DL	40%
>5 · DL	20%

Table 6: Summary of Water Samples and Analytical Methods

Analytical Parameter	**Analytical Method Number[1]**	**Containers (Number, Size/Volume, type)**	**Preservation Requirements[2] (conducted in lab)**	**Maximum Holding Times**
Analyses				
Total Phosphate	365.3, EPA	1 Liter Polyethylene Bottle	Do Not filter samples. Add 2 ml H_2SO_4/L, Refrigerate [5]	28 days
Ortho-Phosphate	365.3 EPA	1 Liter Polyethylene Bottle	Filter Samples. Add 2 ml H_2SO_4/L, Refrigerate [5]	28 days
Nitrate + Nitrite	4500-NO_3^- E Standard Methods, 20th Edition	1 Liter Polyethylene Bottle	Filter samples. - w/in 48 hrs of collection: Refrigerate[5] - w/in 28 days of collection: Add 2 mL H_2SO_4/L, Refrigerate[5]	28 days
Ammonia-N	4500-NH_3 F Standard Methods, 20th Edition	1 Liter Polyethylene Bottle	Filter samples. - w/in 24 hrs of collection: Refrigerate[5] - w/in 28 days of collection: Freeze at -20°C or add H_2SO_4 to pH <2and refrigerate[5]	28 days
Total Kjeldahl Nitrogen	4500-N_{org} C Standard Methods, 20th Edition	1 Liter Polyethylene Bottle	Add H_2SO_4 to pH 1.5 to 2.0 and refrigerate[5]	28 days
Dissolved Inorganic Nitrogen	Calculation[3]	NA	NA	NA
Total Nitrogen	Calculation[4]	NA	NA	NA
Zooplankton	SOP: Zooplankton Analysis	250 ml Polyethylene Bottle	Add 3-5 mL Lugols solution	28 days
Field Measurements				
Temperature, pH, Dissolved Oxygen, Specific Conductivity, Turbidity	See SOP for YSI 6920V2 in Appendix A	NA	NA	Immediate
Temperature, pH, Dissolved Oxygen, Conductivity	See SOP for SEACAT SBE 19 plus in Appendix A	NA	NA	Immediate

[1] SM = Standard Methods for the Examination of Water & Wastewater, 20th Editions; APHA, AWWA, WPCF, American Public Health Association, Washington, DC.
[2] H_2SO_4 = Sulfuric acid
[3] Dissolved Inorganic Nitrogen (DIN): DIN = Total Ammonia + Nitrate + Nitrite
[4] Total Nitrogen (TN): TN = Total Kjeldahl Nitrogen + Nitrate + Nitrite
[5] Refrigerate = storage at 4°C ± 2°C, in the dark

Table 7: Sample Design and Rationale - Pyramid Lake

Sampling Location/ ID Number[3]	**Location**	**Rationale for Sampling Design**
Pyramid Lake/ WP96[1]	North (deep) Basin	Lake stratification, lake mixing (turn-over), productivity, and 'Control Point' for PLPT WQS.
Pyramid Lake/ WP93[2]	South (shallow) Basin	Monitors water entering from the Truckee River into the lake during times of mixing.

Notes:
[1] Pyramid Lake water samples at Station 96 will be collected monthly from discrete depths (10m, 20m, 30m, 45m, 60m, 75m, 90m) including a composite sample (surface + 2.5m + 5m depths) and a sample 5m from the bottom.
[2] Pyramid Lake water samples at Station 93 will be collected quarterly from discrete depths (10m, 20m, 30m, and 45m) including a composite sample (surface + 2.5m + 5M) and a sample 5m off the bottom.
[3] All samples will be analyzed for the analytical parameters and field measurements listed in Table 1.

Table 8: Sample Design and Rationale – Truckee River (monthly)

Sampling Location/ ID Number[1]	**Location**	**Rationale for Sampling Design**
Truckee River/ PD	Pierson Diversion (most upstream site)	Monitors upstream water entering the Pyramid Lake Indian Reservation.
Truckee River/ WB	Wadsworth Bridge	Monitoring Control Point (#1) for the PLPT WQS.
Truckee River/ DO	Dead Ox	Monitoring for TDS and nutrients from groundwater return flow.
Truckee River/ NB	Nixon Bridge	Monitoring Control Point (#2) for the PLPT WQS.
Truckee River/ MBD	Marble Bluff Dam (most downstream site)	Site below Marble Bluff Dam just upstream of Pyramid Lake.

Notes:
[1] If the water source is less than 12" deep, samples will be collected at mid depth and noted in the field logbook. All samples will be analyzed for the analytical parameters and field measurements listed in Table 2.

Table 9: Sample Design and Rationale – Truckee River (annual)[1]

Sampling Location/ ID Number[1,2]	**Location**	**Rationale for Sampling Design**
Truckee River/ I80	Interstate-80 Bridge	Water quality monitoring of bioassessment sample sites.
Truckee River/ BB	Big Bend	Water quality monitoring of bioassessment sample sites.
Truckee River/ WB	Wadsworth Bridge	Water quality monitoring of bioassessment sample sites.
Truckee River/ FN	Fellnagle	Water quality monitoring of bioassessment sample sites.
Truckee River/ SS	S Bar S Ranch	Water quality monitoring of bioassessment sample sites.
Truckee River/ DO	Dead Ox	Water quality monitoring of bioassessment sample sites.
Truckee River/ CYN	Canyon	Water quality monitoring of bioassessment sample sites.
Truckee River/ NU	Nixon Upper	Water quality monitoring of bioassessment sample sites.
Truckee River/ NB	Nixon Bridge	Water quality monitoring of bioassessment sample sites.
Truckee River/ NL	Nixon Lower	Water quality monitoring of bioassessment sample sites.
Truckee River/ MBD	Marble Bluff Dam	Water quality monitoring of bioassessment sample sites.

Notes:
[1] For further information on location rational refer to the Quality Assurance Project Plan for Bioassessment Monitoring in Surface Waters of the Pyramid Lake Indian Reservation, Nevada: Pyramid Lake Paiute Tribe Stream Bioassessment Procedure.
QA EPA Office Document Control Number: WATR469Q04VSF1
[2] If the water source is less than 12" deep, samples will be collected at mid depth and noted in the field logbook. All samples will be analyzed for the analytical parameters and field measurements listed in Table 2.

Table 10: Sample Design and Rationale – Non Point Source Sites

Sampling Location/ ID Number[1]	**Location**	**Rationale for Sampling Design**
Paiute Pit Outlet/ PP	Paiute Pit Gravel Operation Wadsworth, NV	Monitors quality of water from the "Pit Lake" (resulting from gravel pit operations) before entering the Truckee River.
Tile Drain Outlet/ TD	Agricultural field Hill Ranch Road Wadsworth, NV	Monitors quality of agricultural return flows (irrigation season) resulting from the tile drains, before entering the Truckee River.
Numana Wetlands Inlet/ NWI	Behind Numana Hatchery HWY 447	Monitors wastewater from Numana Fish Hatchery pools entering into the Numana Wetlands complex.
Numana Wetlands Outlet/ NWO	Behind Numana Hatchery HWY 447	Monitors efficiency of Numana Wetlands to reduce nutrient discharge into the Truckee River.

Notes:
[1] If the water source is less than 12" deep, samples will be collected at mid depth and noted in the field logbook. All samples will be analyzed for the analytical parameters and field measurements listed in Table 2.

Table 11: Sample Design and Rationale - Mountain Streams

Sampling Location/ ID Number[1]	**Location**	**Rationale for Sampling Design**
Big Canyon Creek/ BC	Virginia Mountain Range (on the PLIR boundary)	Monitors WQ entering PLIR from Big Canyon Ranch operations.
Sharpe's Canyon Creek/ SC	Virginia Mountain Range (above Whittey's ranch)	Monitors WQ where livestock are 'permitted' to graze.
Thunderbolt Canyon Creek/ TB	Virginia Mountain Range (above Whittey's ranch)	Monitors WQ where livestock are 'permitted' to graze.
Poison Canon Creek/ PC	Virginia Mountain Range (above Whittey's ranch)	Monitors WQ where livestock are 'permitted' to graze.
Hardscrabble Creek/ HS1	Virginia Mountain Range (on ranch boundary)	Monitors WQ entering the PLIR from Horgan Ranch operations.
Hardscrabble Creek/ HS2	Sutcliffe, NV (near Pyramid Lake)	Monitors WQ entering Pyramid Lake (town of Sutcliffe).
Rodero Creek/ ROC	Pah Rah Mountain Range (near Block House)	Monitors WQ where livestock are 'permitted' to graze.
Tom Anderson Canyon/ TAC	Pah Rah Mountain Range (near Popcorn Rock)	Monitors WQ where livestock are 'permitted' to graze.
Coal Canyon Creek/ CC	Pah Rah Mountain Range (near TAC)	Monitors WQ where livestock are 'permitted' to graze.
Big Mouth Canyon Creek/ BMC	Pah Rah Mountain Range (near Coal Canyon)	Monitors WQ where livestock are 'permitted' to graze.
Dove Creek/ DOV	Lake Mountain Range (east of HWY 447)	Monitors WQ where livestock are 'permitted' to graze.
Nugent Hole Canyon/ NHC	Lake Mountain Range (east of HWY 447)	Monitors WQ where livestock are 'permitted' to graze.
Nugent Canyon/ NUC	Lake Mountain Range (east of HWY 447)	Monitors WQ where livestock are 'permitted' to graze.

Notes:
[1] If the water source is less than 12" deep, samples will be collected at mid depth and noted in the field logbook. All samples will be analyzed for the analytical parameters and field measurements listed in Table 2.

Table 12: YSI 6920V2 Instrument Calibration, Maintenance, Testing, and Inspection

Analytical Parameter	Instrument	Calibration Activity	Maintenance & Testing/ Inspection Activity	Frequency	Acceptance Criteria	Corrective Action
Temperature	6560 Conductivity/ Temperature Sensor	NA[1]	See manufacturer's manual	NA[1]	NA[1]	Remove from use if not working.
pH	6561 pH Sensor	Initial: Two-point calibration bracketing expected filed sample range (using 7.0 and either 4.0 or 10.0 pH buffer, depending on field conditions) Post: Two-point check with 7 pH and 10 pH buffers	See manufacturer's manual	Initial: Beginning of each day Post: End of each day	Initial: Two-point calibration done electronically Post: ±0.5 pH units of true value with both 7 pH and 10 pH buffers	Initial: Recalibrate Post: Qualify data
Dissolved Oxygen	6150 ROX Dissolved Oxygen Optical Sensor	Initial: One-point calibration with saturated air (need barometric pressure) Post: Single-point check at full saturation	See manufacturer's manual	Initial: Beginning of each day Post: End of each day	Initial: One point calibration done electronically Post: ±0.5 mg/L of true saturated value	Initial: Recalibrate; change membrane and recalibrate Post: Qualify data
Turbidity	6136 Turbidity Sensor	Initial: Two-point calibration using 0 NTU (or deionized water) and 100 NTU standards to bracket expected sample range Post: Two-point check with high (100 NTU) and low (0 NTU) standards	See manufacturer's manual	Initial: Beginning of each day Post: End of each day	Initial: Two-point calibration done electronically Post: Two point check with high (100 NTU) standard ±20% or ±2 NTU (whichever is greater) of true value and low (0 NTU) standard ±1 NTU of true value	Initial: See manufacturer's manual Post: Qualify Data
Conductivity/ Specific Conductance	6560 Conductivity/ Temperature Sensor	Initial: one-point calibration using 6.668 mS/cm standard Post: One-point check with 6.668 mS/cm standard	See manufacturer's manual	Initial: Beginning of each day Post: End of each day	Initial: one-point calibration done electronically Post: One-point check with 6.668 standard ±10% of true value or ±0.2 mS/cm, whichever is greater	Initial: Recalibrate Post: Qualify data

[1] No calibration or maintenance of the temperature sensor is required.

Table 13: SBE 19*plus* SEACAT Profiler Calibration, Maintenance, Testing, and Inspection

Analytical Parameter	Instrument	Calibration Activity	Maintenance & Testing/ Inspection Activity	Frequency	Acceptance Criteria	Corrective Action
Temperature	Temperature Sensor	Calibration done by manufacturer.	See manufacturer's manual.	Annual	NA	Calibration done by manufacturer.
pH	pH Sensor	Calibration done by manufacturer.	See manufacturer's manual.	Annual	NA	Calibration done by manufacturer.
Dissolved Oxygen	DO Sensor	Calibration done by manufacturer.	See manufacturer's manual.	Annual	NA	Calibration done by manufacturer.
Conductivity	Conductivity Sensor	Calibration done by manufacturer.	See manufacturer's manual.	Annual	NA	Calibration done by manufacturer.
PAR	Photosynthetic Active Radiation Sensor	Calibration done by manufacturer.	See manufacturer's manual.	Annual	NA	Calibration done by manufacturer.
Chlorophyll	Chlorophyll sensor	Calibration done by manufacturer.	See manufacturer's manual.	Annual	NA	Calibration done by manufacturer.

Manufacturer recommends that the 19*plus* be returned to Sea-Bird for calibration annually.

Table 14: GPS Coordinates of Sampling Locations

Sample Site	Latitude	Longitude
Pyramid Lake Stations		
Station 96	39.06656	-119.5600
Station 93	39.98098	-119.4873
Truckee River Monthly Sites		
Pierson Diversion	39.61307	-119.3034
Wadsworth Bridge	39.63193	-119.2834
Dead Ox	39.74020	-119.3201
Nixon Bridge	39.74581	-119.3562
Marble Bluff Dam	39-85306	-119.3994
Non Point Source Sites		
Paiute Pit Outlet	39.65086	-119.2798
Tile Drain	39.66803	-119.2706
Numana Wetland Inlet	39.19777	-119.3183
Numana Wetland Outlet	39.72413	-119.3191
Truckee River Bioassessment Sites		
I-80	39.61470	-119.2992
Big Bend	39.62097	-119.2897
Wadsworth Bridge	39.63193	-119.2833
Fellnagle	39.64301	-119.2912
S Bar S	39.69533	-119.2919
Dead Ox	39.74020	-119.3201
Canyon	39.77818	-119.3374
Nixon Upper	39.80341	-119.3510
Nixon Bridge	39.82915	-119.3562
Nixon Lower	39.83940	-119.3694
Marble Bluff Dam	39.85306	-119.3994
Stream Sites		
Big Canyon	40.08621	-119.7209
Sharpe's	40.07650	-119.7163
Thunderbolt	40.07068	-119.7193
Poison	40.06361	-119.7159
Hardscrabble (HS1)	39.94345	-119.6183
Hardscrabble (HS2)	39.95248	-119.6012
Rodero	39.87256	-119.4969
Tom Anderson	39.83879	-119.4732
Coal Canyon	39.80741	-119.4252
Big Mouth Canyon	39.77572	-119.4175
Dove Creek	40.08267	-119.4064
Nugent Hole	40.14713	-119.4087
Nugent Canyon	40.16030	-119.4061

Appendices

Appendix A: Field Standing Operating Procedures

Standard Operating Procedure for:

Calibration and Field Measurement Procedures for the YSI Model 6920V2 Sonde and Data Logger

1.0 Introduction

The YSI 6920V2 sonde is a multi-parameter device used to measure and record water quality measurements in surface waters for the following: temperature, dissolved oxygen, pH, specific conductivity, salinity, total dissolved solids and turbidity.

2.0 Purpose

The standard operating procedure (SOP) describes the method to calibrate and use the YSI 6920V2 sonde in the field.

3.0 Method

See the YSI 6-Series Multiparameter Water Quality Sondes User Manual for complete instructions for maintenance, care, and use of the 6920V2 sonde and sensors.

4.0 Pre-Calibration Checks

4.1 On the day before a calibration and sampling event inspect the YSI 6920V2 Sonde.

4.1.1 Remove the calibration cup from the sonde.
4.1.2 Confirm that the sponge stored in the calibration cup has not dried out, keeping the sensors moist.
4.1.3 Inspect the Turbidity Sensor to see if wiper needs to be changed. If it does, then follow the directions found in the YSI User's Manual to change the wiper and record in equipment log book.
4.1.4 Inspect the Dissolved Oxygen sensor to see if wiper needs to be changes. If it does, then follow the direction found in the YSI User Manual to replace the wiper and record in equipment log book.
4.1.5 Inspect all other sensors and plugs to make sure they are all inserted properly.
4.1.6 Re-wet the sponge that is stored in the calibration cup and attach cup on sonde.

4.2 Also on the day before calibration, make sure that there are sufficient amounts of calibration standards available at the Environmental Department building. If not then it is necessary to get calibration standards from the PLPT WQ Laboratory and/or reorder.

5.0 Materials Needed for YSI 6920 Sonde Calibration

- Equipment stand with clamp
- Conductivity 6.668 calibration standard
- pH 7 and 10 calibration standards
- Turbidity 0 and 126 NTU calibration standards

- Sonde
- Calibration cup (on bottom)
- MDS 650 display
- Connection cable
- YSI Calibration Worksheet

6.0 Calibrations

6.1 Obtain an YSI 6920V2 Sonde Calibration Worksheet (See Worksheet Below). Record all calibration data onto this worksheet.
6.2 Record the date of calibration.
6.3 Record the names of the calibration technician(s).
6.4 Indicate by circling 'Y' or 'N' if the dissolved oxygen sensor wiper has been changed, this information can be found in the equipment log book.
6.5 Indicate by circling 'Y' or 'N' if the turbidity wiper has been changed, this information can be found in the equipment log book.
6.6 Remove the calibration cup from the sonde and remove the small sponge that is stored in the cup while sonde is not in use.
6.7 Visually inspect the sensors to ensure that they are free from any debris and electrodes are in good condition as well as the turbidity and dissolved oxygen wipers.
6.8 Indicate by circling 'Y' or 'N' if the turbidity wiper is parked about 180° from the optics. This can be done by visually inspecting the turbidity probe.
6.9 Indicate by circling Y' or 'N' if the dissolved oxygen wiper is parked about 180° from the optics. This can be done by visually inspecting the dissolved oxygen probe.
6.10 Check that the sonde is connected to the 650 MDS Display with the provided cable.

6.11 <u>Dissolved Oxygen Calibration</u>

6.11.1 Dry the temperature sensor and the ROX membrane to remove any water droplets.
6.11.2 Place approximately 1 inch of tap water in the calibration cup. The calibration cup twists on/off the bottom of the sonde.
6.11.3 Place the probe end of the sonde into the cup, making sure that the D.O. and temperature probes are not immersed in the water.
6.11.4 Place the calibration cup onto the sonde and thread onto the sonde ½ thread. Place sonde in an upside down position on a stand.
6.11.5 Wait approximately 15 minutes for the air in the calibration cup to become water saturated and the temperature to equilibrate.
6.11.6 While waiting, call the Reno weather station at (775) 673-8107, and ask for the "corrected" barometric pressure for the Nixon, Nevada area. Record this number in the first space in the 'Barometric Calculation' section.
6.11.7 Barometric Calculation:
  6.11.7.1. Multiply the "corrected" barometric pressure by 25.4, and then subtract 98.5 (for elevation correction). Record the value in the space provided.
  6.11.7.2. Divide the value received in 6.11.7.1 by 760, and then multiply by 100. Record the value in the space provided. This is the % DO Check Value.
6.11.8 Power the sonde by pressing the green power button, on the upper left hand corner of the 650 MDS display unit (connected to the sonde by a cable).

6.11.9 Using the 'down arrow' cursor key, select "Sonde menu" from the 650 MDS display menu, then press the enter key.
6.11.10 Using the cursor key, select "Calibrate" from the main menu, and then press 'enter.'
6.11.11 Using the cursor key, select "Optic-T Dissolved Oxy" from the menu, and then press 'enter.'
6.11.12 Select "ODOsat%" from the DO calibration menu, then press the enter key.
6.11.13 Using the cursor key, select "1 point" from the menu, and press 'enter'.
6.11.14 Enter the value from step 6.11.7.1 into the 650 MDS using the numbers on the touch pad.
6.11.15 Press the 'enter' key. The sonde will enable all sensors. Wait for one minute while sensors stabilize.
6.11.16 Record the OSO mg/L measurement in the 'Before Calibration' column.
6.11.17 Use the cursor key to select "Calibrate", press 'enter'.
6.11.18 The current values of all enabled sensors will appear on the screen, observe the reading under 'DO %'. Record the reading in the 'After Lab Calibration' column.
6.11.19 Compare the reading to the % DO Check Value. Record a checkmark on the calibration worksheet if the reading is equal to the % DO Check Value obtained in Step 6.11.7.2.
6.11.19.1. If it is not equal then repeat steps 6.11.1 - 6.11.19. If calibration value is still not correct see user's manual for troubleshooting.
6.11.20 Observe the reading under 'ODO %' and record the reading in the 'After Lab Calibration' column.
6.11.21 Record the Battery Voltage displayed on the 650 MDS.
6.11.22 Press the 'enter' key to continue calibration.
6.11.23 Press the 'escape' key twice on the touch pad to go back to the main menu.
6.11.24 Using the cursor key, select "Advanced", press 'enter'.
6.11.25 Using the cursor key select "Cal Constants", press 'enter'.
6.11.26 Record the "ODO gain" calibration constant on the Calibration Worksheet.
6.11.26.1. This number should be 0.85 - 1.15. If it is out of range see YSI Users manual for more information
6.11.27 Press the "Esc" key twice to get back to the main menu. Using the cursor select "Calibrate".
6.11.28 Discard water in calibration cup, and rinse probes twice with tap water.

6.12 Specific Conductivity Calibration

6.12.1 From the calibration menu, use 'up arrow' cursor key to select "Conductivity", then press the enter key.
6.12.2 Select "Sp Cond" from the "Cond calibration" menu, then press the enter key.
6.12.3 Fill the calibration cup with 2.5 cm (1 inch) of the 6.668 mS/cm conductivity standard.
6.12.4 Replace the calibration cup on the sonde, shake 4 seconds to rinse the probes, and then discard the conductivity solution.
6.12.5 Fill the calibration cup with 200 ml of the 6.668 mS/cm conductivity standard, covering all sensors. Replace the calibration cup gently moving the sonde up and down to remove any bubbles from the conductivity cell. The probe must be completely immersed past its vent hole. Place the sonde in an upside down position on a stand (see figure above).
6.12.6 Enter the standard calibration mS/cm value of "6.668" into the 650 MDS using the numbers on the touch pad. Then press the enter key.
6.12.7 Wait one minute for the sonde to calibrate. The current values of all enabled sensors will appear on the screen and will change in time as they stabilize.
6.12.8 Observe the readings under 'mS/cm'. When they show no significant change for approximately 30 seconds, record the specific conductivity reading in the 'Before Calibration' column.

6.12.9 Press the 'enter' key to calibrate and record the specific conductivity reading in the 'After Lab Calibration" column.
6.12.10 The 6920V2 sonde is now calibrated at 25 °C for Specific conductivity to be read in the 0 –100 mS/cm range.
6.12.11 Press the 'enter' key, to continue. Press the 'Esc' twice to go to the main menu.
6.12.12 Using the cursor key, select "Advanced", press 'enter'.
6.12.13 Using the cursor key select "Cal Constants", press 'enter'.
6.12.14 Record the "Cond" calibration constant on the Calibration Worksheet.
6.12.14.1. This number should be 5.0 +/- 0.45. If it is out of range see YSI Users manual for more information
6.12.15 Press the "Esc" key twice to get back to the main menu. Using the cursor select "Calibrate".
6.12.16 Discard conductivity solution, and rinse probes twice with tap water.

6.13 <u>pH Calibration</u>

6.13.1 From the calibration menu, use 'up arrow' cursor key to select "ISE1 pH", then press the enter key.
6.13.2 Select "2 point" from the "pH calibration" menu, then press the enter key.
6.13.3 Fill the calibration cup with 2.5 cm (1 inch) of the 7.0 pH standard.
6.13.4 Replace the calibration cup on the sonde, shake to rinse the probes, and then discard the solution.
6.13.5 Fill the calibration cup with 100 ml of the 7.0 pH standard, enough to cover all sensors. Replace calibration cup. Place the sonde in an upside down position on a stand (see figure above).
6.13.6 Enter the standard calibration pH value of "7.00" into the 650 MDS using the numbers on the touch pad. Then press the enter key. Wait one minute for the sensors to stabilize.
6.13.7 Observe the readings under "pH", and when they show no significant change for approximately 30 seconds, record the value in the 'Before Calibration' column.
6.13.8 Press the 'enter' key to calibrate, and record the calibrated value in the 'After Lab Calibration' column.
6.13.9 Record the pH MV reading on the 650 MDS in the 'Diagnostic Check' column.
6.13.10 Press the 'enter' key on the touch pad to continue with the 2-point pH calibration.
6.13.11 Discard ph calibration solution, and rinse probes twice with tap water.
6.13.12 Repeat steps 6.12.3 to 6.12.11 using the pH 10.0 standard.
6.13.13 The 6920V2 sonde is now calibrated for pH.
6.13.14 Check the pH MV slope.
6.13.14.1. The difference between the pH MV readings should be between 165-180. If the difference is outside of these limits then the pH probe needs to be replaced.
6.13.15 Press 'escape' to get back to the calibration menu.

6.14 <u>Turbidity Calibration</u>

6.14.1 From the calibration menu, use 'up arrow' cursor key to select "Optic T- Turbidity", then press the enter key.
6.14.2 Select "2 point" from the turbidity calibration menu, then press the enter key.
6.14.3 Fill the calibration cup with 2.5 cm (1 inch) of the 0 NTU standard or deionized water.
6.14.4 Replace the calibration cup on the sonde, shake to rinse the probes, and then discard the solution.

6.14.5 Fill the calibration cup with 100 ml of the 0 NTU standard, enough to cover all sensors. Replace calibration cup. Place the sonde in an upside down position on a stand (see figure above).
6.14.6 Enter the standard calibration value of "0.0" into the 650 MDS using the numbers on the touch pad. Then press the enter key. Wait one minute for the sensors to stabilize.
6.14.7 Observe the readings under "NTU", and when they show no significant change for approximately 30 seconds, record the value in the 'Before Calibration' column.
6.14.8 Press the 'enter' key to calibrate, and record the calibrated value in the 'After Lab Calibration' column.
6.14.9 Press the 'enter' key on the touch pad to continue with the 2-point Turbidity calibration.
6.14.10 Discard 0 NTU calibration solution, and rinse probes twice with tap water.
6.14.11 Repeat steps 6.14.3 to 6.14.10 using the 126 NTU standard.
6.14.12 Press 'escape' to get back to the calibration menu.

6.15 Salinity Calibration

6.15.1 Salinity is determined automatically from the sonde conductivity and temperature readings. See YSI "Environmental Monitoring Systems Operations Manual" Section 5.2 for a detailed explanation**.**

6.16 Total Dissolved Solids Calibration

6.16.1 Total Dissolved Solids Calibration (grams/liter) is determined automatically from the sonde conductivity, multiplied by a default constant of 0.65. See YSI "Environmental Monitoring Systems Operations Manual" Section 5.3 for a detailed explanation.

6.17 Press the 'escape' key twice to disconnect the 650 MDS from the sonde and then press the 'power' key to turn off the equipment.
6.18 Moisten a 1.0 inch sponge is and insert into the bottom of the calibration/storage cup. The sponge will keep the sensors moist.
6.19 Attach the calibration cup to the sonde, calibration cup will remain in place until sonde is used in the field.

7.0 Procedure: In Field

7.1 Remove calibration cup, and replace with the sonde sensor 'protector' cap.
7.2 The entire sonde should be immersed in the water to be sampled. The sensors should be cleaned and free of any debris.
7.3 Power on the sonde, using the cursor key highlight, "Sonde run" then press the 'enter' key to enable the sensors. The 650 MDS will display all active parameters.
7.4 Observe the readings under Temperature (ºC), Dissolved Oxygen (DO mg/L), and Specific Conductivity (mS/cm). When they show no significant change (approximately 60 seconds), record the measurements in field notebook and/or pre-printed water quality monitoring worksheet using a pencil.
7.5 Power off the sonde, remove sonde from water and replace the calibration cup. Be sure to remove any debris from the sonde before replacing the calibration cup.

8.0 Post Field Calibrations

8.1 After field use rinse sonde, probes, 'protector' cap, and calibration cup with tap water to remove any debris that may have become attached to equipment in the field.
8.2 Record post calibration QC check data on the calibration sheet in the 'Post Calibration' column.

8.3 Dissolved Oxygen Post Calibration QC Check

8.3.1 Place approximately 3 mm (1/8 inch) of tap water in the calibration cup. The calibration cup twists on/off the bottom of the sonde.
8.3.2 Place the probe end of the sonde into the cup, making sure that the D.O. and temperature probes are not immersed in the water.
8.3.3 Engage only 1 or 2 threads of the calibration cup to insure that the probe is vented to the atmosphere. Place sonde in an upside down position on a stand.
8.3.4 Wait approximately 10 minutes for the air in the calibration cup to become water saturated and the temperature to equilibrate.
8.3.5 Power on the sonde, using the cursor key highlight, "Sonde run" then press the 'enter' key to enable the sensors. The 650 MDS will display all active parameters.
8.3.6 Observe the readings when they show no significant change (approximately 60 seconds), record the % dissolved oxygen and mg/L measurements in the 'Post Calibration' column.
8.3.7 Remove calibration cup and rinse probes twice with tap water.

8.4 Specific Conductivity Post Calibration QC Check

8.4.1 Fill the calibration cup with 2.5 cm (1 inch) of the 6.668 mS/cm conductivity standard.
8.4.2 Replace the calibration cup on the sonde, shake 4 seconds to rinse the probes, and then discard the solution.
8.4.3 Fill the calibration cup with 200 ml of the 6.668 mS/cm conductivity standard, covering all sensors. Replace the calibration cup gently moving the sonde up and down to remove any bubbles from the conductivity cell. The probe must be completely immersed past its vent hole. Place the sonde in an upside down position on a stand (see figure above).
8.4.4 Observe the readings under 'mS/cm'. When they show no significant change for approximately 30 seconds, record the reading in the 'Post Calibration' column.
8.4.5 Discard solution, and rinse probes twice with tap water.

8.5 pH Post Calibration QC Check

8.5.1 Fill the calibration cup with 2.5 cm (1 inch) of the pH 7.0 standard.
8.5.2 Replace the calibration cup on the sonde, shake 4 seconds to rinse the probes, and then discard the solution.
8.5.3 Fill the calibration cup with 200 ml of the pH 7.0 standard, covering all sensors. Place the sonde in an upside down position on a stand.
8.5.4 Observe the readings under 'pH'. When they show no significant change for approximately 30 seconds, record the reading in the 'Post Calibration' column.
8.5.5 Discard solution, and rinse probes twice with tap water.
8.5.6 Repeat steps 8.5.1 - 8.5.5 with the ph 10.0 standard.

8.6 Turbidity Post Calibration QC Check

8.6.1 Fill the calibration cup with 2.5 cm (1 inch) of the 0 NTU standard.

8.6.2 Replace the calibration cup on the sonde, shake 4 seconds to rinse the probes, and then discard the solution.

8.6.3 Fill the calibration cup with 200 ml of the 0 NTU standard, covering all sensors. Place the sonde in an upside down position on a stand.

8.6.4 Observe the readings under 'NTU'. When they show no significant change for approximately 30 seconds, record the reading in the 'Post Calibration' column.

8.6.5 Discard solution, and rinse probes twice with tap water.

8.6.6 Repeat steps 8.6.1 - 8.6.5 with the 100 NTU standard.

8.7 Press the 'escape' key to disconnect the 650 MDS from the sonde and then press the 'power' key to turn off the equipment.

8.8 Moisten a 1.0 inch sponge is and insert into the bottom of the calibration/storage cup. The sponge will keep the sensors moist.

8.9 Attach the calibration cup to the sonde engaging all threads.

8.10 Complete the Post Calibration QC Check equations on the Calibration Worksheet. Store Calibration Worksheets in binder for future reference.

9.0 Calibration Standards

9.1 Conductivity Standard: This is made at the PLPT WQ Laboratory. 74.557 grams of Potassium Chloride (KCl) is dissolved in 700 mL of deionized water, and then diluted to one liter with deionized water. This produces a 111.9 mS/cm solution. The 6.668 mS/cm standard is made by taking 50 mL of the 111.9 mS/cm solution, and diluting to one liter with deionized water.

9.2 Turbidity and pH standards are purchased commercially. See YSI user's manual for acceptable standards.

10.0 Storage

10.1 <u>After field use</u>: Rinse the sensors with tap water, making sure there is no debris in the sensors. Place 0.5 inch of tap water inside the calibration cup before placing on the sonde. No sensor should be immersed in water. The calibration cup should be sealed to prevent evaporation. The storage chamber should remain at 100% humidity. Store upside down and close to room temperature the day prior to calibration.

10.2 <u>For long-term storage (over 45 days):</u> Store the pH sensor in ORP solution (provided with instrument) to prevent the probe from drying out. After removing this sensor from sonde, replace it with a plug (provided with the sonde). All other sensors can remain on the sonde. See YSI "Environmental Monitoring Systems Operations Manual" Section 2.10 for detailed explanation of the care, maintenance, and storage of the 6920 sonde/ probes.

10.3 <u>Winter (off season) Storage</u>: Remove batteries from logging instruments. Power down instruments by turning them off and removing batteries. Remove all organic material from instrument. If multiprobe has pins, remove cables and attach dummy plugs. Fill storage cup full of tap water and make sure all sensors are submerged.

11.0 References

YSI Incorporated 6-Series Multiparameter Water Quality Sondes User Manual; September 2009, Revision F, Item #069300; YSI Inc.; 1700/1725 Brannum Lane; Yellow Springs, Ohio 45387.

YSI 6920V2 Sonde Calibration Worksheet

YSI 6920V2 Sonde Calibration Worksheet

Date of Calibration: _____ **Technician(s):** _____

DO wiper changed? Y N **Wiper parks ≈ 180° from optics?** Y N

Turbidity wiper changed? Y N **Wiper parks ≈ 180° from optics?** Y N

Barometric Calculation = [(_____ X 25.4) -98.5] = _____ (value for DO calibration)

_____ / 760 = _____ **(%DO Check ___)** **Battery Voltage:** _____

Parameter	Before Calibration (A)	After Lab Calibration (B)	Post Calibration (after sampling) (C)	Diagnostic Check (record after calibration of parameter)
% D.O.				% DO Check above
D.O. mg/L				ODO Gain: Range: 0.85 to 1.15
Sp.Conductivity mS/cm				Cal Constant: Range: 4.55 to 5.45
pH 7				pH MV Buffer 7: Range: -50 to 50
pH 10				pH MV Buffer 10: Range: -230 to -130
Turbidity (0 NTU)				pH MV Slope: Range: 165 to 180 MV
Turbidity (126 NTU)				

Post Calibration QC Checks

Parameter	QC Equation	QC Check		
D.O. mg/L	B - C =	Answer = ±0.5 mg/L	Y	N
Sp.Conductivity mS/cm	B - C = or {(B / C) -1} x 100 =	Answer = ±0.2 mS/cm or ±10%, whichever is greater	Y	N
pH 7	B - C =	Answer = ±0.5 mg/L	Y	N
pH 10	B - C =	Answer = ±0.5 mg/L	Y	N
Turbidity (0 NTU)	B - C =	Answer = ±1 NTU	Y	N
Turbidity (126 NTU)	B - C = or {(B / C) -1} x 100 =	Answer = ±2 NTU or ±20%, whichever is greater	Y	N

Notes:

Standard Operating Procedure for:

Calibration and Field Measurement Procedures for the SBE 19*plus* SEACAT Profiler

1.0 Introduction

The SBE 19*plus* SEACAT Profiler is a multi-parameter device used to measure conductivity, temperature, and pressure in marine or fresh-water environments at depths up to 7000 meters.

2.0 Purpose

This standard operating procedure (SOP) describes the method to calibrate and use the SBE 19*plus* SEACAT Profiler.

3.0 Method

See the SBE 19*plus* SEACAT Profiler User's Manual for complete instructions for maintenance, care, and use.

4.0 Calibration

Sea-Bird sensors are calibrated by subjecting them to known physical conditions and measuring the sensor responses. Coefficients are then computed, which may be used with appropriate algorithms to obtain engineering units. The conductivity, temperature, and pressure sensors on the SBE 19*plus* are supplied fully calibrated, with coefficients stored in EEPROM in the 19*plus* and printed on their respective Calibration Certificates.

The manufacturer recommends that the SBE19*plus* be returned to Sea-Bird for calibration. The equipment will be returned to the manufacturer yearly for calibration.

5.0 Equipment Needed

- SBE 19*plus*
- Boat with wench
- Lowrance LC X-18 C depth/fish finder
- Sinemaster 1G 2000p generator
- Connector bolt
- Zip ties
- 1 liter of deionized water
- Logbook
- Pen/Pencil

6.0 Setup for Deployment

6.1 Install new batteries or ensure the existing batteries have enough capacity to cover the intended deployment. (see Section 5: Routine Maintenance and Calibration)

6.2 Program the SBE 19*plus* for the intended deployment using SEATERM (see Section 3: Power and Communications Test)

6.2.1 Set date and then time.

6.2.2 Initialize logging to overwrite previous data in memory. Ensure all data has been uploaded to make the entire memory available for recording. If the data is not uploaded, the new data will be stored after the last recorded sample.

6.2.3 Establish the setup and logging parameters.

6.2.3.1. Set up with strain-gauge pressure sensor and 1 voltage sensor.

6.2.3.2. Set up with a 60-second pump turn-on delay after pump enters water, to ensure pump is primed before turning on.

6.2.3.3. Set up to initiate logging with the magnetic switch.

6.2.4 Verify setup with status command.

6.2.5 Send power-off command.

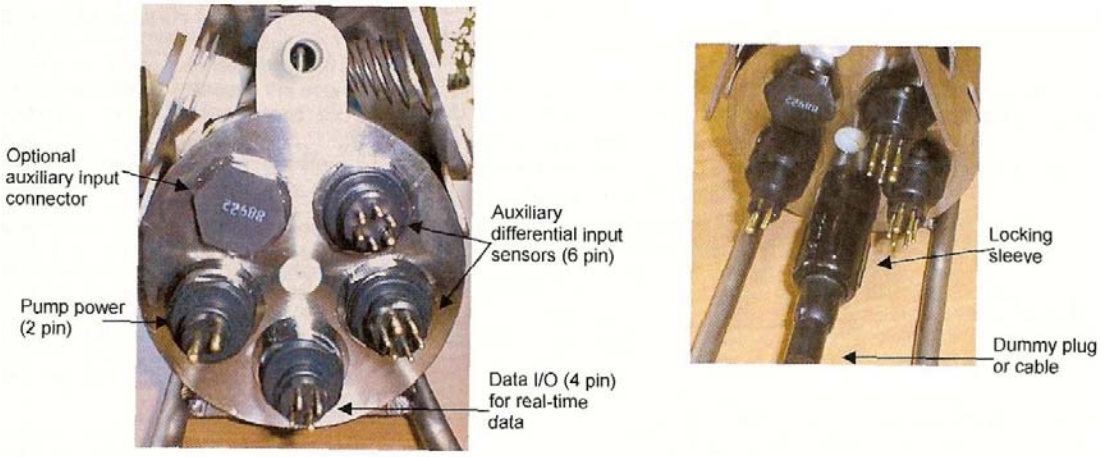
6.2.6 Install a cable or dummy plug for each connector on the 19*plus* sensor end cap:

6.2.6.1. Lightly lubricate the inside of the dummy plug/cable connector with silicone grease (DC-4 or equivalent).

6.2.6.2. Standard Connector- Install the plug/cable connector, aligning the raised bump on the side of the plug/cable connector with the large pin (pin 1-ground) on the 19*plus*. Remove any trapped air by *burping* or gently squeezing the plug/connector near the top and moving your fingers toward the end cap. OR MCBH Connector- Install the plug/cable connector, aligning the pins.

6.2.6.3. Verify that a cable or dummy plug is installed for each connector on the system before deployment.

6.2.6.4. Place the locking sleeve over the plug/cable connector. Tighten the locking sleeve finger tight only. Do not over tighten the locking sleeve and do not use a wrench or pliers.



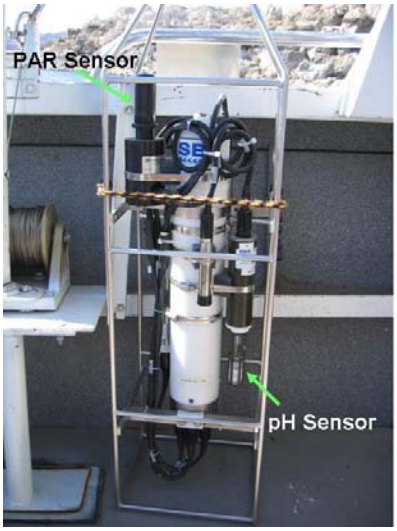
6.2.7 Connect the other end of the cables installed in Section 6.2.6 to the appropriate sensors.

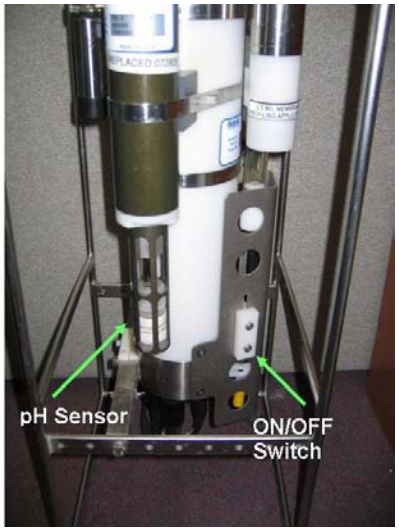
6.2.8 Verify that the hardware and external fittings are secure.

6.2.9 If applicable, remove the Tygon tubing that was looped end-to-end around the conductivity cell for storage. Reconnect the system plumbing (see Users Manual Section 2: Description of SBE19*plus*).

7.0 Use in the Field

7.1 Using the Lowrance LC X-18 C depth/fish finder find the depth of the bottom of the lake. Record this number in the field book. In order not to disturb the substrate, subtract 5m from the depth of the bottom to find the depth at which the deepest WQ reading will be taken from. Record this number in the field book.





7.2 Remove the Photosynthetically Active Radiation (PAR) sensor cover.

7.3 Twist to remove the pH sensor protector. Remove the pH sensor storage bottle/storage solution. Replace pH sensor cover.

7.4 Attach SBE 19*plus* cage to cable (see below).

7.5 Connect wench to the Sinemaster 1G 2000p generator, then start generator.

7.6 Immediately prior to deployment start logging by putting magnetic switch in ON position.

7.7 Put SBE 19*plus* in water, and allow to soak for at least the time required for pump turn-on (one minute) before beginning downcast.

7.8 Press and hold the OUT button on the wench control (see above picture) to lower the SBE 19*plus* through the water column.

7.9 Using the Lowrance LC X-18 C depth/fish finder track the depth of the SBE 19*plus*, when the instrument has reached the depth of 5 m from the bottom, release the OUT button.

7.10 Press and hold the IN button to raise the SBE 19*plus*. When the SBE 19*plus* has reached the surface, lift the instrument back into the boat.
7.11 The cast is complete, stop instrument logging by putting magnetic switch in OFF position.
7.12 Disconnect the SBE 19*plus* cage from the cable and turn generator off.
7.13 Rinse the SBE 19*plus* with fresh water.
7.14 Replace PAR sensor cover.
7.15 Twist to remove pH sensor protector, replace pH storage bottle with solution, and replace pH sensor protector.

8.0 Storage

8.1 Rinse the SBE 19*plus* with fresh water after use and prior to storage.
8.2 If the batteries are exhausted, new batteries must be installed before the data can be extracted. Stored data will not be lost as a result of exhaustion or removal of batteries.
8.3 If immediate redeployment is not required, it is best to leave the 19*plus* with batteries in place and in a quiescent state (QS), the batteries can be left in place without significant loss of capacity.

9.0 Data Recovery

9.1 Upload data in memory, in format SBE Data Processing can use.
9.1.1 See Section 4 of User's Manual for more information about uploading data.
9.2 Send power-off command.

10.0 References

SBE 19plus SEACAT Profiler User's Manual. July 2007. Manual Version #016. Sea-Bird Electronics, Inc. 1808 136th Place NE Bellevue, WA 98005.

Standard Operating Procedure for:

Surface Water Sampling

1.0 Scope and Purpose

This SOP applies to the collection of surface water samples from the lower Truckee River, streams, and non-point source sample sites within the Pyramid Lake Indian Reservation. It includes procedures for collecting samples for delivery to the Pyramid Lake Paiute Tribe's Water Quality Laboratory for analysis of nutrients (i.e., species of nitrogen and phosphorus).

2.0 Sampling Preparation

Before the scheduled sampling event gather sampling equipment and notify laboratory of the sampling event. The following is a list of sampling equipment needed.

Checklist:

- Copy of SOP
- Chain of Custody form in sealed plastic bag
- Field notebook
- Clip board
- Pre-printed sampling sheet
- Pencil
- Ball point pen
- Permanent marker
- Pre-cleaned HDPE sample bottles - 1 Liter
- 500 mL HDPE waste bottle
- Deionized water for field method blank
- Ice chest
- Blue ice (double bagged in ziplock bags)
- Sampling pole

3.0 Sampling Procedures

3.1 Label Sample bottle with the following information.

- Sampling location or name,
- Unique sample number,
- Sample description (e.g., grab, composite),
- Data and time of collection,
- Initials/signature of sampler,
- Analytical parameter(s), and
- Method of preservation.

3.2 Collection of water samples will be conducted prior to or upstream from any other sampling activities that could disturb stream sediments and impact water quality. Try not to disturb the

water upstream of the sampling location. If this does happen, then allow sufficient time and flow to pass for stream to clear itself before collecting a sample.

3.3 Collect representative samples from flowing water, as close to mid-stream as possible. For wider streams, a sampling pole can be used to sampling pole can be used to sample approximately 5 - 10 feet from the shoreline by placing a bottle into the bottle holder of the pole.

3.4 Take care as not to touch the lip of the bottle opening or inside of bottle cap.

3.5 In deeper streams where the 1 Liter sample bottle can be fully submerged 12 inches below the water surface.

3.5.1 Rinse Sample Bottle. Remove the sample bottle cap and submerge bottle below the water surface with the opening facing upstream and tilted slightly up and fill ¼ full. Shake and rinse all internal surfaces. Discard the rinse water downstream while pouring out over the bottle cap. Shake water droplets out of the bottle and cap. Repeat this step once more.

3.5.2 Collect Sample. Submerge the bottle 12 inches below the water surface with the opening facing down, then tilt the bottle up until it is parallel to the flow and hold in position until bottle is filled with water. When bottle is filled tilt bottle opening up and remove from the water. Replace cap on the water bottle.

3.6 In shallow streams where the 1 Liter sample bottle cannot be fully submerged 12 inches below the water surface. Use a smaller bottle, 500 mL, as a 'collection vessel', and then transfer water into the 1 Liter sample bottle.

3.6.1 Rinse 500 mL Sample Bottle. Remove the sample bottle cap and submerge bottle below the water surface with the opening facing upstream and tilted slightly up and fill ¼ full. Shake and rinse all internal surfaces. Discard the rinse water downstream while pouring out over the bottle cap. Shake water droplets out of the bottle and cap. Repeat once more.

3.6.2 Rinse 1 Liter Sample Bottle. Submerge the 500 mL bottle below the water surface with the opening facing down, then tilt the bottle up until it is parallel to the flow and hold in position until bottle is filled with water. When bottle is filled tilt bottle opening up and remove from the water. Pour half of the contents of the 500 mL bottle into the 1 Liter bottle and rinse the all internal surfaces of the larger bottle. Discard the rinse water downstream while pouring out over the bottle cap. Shake water droplets out of the bottle and cap. Repeat with the remaining water in the smaller bottle.

3.6.3 Collect Sample. Submerge the 500 mL bottle mid-depth below the water surface with the opening facing down, then tilt the bottle up until it is parallel to the flow and hold in position until bottle is filled with water. When bottle is filled tilt bottle opening up and remove from the water. Pour the contents of the 500 mL bottle into the 1 Liter bottle and repeat until the larger bottle is filled. Replace cap on the 1 Liter bottle.

3.6.4 Decontaminate 'collection vessel' if it will be used again following the procedures outlined below. If multiple clean bottles were brought to be used as 'collection vessels' then cleaning in the field will not be needed. Return the used bottles to the laboratory to be cleaned.

3.7 QC Sample Collection. All QC samples collected shall be treated in the same manner as regular samples, including labeling and storage.

3.7.1 Collect Field Duplicate Sample. To collect a field duplicate sample, use the same procedure to collect a sample immediately after collecting the regular sample. Field Duplicates should be collected at a rate of one per 10 samples.

3.7.2 Collect Field Blank Sample. Blank sample bottles are rinsed two times with deionized water and then the sample bottle is filled with deionized water.

4.0 Post Sample Collection Activities

4.1 After sample collection store samples in the ice chest. Make sure that the sample lids are tightened and blue ice is double bagged in plastic to decrease the probability of sample contamination. Ensure the ice chest lid is closed to keep samples cool.

4.2 Record sampling information into field logbook. The listed information should be included.

- Sample location and description,
- Sampler's names,
- Date and time of sample collection,
- Designation of sample as composite or grab,
- Type of sampling equipment used,
- Type of field measurement instruments used, along with equipment model and serial number,
- Field measurement instrument readings,
- Field observations and details related to analysis or integrity of samples
- Preliminary sample descriptions
- Sample preservation,
- Lot numbers of the sample containers, sample identification numbers and any explanatory codes, and
- Name of recipient laboratory.

4.3 Fill out Chain of Custody and Test Request Form.

4.4 Decontamination of 'collection vessel'.

4.4.1 Decontamination will occur prior to each use of a piece of equipment and after use at each sampling location. If the equipment will not be used more than once it can be returned to the laboratory for cleaning.

4.4.1.1 Clean 'collection vessel' with non-phosphate detergent and tap water wash. Rinse with tap water until all detergent is removed from equipment. Rinse twice with deionized water.

4.4.1.2 Equipment will be decontaminated in a pre-designated area on plastic sheeting. Cleaned small equipment will be stored in plastic bags.

4.4.1.3 'Collection vessel' can be used again if needed at another site.

5.0 Transport Samples to Laboratory

5.1 Transport samples and paperwork as soon as possible to the laboratory.

6.0 Health and Safety

6.1 Sampling team should consist of at least 2 people for safety reasons, especially if one is wading in the water.

6.2 When wading in streams where water depths may be 1 meter deep or more, wear a life preserver and/or remove hip boots or chest waders. Currents can force wading field workers into deep water and water-filled boots can make swimming difficult.

6.3 When walking through densely vegetated areas along streams, be sure to look for and avoid toxic plants like poison ivy. Be sure to wear appropriate insect repellent and protective clothing for protection from mosquitoes, chiggers, and ticks. In addition, probe areas in your path with a walking stick to warn and disperse poisonous snakes which may inhabit riparian areas.

6.4 Field staff should protect themselves from water borne illness by wearing disposable gloves, and avoid touching eyes, nose and mouth. Be sure to clean up with bacteria disinfectant soap and water after wading in streams. This is particularly important for streams that drain livestock areas, sewage treatment plant effluents, and other obvious pollution sources. Under no circumstances should you drink the water from any stream.

7.0 References

Draft Standard Operating Procedures For Surface Water Sample Collection. Lahontan Regional Water Quality Control Board. July 2001.

Field Sampling Guidance Document #1225, Revision 1. US EPA Region 9 Laboratory. September 1999.

Standard Operating Procedure for: Water Sample Collection, Revision 2. Missouri State University and Ozarks Environmental and Water Resources Institute. January 2007.

Standard Operating Procedure for:

Discrete Depth Water Sampling

1.0 Scope and Purpose

This SOP applies to the collection of surface water samples from Pyramid Lake. It includes procedures for collecting samples for delivery to the Pyramid Lake Paiute Tribe's Water Quality Laboratory for analysis of nutrients (i.e., species of nitrogen and phosphorus).

2.0 Sampling Preparation

Before the scheduled sampling event gather sampling equipment and notify laboratory of the sampling event. The following is a list of sampling equipment needed.

Checklist:

- Copy of SOP
- Chain of Custody form in sealed plastic bag
- Field notebook
- Pencil
- Ball point pen
- Permanent marker
- Pre-cleaned HDPE sample bottles - 1 Liter
- Van Dorn sampling device
- Weighted messenger
- Down rigger with cable
- Lowrance LC X-18 C depth/fish finder
- 5-Gallon Bucket (designated for lake sampling)
- Deionized water for field method blank
- Ice chest
- Blue ice (double bagged in ziplock bags)

3.0 Sampling Procedures

3.1 Collection of water samples will be conducted monthly at Station 96 and quarterly at both Station 96 and Station 93.

3.2 Label Sample bottle with the following information.

- Sampling location or name,
- Unique sample number,
- Sample description (e.g., grab, composite),
- Data and time of collection,
- Initials/signature of sampler,
- Analytical parameter(s), and
- Method of preservation.

3.3 Using the Lowrance LC X-18 C depth/fish finder, determine the approximate depth of the water. The “bottom” sample will be taken 5 m from the bottom of the lake, to avoid bottom disturbance.

3.4 Attach the Van Dorn sampling device to the downrigger cable.

3.5 Set the sampling device so that the sampling end pieces are pulled away from the sampling tube allowing the water to be sampled to pass through this tube (above left).

3.6 Using the downrigger (above right), lower the pre-set Van Dorn sampling device to the predetermined depth, using the Lowrance LC X-18 C depth/fish finder to ensure the sampling device is lowered to the correct depth.

3.6.1 Station 96 Sampling Depths:

- Composite (0 m, 2.5 m, 5 m)
- 10 m
- 20 m
- 30 m
- 45 m
- 60 m
- 75 m
- 90 m
- Bottom (5 m from bottom)

3.6.2 Station 93 Sampling Depths:

- Composite (0 m, 2.5 m, and 5 m)
- 10 m
- 20 m
- 30 m
- 45 m
- 60 m
- Bottom (5 m from bottom)

3.7 When the sampling device is at the required depth send down the messenger to close the sampling device.

3.8 Retrieve the sampling device.

3.9 Discharge the first 10 to 20 mL to clear any potential contamination on the valve.

3.10 Rinse sample bottle. Remove cap of sample bottle, discharge 50 mL of the sample into the sample bottle. Shake and rinse all internal surfaces. Discard the rinse water overboard while pouring out over the inside of the bottle cap. Shake water droplets out of the bottle and cap. Repeat this step once more.

3.11 Collect sample. Discharge 1 liter of the sample into the sample bottle. (below left) Once bottle is full replace cap on sample bottle. Take care as not to touch the lip of the bottle opening or inside of bottle cap while sampling.

3.12 Collecting the composite and field split sample.

3.12.1 Rinse the composite bucket with lake water twice before sampling.

3.12.2 Collect samples from the predetermined depths using the description in steps 3.4 – 3.8. Discharge all three entire samples into a 5-gallon bucket. (above right)

3.12.3 Rinse sample bottle. Remove the sample bottle cap and submerge bottle below the water surface with the opening tilted slightly up and fill 1/5 full. Shake and rinse all internal surfaces. Discard the rinse water overboard while pouring out over the inside of the bottle cap. Shake water droplets out of the bottle and cap. Repeat this step once more.

3.12.4 Collect Composite Sample. Submerge the bottle below the water surface with the opening facing sideways, then tilt the bottle up and hold in position until bottle is filled with water. When bottle is filled tilt bottle opening up and remove from the water. Replace cap on the water bottle.

3.12.5 Collect Field Split Sample. Follow the directions in step 3.12.4 to collect the field split sample.

3.12.6 Discard the remaining water in the bucket overboard.

3.13 QC Sample Collection. All QC samples collected shall be treated in the same manner as regular samples, including labeling and storage.

3.13.1 Collect Field Duplicate Sample. To collect a field duplicate sample, use the same procedure to collect a sample immediately after collecting the regular sample. Field Duplicates should be collected at a rate of one per 10 samples.

3.13.2 Collect Field Blank Sample. Blank sample bottles are rinsed two times with deionized water and then the sample bottle is filled with deionized water.

4.0 Post Sample Collection Activities

4.1 After sample collection store samples in the ice chest. Make sure that the sample lids are tightened and blue ice is double bagged in plastic to decrease the probability of sample contamination. Ensure the ice chest lid is closed to keep samples cool.

4.2 Record sampling information into field logbook. The listed information should be included.

- Sample location and description,
- Sampler's names,
- Date and time of sample collection,
- Designation of sample as composite or grab,
- Type of sampling equipment used,
- Type of field measurement instruments used, along with equipment model and serial number,
- Field observations and details related to analysis or integrity of samples
- Preliminary sample descriptions
- Sample preservation,
- Lot numbers of the sample containers, sample identification numbers and any explanatory codes, and
- Name of recipient laboratory.

4.3 Fill out Chain of Custody and Test Request Form.

5.0 Transport Samples to Laboratory

5.1 Transport samples and paperwork as soon as possible to the laboratory.

6.0 Health and Safety

6.1 When on the boat make sure there are an adequate number of life preservers.
6.2 Only properly trained individuals are to handle boat operations.
6.3 Floors on the boat may become slippery, use caution when moving on the boat.
6.4 Use caution when leaning overboard to set or retrieve sampling equipment.

7.0 References

Field Sampling Guidance Document #1225, Revision 1. US EPA Region 9 Laboratory. September 1999.

Standard Operating Procedure for:

Secchi Disk Measurements

1.0 Scope and Purpose

This SOP applies to the collection of water clarity measurements using a Secchi disk.
The Secchi disk is a round, flat disk painted with alternating black and white quadrants and is one of the most widely used tools for water quality monitoring. A secchi disk reading can provide information about water clarity from which characteristics such as turbidity and productivity can be inferred. Secchi depth changes over the course of seasons within an individual lake with algal blooms, storm turbulence, and seasonal plankton fluctuations. The more phytoplankton or suspended sediment in a lake, the lower the Secchi disk reading will be.

2.0 Sampling Preparation

Before the scheduled sampling event gather the following sampling equipment

Checklist:

- Copy of SOP
- Field notebook
- Pencil
- Ball point pen
- Secchi disk on a marked line; 0.5 m increments marked

3.0 Sampling Procedures

3.1 Lower the disk into the water on the shaded side of the boat, while keeping a firm grip on the line.

3.2 Keep lowering the disk until it is no longer visible; determine the depth of the Secchi disk using the line marks and record in the field notebook.

3.3 Record sampling information into field logbook. The listed information should be included.

- Sample location and description
- Sampler's names
- Date and time of sample collection
- Secchi disk depth readings
- Field observations and details related to analysis or integrity of samples

4.0 References

Lake Monitoring Field Manual. Meredith Becker Nevers and Richard L. Whitman: U.S. Geological Survey. Lake Michigan Ecological Research Station 1100 N. Mineral Springs Rd. Porter, IN 46304.

Standard Operating Procedure for:

Zooplankton Sampling

1.0 Scope and Purpose

This SOP applies to the collection of zooplankton samples from Pyramid Lake. It includes procedures for collecting samples for delivery to the Pyramid Lake Paiute Tribe's Water Quality Laboratory for analysis.

2.0 Sampling Preparation

Before the scheduled sampling event gather sampling equipment and notify laboratory of the sampling event. The following is a list of sampling equipment needed.

Checklist:

- Copy of SOP
- Field notebook
- Pencil
- Ball point pen
- Permanent marker
- Pre-cleaned HDPE sample bottles – 500 mL
- Wisconsin sampler
- Down rigger with cable
- Lowrance LC X-18 C depth/fish finder
- Squirt bottle
- Ice chest
- Blue ice (double bagged in ziplock bags)

3.0 Sampling Procedures

3.1 Collection of zooplankton samples will be conducted monthly at Station 96 and quarterly at both Station 96 and Station 93.

3.2 Label sample bottles with the following information.

- Sampling location or name,
- Unique sample number,
- Sample description (e.g., grab, composite),
- Data and time of collection,
- Initials/signature of sampler, and
- Method of preservation.

3.3 Collect 100 m composite sample.

3.3.1 Using the Lowrance LC X-18 C depth/fish finder, determine the approximate depth of the water. The "bottom" sample will be taken 5 m from the bottom of the lake, to avoid bottom disturbance.

3.3.2 Attach the Wisconsin sampling device (below left) to the downrigger cable (below right).

3.3.3 Clamp the tube on the bottom of the Wisconsin sampler to stop sample loss.
3.3.4 Using the downrigger (above right), lower the sampling device to the predetermined "bottom" depth, using the Lowrance LC X-18 C depth/fish finder to ensure the sampling device is lowered to the correct depth.
3.3.5 When the sampling device is at the required depth raise the sampling device to the surface.
3.3.6 Retrieve the sampling device.
3.3.7 Remove sample bottle cap. Situate tube on the bottom of the sampling device in the sample bottle and release the clamp, emptying the sample into the sample bottle. Using the squirt bottle rinse the Wisconsin sampler cup sides to ensure entire sample collected is transferred to the sample bottle. Replace sample bottle cap.
3.3.8 Store the sample bottle in the cooler for transport to the PLPT WQ Laboratory.

3.4 Collect 10 m composite sample.
3.4.1 Attach the Wisconsin sampling device (below left) to the downrigger cable (below right).
3.4.2 Clamp the tube on the bottom of the Wisconsin sampler to stop sample loss.
3.4.3 Using the downrigger (above right), lower the sampling device to 10 m, using the Lowrance LC X-18 C depth/fish finder to ensure the sampling device is lowered to the correct depth.
3.4.4 When the sampling device is at the required depth raise the sampling device to the surface.
3.4.5 Retrieve the sampling device.
3.4.6 Remove sample bottle cap. Situate tube on the bottom of the sampling device in the sample bottle and release the clamp, emptying the sample into the sample bottle. Using the squirt bottle rinse the Wisconsin sampler cup sides to ensure entire sample collected is transferred to the sample bottle. Replace sample bottle cap.
3.4.7 Repeat steps 3.4.2 – 3.4.6 two more times.
3.4.8 Store the sample bottle in the cooler for transport to the PLPT WQ Laboratory.

4.0 Post Sample Collection Activities

4.1 After sample collection store samples in the ice chest. Make sure that the sample lids are tightened and blue ice is double bagged in plastic to decrease the probability of sample contamination. Ensure the ice chest lid is closed to keep samples cool.

4.2 Record sampling information into field logbook. The listed information should be included.

- Sample location and description,
- Sampler's names,
- Date and time of sample collection,
- Designation of sample as composite or grab,
- Type of sampling equipment used,
- Type of field measurement instruments used, along with equipment model and serial number,
- Field observations and details related to analysis or integrity of samples
- Preliminary sample descriptions
- Sample preservation,
- Lot numbers of the sample containers, sample identification numbers and any explanatory codes, and
- Name of recipient laboratory.

4.3 Fill out Chain of Custody and Test Request Form.

5.0 Transport Samples to Laboratory

5.1 Transport samples and paperwork as soon as possible to the laboratory.

6.0 Health and Safety

6.1 When on the boat make sure there are an adequate number of life preservers.
6.2 Only properly trained individuals are to handle boat operations.
6.3 Floors on the boat may become slippery, use caution when moving on the boat.
6.4 Use caution when leaning overboard to set or retrieve sampling equipment.

7.0 References

Field Sampling Guidance Document #1225, Revision 1. US EPA Region 9 Laboratory. September 1999.

Standard Operating Procedure for:

Chain of Custody Practices and Form Completion

1.0 Introduction/Purpose

The following procedures are to be used by all PLPT/PLF staff who are participating in water quality sampling to ensure accountability for and documentation of sample integrity from the time all samples are collected until receipt by the receiving laboratory. These procedures are intended to document each stage of the sample's life cycle (i.e., collection, transport, and delivery).

2.0 Definitions

Custody - Samples and data are considered to be in your custody when:

- They are in your physical possession;
- They are in your view, after being in your physical possession;
- They are in your physical possession and then locked up so that tampering cannot occur;
- They are kept in a secured area with access restricted to authorized personnel only.

Sample - A portion of the environmental or source matrix that is collected and used to characterize that matrix.

Sample Custodian - The person possessing custody of the sample.

Chain of Custody - A process whereby a sample is maintained under physical possession or control. Chain of custody procedures are one piece of a large quality assurance program to assure data and conclusions are defensible in a legal or regulatory situation.

Chain of Custody and Test Request Form - The laboratory provides the form used to record sample collection information, test(s) requested, and sample custody.

Sample Set - Collection of samples collected during one sampling event.

3.0 Sample Collection

3.1 Sampling. Samples are routinely collected by PLPT/PLF employees using standard collection procedures defined by specific Standard Operating Procedures.

3.2 Custody Assignment. The sampler shall ensure proper collection, preservation and labeling of the sample. The sampler will also initiate the chain of custody documentation process, prepare sample submission information, and store samples for transport to the laboratory. Since as few people as possible should handle samples, the sampler is responsible for the initial custody of the sample.

3.3 Sample Identification. To ensure samples are traceable, samples shall be clearly labeled immediately upon collection. Labeling information may vary by SOPs, but labels must be written legibly, using a ballpoint (indelible) pen, unique for the sample/case and firmly fixed to the sample. The sample shall contain the unique sample number or identification, sample type, name of sampler, preservation method, and data and time of collection.

4.0 Sampling Documentation

4.1 Field Logbooks. In any sampling effort, there are field information and measurements that need to be recorded. This information shall be retained in a sampler's field log. Examples of information entered include: purpose of sampling, type of sample, address, sample composition, description of sampling point, sampling method, date and time of collection, sample identification number, field data, and preservation method. This record may be considered evidence and part of the larger aspect of data defensibility. Logbooks shall be kept in a safe place.

4.2 Chain of Custody Records. Chain of custody shall be documented on the form Chain of Custody and Test Request Form. Chain of custody forms shall be completed by the sampler at the time of sample collection and shall be submitted with each sample set. The sampler shall print their name, sign, and date the form. The completed form shall be signed by the sampler and dated (chain of custody block) and placed in a waterproof carrier (e.g., zip-lock bag) if it is a water sample. The form shall be packaged with the sample for transport to the laboratory. The original COC form, all results, and documentation are maintained by PLPT WQ Laboratory in a case file.

5.0 Chain of Custody and Test Request Form

With each sample set submitted to the laboratory for analysis, the sampler shall include the Chain of Custody and Test Request Form, see figure below, with the following information indicated on the form.

- Project area. If 'Other' then provide laboratory with information about the water body, sample sites, and any field measurements collected.
- Analyses requested.
- Date sample collected.
- Time sample collected.
- Sample identification number/name.
- Sample description.
- Remarks.
- Sampler's name, signature and date.
- Additional instructions for laboratory.
- Changes in custody indicated by signatures.

6.0 Sample Packaging, Transport and Transfer of Custody

6.1 Sample packaging. The correct preparation and preservation of samples for transport are critical to ensure sample integrity.

6.1.1 The sampler should contact the laboratory if unsure of any aspect of sample collection, preservation, packaging, and transport.

6.1.2 Samples must be labeled, tightly sealed in the appropriate container.

6.1.3 The original Chain of Custody and Test Request form and any other documentation are to be sealed tightly in a plastic zip-lock bag.

6.1.4 If not immediately delivered to the laboratory by the sampler, containers shall either be locked with a personal padlock, or sealed with a custody seal, or stored in a secure area.

6.2 Sample Transport. Samples are to be delivered to the laboratory by the sampler. If for some reason the sampler cannot transport the samples to the laboratory, the chain of custody must

be documented on the Chain of Custody and Test Request Form and the new sample custodian must transport the samples to the laboratory. Samples are to be delivered to the PLPT WQ Laboratory as soon as possible after collection. The sample shall remain in the Sample Custodian's possession or sight at all times.

7.0 References

Chain of Custody Policy and Procedures. Guidance Memo No. 00-2016, Amendment #1. Commonwealth of Virginia, Department of Environmental Quality. March 14, 2006.

Quality Assurance Project Plan Development Tool, Version 1.1. US EPA Region 1 and Region 9. December 2005.

Chain of Custody and Test Request Form

Pyramid Lake Paiute Tribe Water Quality Laboratory
Chain of Custody and Test Request Form

Project: ☐ PL-ST 96 ☐ PL-ST 93 ☐ LTR/NPS ☐ RBA ☐ Streams ☐ NPS ☐ Wetlands ☐ Other[1]

Test(s) Requested: ☐ All ☐ Ortho-Phosphate ☐ Total Phosphate ☐ Nitrate+Nitrite ☐ Ammonia-N ☐ Total Kjeldahl Nitrogen

Date Sampled	Time Sampled	Sample ID	Sample Description	Remarks

Sampled By:

Print Name Signature Date

Additional Instructions:

Signature	Print Name	Date	Time
Relinquished by:			
Received by:			
Relinquished by:			
Received by:			
Relinquished by:			
Received by:			

[1]Sampler to provide information about water body, sample sites and any field measurements collected.

Only listed tests are offered at the PLPT WQ Laboratory at this time. Water samples are only received from departments within the PLPT organization. All sample bottles provided by the PLPT WQ Laboratory are 1 Liter Polyethylene bottles. Sample preservation is completed at the laboratory.

Appendix B: Workplace Safety Program

Pyramid Lake Paiute Tribal Council
Post Office Box 256
Nixon, Nevada 89424
Telephone: (702) 574-1000 / 574-1001 / 574-1002
FAX (702) 574-1008

WORKPLACE SAFETY PROGRAM

OCTOBER 06, 1995

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POLICY STATEMENT

It is the policy of the Pyramid Lake Paiute Tribe that the first consideration in the performance of work shall be the safety of employees.

All reasonable methods, procedures and equipment necessary to achieve this will be used.

There will be no compromise with safety.

Tribal Administration will implement this policy by:

1.) Providing all employees with all necessary equipment to perform their work safely;

2.) Providing only safe tools and equipment;

3.) Providing instruction to all employees in safe work performance;

4.) Providing on-going safety instructions; and

5.) Continually investigating improved safety methods, concepts and techniques.

This policy requires all employees to:

1.) Be vigilant within their work area and at all job sites for potential hazards (i.e., unsafe practices, unsafe equipment, etc.);

2.) Wear and use required safety equipment;

3.) Maintain work habits and attitudes that will protect other employees and themselves.

Signed,

Norman Harry

Norman Harry, Chairman
PYRAMID LAKE PAIUTE TRIBE

Gina Wadsworth

Gina Wadsworth, Secretary
PYRAMID LAKE PAIUTE TRIBE

PYRAMID LAKE PAIUTE TRIBE
SAFETY RESPONSIBILITIES

The Workplace Safety Program at the Pyramid Lake Paiute Tribe, is designed to increase the efforts to achieving safe and healthful working conditions. All directors, supervisors and employees are expected to familiarize themselves with the requirements of our Workplace Safety Program and contribute to ensure the success of the program.

While all members of management and all employees are ultimately responsible for safety and health in the workplace, there is one person directly responsible for coordination of all safety issues at the Tribe:

SAFETY DIRECTOR: _____ Phone: _____

Some of the responsibilities of the Safety Director include:

* Setting policy and assigning responsibility and accountability to individuals.
* Reviewing and evaluating results.
* Providing active leadership by participation, example, and a demonstrated interest in the program.
* Maintaining up-to-date information on local, state and federal standards.
* Maintaining all required safety posters.
* Planning, organizing and coordinating safety and health training.
* Ensuring that policies for the medical treatment of injured employees are followed; this includes first aid equipment, designated trained first aid providers, and procedures to provide additional medical treatment.

TRIBAL CHAIRMAN: _____ Phone: (702) 574-1000

* The Tribal Chairman is responsible for ensuring that all safety policies and procedures are followed by everyone under his/her jurisdiction.

SUPERVISOR: _____ PHONE: _____

All supervisors are expected to take a direct interest in workplace safety. Some of the responsibilities all supervisors have include:

* Keeping informed about safety and health regulations affecting the operations they supervise.

* Making sure that each employee is able to complete each task to which he/she is assigned in a safe manner.

* Making sure that machines and equipment are maintained in a safe operating condition.

* Making sure that employees follow all safety and health regulations and work practices, including using required personal protective equipment.

* Investigating accidents that may occur and identifying the corrective action necessary to prevent a similar accident from occurring.

* Reporting without delay, any unsafe or unhealthful conditions which they cannot correct themselves to management.

* Assisting employees to complete a NOTICE OF INJURY OR OCCUPATIONAL DISEASE form in the event of accidents.

SAFETY COMMITTEE:

The Safety Committee is an important part of our Workplace Safety Program. It is through the Safety Committee that vital safety issues and possible solutions can be brought to the attention of top management.

The Safety Committee will consist of one employee from each department. Each year in the month of December, volunteers from the work force who are willing to serve on the committee will be chosen by employees. Because service on the committee is an important opportunity for employee development, employee members may not serve consecutive terms. To ensure continuity, half of the employee members of the committee will initially serve a one year term, and the other half will serve a two year term. Thereafter, half of the committee positions will be filled each year for a two-year term.

In addition to the members chosen by employees, the Tribe will appoint one individual to serve as a liaison with management. However, this individual will not be a voting member of the committee and will not have the power to reject committee actions.

The safety committee will meet once each month on the company premises. Time spent in committee meetings will be recorded as time worked. The committee shall select a chairperson whose role is to lead the discussion and coordinate the committee's efforts. The Committee shall also select a secretary who will keep accurate minutes of each meeting and maintain records (such records must be maintained for at least three years).

DUTIES OF THE COMMITTEE

Members of the safety committee are responsible to:

* Participate in monthly safety inspections.

* Create and maintain an active interest in safety and health among employees.

* Review and analyze accidents involving injuries or illness with an eye to suggest how such situations could be avoided.

* Review the potential safety or health hazards of all new processes, methods, or materials introduced into the workplace.

* Inform management of safety hazards which are observed by or reported to the Committee.

* Ensuring that accident reporting and investigation procedures are followed.

* Plan educational programs on safety.

* Serve as a conduit for communication between employees and management on safety issues.

It is <u>not</u> the role of the safety committee to make specific proposals to management or negotiate over safety issues or the abatement of hazards. Further, the committee is not authorized to deal with management on any issues relating to wages, hours or working conditions.

SAFETY TRAINING

As part of our Workplace Safety Program, the Tribe will provide several different types of safety training for all our employees:

* A general safety orientation for all new employees.

* Specific training on how to safely perform their assigned job.

* Special training when they work with hazardous materials or complex types of machinery or other equipment.

* Training on how to use any personal protective equipment provided (e.g. respirators, hearing protections, etc.)

These requirements are based upon the assumption that when employees know how to do their jobs properly, and know the hazards of the job, they will work safely.

Safety education and training for employees should commence at the time of employment. Before people actually begin an assigned task, the Human Resources department must assure that employees are provided the following instruction in New Employee Orientation:

* An explanation of the Tribe's safety policy.

* Familiarization with the SAFETY RULES of the company and enforcement policies.

* The requirement for immediately reporting all injuries and completing a NOTICE OF INJURY OR OCCUPATIONAL DISEASE form.

* The necessity and manner for reporting all unsafe conditions to their supervisor (SAFETY ACTION REQUEST FORMS are included).

* A clear statement that no employee should attempt to do a job or work with equipment that appears to be unsafe.

* A copy of the pamphlet "WORKPLACE SAFETY: Your Rights and Responsibilities" (each new employee must complete a statement attesting that they have read and understand their rights and responsibilities; a copy of this statement must be maintained in each employee's personnel file).

* A listing of the names and addresses of all medical providers under contract to treat employees injured on the job.

After a person is assigned to a job, the responsibility for safety training passes to the immediate supervisor. The supervisor must discuss the following with each employee:

* The safety rules of the department in which the employee will work.

* The correct job procedure for the employee to follow in any particular job; it also should be emphasized that doing the job incorrectly may hurt people or damage equipment or supplies.

* How to use any personal protective equipment required.

* All information pertinent to the area regarding hazardous materials (see the section on "HAZARD COMMUNICATION".

This initial training is to be documented on the EMPLOYEE SAFETY ORIENTATION FORM and signed by the employee and returned to Human Resources to be retained in his/her personnel file.

It is most desirable to follow this initial instruction with a complete review within a week or two after assignment to the job. This will assure that the new employee fully understands the information given at the time of employment and at the time of assignment to the job.

The supervisor will also provide additional training whenever the employee's job changes, new hazardous materials are introduced, new machines/tools are used, new safety protection equipment is needed, or there are incidents of recurring injuries, and retrain

any employees that do not seem to understand proper safety procedures. The SAFETY TRAINING DOCUMENTATION for must be used to document any of these types of training.

Whenever personnel are used from a temporary employment service, the supervisor must provide them specialized training for the jobs they will be performing before they actually begin work or as soon as possible thereafter. This training also needs to be recorded with a SAFETY TRAINING DOCUMENTATION form.

Effective safety training will result in the following benefits for the Tribe:

* Reduction in injuries.
* Reduction in damage in property and supplies.
* Reduction in retraining time.
* Reduction in Tribal liability.
* Increase in asking for help when it is needed.
* Increase in morale.
* Increase in productivity.
* Increase in profits.
* Decrease in absenteeism.

SAFETY RULES AND DISCIPLINE

Federal and state law requires the Tribe to have "a method for ensuring that employees comply with safe rules and work practices." Safety rules are a basic part of our Workplace Safety Program and are also part of our disciplinary procedures. However, the point of having safety rules is not to discipline, but rather to ensure a safe working environment. All employees need to know and follow these Safety Rules and all supervisory and management personnel must enforce them.

The task of our management team is to change unsafe behavior by:

* Recognizing (rewarding) safe performance:

It is important that supervisors and directors recognize correct job performance and compliment employees for it. Putting a citation in writing and making it part of an employee's personnel record is but one means of recognizing safety excellence.

* Correcting (disciplining if necessary) unsafe behavior:

Unsafe job procedures will not be tolerated. Any member of management observing unsafe job behavior is expected to bring it to the attention of the employee's supervisor. Supervisors are expected to take steps immediately to correct the behavior.

The following safety rules are representative:

1. Employees must immediately report all accidents, injuries, unsafe and/or unhealthful conditions in the workplace, including defective tools or other equipment, to their supervisor.

2. Established safe job procedures must be followed by all employees. Changes in regular job procedures require the approval of the immediate supervisor.

3. Employees who are unsure on how to operate a machine or perform any assigned task that they are required to do must ask their supervisor before proceeding.

4. Employees must not tamper with, remove, destroy or otherwise interfere with the use of any safety device or safeguard provided for protection of employees or customers.

5. Personal protective equipment must be worn or used in any area where it is required.

6. Employees must use only the proper tool for the job. If the proper tool is not available, contact the supervisor.

7. Employees are required to get help in lifting any item which is so bulky, awkward or heavy that they feel they cannot lift it safely.

8. If a repetitive task causes employees discomfort, or they feel it is unsafe or unhealthful, they must report this condition to their supervisor immediately.

9. Safe attire for employees working in all production areas is a must: (i.e., steel toe boots, no high heels, etc.) Hair longer than shoulder level must be tied up and/or hair nets worn. No loose fitting sleeves or loose jewelry. No shorts, dresses or skirts. No short blouses or shirts that do not reach the waist band.

10. Horse play, including fighting and throwing articles, as well as running is forbidden.

11. If an employee is suspected of using alcohol and other drugs in the workplace, this will be investigated and, if necessary, disciplinary measures will be taken.

The four-step procedure outlined below would normally be followed for violations of safety rules. However, each case may be

considered in light of a number of factors (e.g. employee's length of service, seriousness of the incident, other mitigating factors). The company reserves the right to invoke any level of discipline at any point and, in fact, an employee may be terminated for the first instance of a serious safety violation.

1. The first time an infraction is noted, the supervisor will discuss the behavior with the employee and make sure the employee understands the nature of the violation and the consequences that will result if there is a repeat violation. This verbal warning will be noted in the employee's personnel file.

2. The second violation will generate a written warning. A copy will be placed in the employee's personnel file.

3. The third violation results in the employee being placed on an unpaid disciplinary suspension (leave without pay). This action will be reviewed first with the Human Resources department.

4. A fourth safety violation will result in the employee being terminated. This action will be reviewed first with the Human Resources department.

These steps are not necessarily followed if the safety violation is a serious hazard either to the employee or to the Tribe. In the case of a serious safety violation, the Tribe may elect to immediately proceed to a higher step.

IDENTIFYING AND EVALUATING WORKPLACE RISKS

In order to comply with state and federal regulations, the tribe will conduct regular and frequent inspections of the workplace. To maintain a safe and healthful workplace the Tribe needs to have ways to identify hazards in the workplace.

Inspection of the workplace is our primary tool to identify unsafe conditions and practices. All supervisors are responsible for routinely inspecting their area(s) of responsibility. Supervisors need to document their inspections of the workplace by using the SUPERVISOR'S DAILY SAFETY INSPECTION FORM (attached). This form must be completed on a daily basis and be forwarded to the Safety Director.

Supervisors must constantly check for:

* Violations of safety rules.

* Machinery or other equipment without the necessary guards.

Unsafe use or storage of chemicals, including flammables.

* Obvious violations of good housekeeping practices.

* Personal protective equipment not being used where required or being used improperly.

* Machinery, hand tools or other equipment in poor condition or being used improperly.

* Areas where there have been recurring injuries.

* Any other deviation from accepted safe practices.

In addition to on-going, day-to-day vigilance, the Safety Committee will conduct an inspection of the workplace prior to it's monthly safety committee meeting. The purpose of this inspection is to have a team of management and employees inspect the workplace on a regular and frequent basis, looking for both unsafe physical conditions as well as unsafe job practices or acts of employees. The inspection findings are to be documented on the SAFETY COMMITTEE'S MONTHLY SAFETY INSPECTION REPORT. A copy of this report is to be attached to the copy of the SAFETY COMMITTEE MINUTES.

SAFETY COMMUNICATION

Unless a system for communicating safety down and up throughout the organization is established, employees may not understand management's policies and intentions, and management may not get unfiltered communication from the employees about safety hazards or their needs.

We have established a safety suggestion box to make it easy for any employee to report a safety hazard or make a suggestion to improve safety and health conditions in the workplace. While it is true that supervisors traditionally act as the link between employees and management, sometimes employees have difficulty expressing a safety need to their immediate supervisor. The Tribe expects supervisors to encourage and welcome suggestions, but we have provided the safety suggestion box as an alternate route for employees to communicate safety matters to management. Employees may submit their safety suggestions on a SAFETY ACTION REQUEST FORM.

The Safety Committee Chairperson is responsible for maintaining the safety suggestion box and reviewing the contents with the committee each month. However, urgent items are to be acted upon immediately, action taken, and then reviewed at the next Safety Committee meeting.

The Safety Committee will respond -- in writing -- to the person making the suggestion, indicating what action has been taken.

Supervisors need to bring to the attention of employee's all types of safety communication including the safety suggestion box, safety memos, safety posters, safety banners, paycheck inserts,

articles in the Tribal newsletter, etc.

HAZARDOUS MATERIALS

The Hazard Communication Standard (29 CFR section 1910) is a regulation established by OSHA and adopted by the Nevada Division of Occupational Safety & Health. The standard requires that every business using hazardous chemicals establish a comprehensive hazard communication program which includes these four basic elements:

1. A written program that details the company's plan;

2. An inventory of hazardous materials in the workplace, with a Material Safety Data Sheet for each material, readily available to employees;

3. Labeling of all containers of hazardous materials in the workplace; and

4. An employee training program, actively informing employees of potential hazards associated with these materials and the precautions they should take when using these materials.

All hazardous chemical containers in work areas shall be labeled with either the producer's original label or an in-house chemical hazard identification label. Labels will include the name of the hazardous substances in the containers, and hazard warnings listing physical and acute/chronic health hazards.

In addition to the labels, the company will maintain for each hazardous substance a MATERIAL SAFETY DATA SHEET (MSDS) which is a detailed information bulletin prepared by the chemical producer. The MSDS describes the physical and chemical properties, physical and health hazards, routes of exposure, precautions for safe handling and use, emergency and first-aid procedures, and control measurers. The information provided on the MSDS assists us in selecting safe chemicals and helps us to respond effectively and safely to daily use situations.

The Safety Director will maintain binders of MSDS's in the Human Resources department. Additional MSDS's are available to supervisors at their work stations. Purchasing also is responsible for maintaining binders of MSDS's. These binders will be updated as new chemicals are introduced to the individual work areas. All employees are to have access to these binders at all times.

On an annual basis, a complete chemical inventory will be performed to ensure that MSDS's have been obtained for all hazardous chemicals used. This inventory list will be available for inspection by employees, auditors and appropriate regulatory agencies.

The Purchasing department will be responsible for requesting an

MSDS for any new chemical purchase. Other employees, such as department heads requesting a chemical from a vendor even on a trial basis, must ensure that an MSDS is received prior to or with the chemical shipment. Departments shipping or receiving are authorized to deny the acceptance of any chemical into their facility for which an MSDS has not been received.

Everyone who works with, or is potentially exposed to, hazardous chemicals will receive initial training on the safe use of those hazardous chemicals by the supervisor of the department where chemicals are used. Training will focus on the types and hazards of the chemicals with which employees work and to which they may be exposed during their daily activities. Employees should be trained on the following:

* Operations in their work areas in which hazardous materials are used.
* The name and physical characteristics (color, odor, gas, liquid, etc.) of these chemicals.
* Methods used to detect the presence or release of chemicals in the workplace.
* Any potential health hazards associated with the use of the chemicals including signs and symptoms of exposure to chemicals.
* How to read and understand the Material Safety Data Sheets and the location and availability of MSDS's.
* Information provided by labels and details of the labeling system.
* Proper handling of chemicals to protect from the hazards, including appropriate work practices, personal protective equipment and emergency procedures and equipment (eye wash, showers, etc.).
* What to do in the event of spills or leaks of hazardous materials in the work area.

Employees must also be trained in the hazards associated with non-routine activities. This includes tasks performed infrequently or activities employees have not performed previously. Examples include tank cleaning, chemical spill clean-up and gas cylinder changing. Whenever a new hazard is introduced, additional training will be provided.

EMERGENCY SITUATIONS

In case of an emergency, dial 911 and be prepared to give:

1. Your name and your Pyramid Lake Paiute Tribal department's name.

2. Phone number and extension.

3. Nature of the emergency and the assistance you require (Fire Department, Paramedics, Police, etc.)

4. Location of the incident/injury including the building address, which part of the building and nearby cross streets.

5. Prepare to have someone meet emergency vehicle in front of the building or facility.

6. During business hours, call the Human Resources department and give the information about the emergency to them. In the event of an injury/illness they will gather the employee's medical records, insurance information and emergency contact.

7. If the event occurs during the hours that the Human Resources department is closed, you should carefully document the event and give it to Human Resources as soon as possible. If the situation is serious, you should call: Payroll at 574-1000, ext. 111.

EMERGENCY EVACUATION

Supervisors and their employees must become familiar with the evacuation routes for their work areas. Part of the supervisor's orientation with new employees should include a discussion of emergency evacuation procedures. Supervisors are responsible for the safe evacuation of their department in an emergency.

To be prepared for an evacuation: locate the nearest appropriate exit and assembly areas. Remind employees to walk, not run.

If evacuation is necessary:

1. Attempt to sue the public address system to order an evacuation. On the telephone, lift receiver to ear, push ALL CALL button and speak. Repeat the message for as long as needed.

2. If possible, inspect the area to ensure all employees have been safely evacuated.

3. Shut -- but do not lock -- doors and assemble with your co-workers in the nearest designated assembly area and verify that all employees are there. Notify employees when it is safe to return to the building.

EMERGENCY HAZARDOUS WASTE PROCEDURES

In the event of a release or threatened release of a hazardous material, the Safety Director, Tribal Manager or the Tribal Chairman must be contacted immediately. They will report the event or

authorize you to report the event to:

1. The Fire Department: 911
2. Nevada Emergency Management Division: 687-4240 or 687-5300.
3. Nevada Environmental Commission: 687-4670.
4. National Response Center: (800) 424-8802.

When calling any of the above agencies, be prepared to give:

** Your name and phone number.

** Name of business and address.

** Location and type of incident.

** Identity of substance (chemical name).

** Degree of hazard, physical nature and volume.

** Impact on the environment.

** If employees are injured, extent and type of injuries.

** Ask the person you report to for their name and title.

WORKERS' COMPENSATION GENERAL INFORMATION

A work related injury/illness is one which arises out of employment and must occur while the employee is acting in the course of his or her employment. A work related injury/illness may be a specific injury/illness, an existing condition aggravated by work, or an injury/illness caused by continuous trauma developing slowly, over a period of time. The Tribe provides insurance in the event of a work related injury/illness through the State Industrial Insurance System (SIIS).

If an employee is injured on the job, he/she must notify their supervisor as soon as practicable and complete a NOTICE OF INJURY AND OCCUPATIONAL DISEASE form. If notice of the injury is not given in a timely manner, an injured employee could lose his/her workers' compensation benefits. Supervisors who are notified that an employee has been injured on the job should assist the employee in completing the above form, and complete an ACCIDENT REPORT form. Supervisors should then notify the Human Resources department of the injury as soon as possible, and provide them with the completed documentation.

The Tribe attempts to maintain qualified personnel trained in first aid on all shifts. In case of an emergency, the Tribe will arrange for the injured employee to be transported to the

appropriate and most readily available doctor/hospital.

In the event of a work related injury/illness, the supervisor's responsibility is to:

1. Complete an ACCIDENT REPORT form. This must be completed and given to the Human Resources department within 24 hours.

2. Have the employee complete a NOTICE OF INJURY OR OCCUPATIONAL DISEASE form and give it to Human Resources.

3. Always ask the employee how he/she feels and if they need medical care. If they do need medical care, make arrangements to have them transported to the appropriate facility.

4. Using the WORKERS' COMPENSATION INJURY CHECK-OFF LIST, make sure you have completed all the necessary reports and procedures.

5. Inform your Supervisor of the work injury.

6. Maintain open, positive, frequent, sincere communication with the injured employee.

7. Contact Human Resources immediately with any pertinent information that may affect a claim for industrial insurance benefits.

8. If the employee is sent home for the remainder of the day, pay the employee for any hours lost that day.

WORK INJURY STATUS

After medical treatment for a work related injury/illness, the following conditions may exist:

1. If an employee is not released by a physician or chiropractor and cannot return to regular work, then mark the employee's card or timesheet "WI" for work injury. Supervisors should always check with Human Resources to verify that the doctor has released the injured employee before allowing him/her to return to work.

2. If an employee is released by a physician or chiropractor to return to modified work, the Tribe will make an effort to provide a light duty assignment that accommodates the restrictions indicated.

3. If the employee is released by a physician or chiropractor to return to his/her regular job duties, supervisors should be sure that Human Resources has a copy of the release.

4. A follow-up appointment for work related injuries is usually scheduled within the next week. At that time, the employee's

restrictions may be continued or the employee may return to regular job duties. If at any time the employee is unable to return to work in accordance with the physician or chiropractor's restrictions, supervisors should notify Human Resources immediately.

WORKERS' COMPENSATION FRAUD

All employees can help in reducing worker's compensation costs by reporting suspected fraudulent work injuries/illness to Human Resources or by calling 688-1818 or 1-800-266-8688. Workers' compensation fraud is illegal in Nevada and ultimately hurts all employees. Anyone who files or contributes to the filing of a false claim is committing a crime which can be punishable by imprisonment of up to 10 years, forfeiture of all rights of workers' compensation and liability for reasonable costs incurred to investigate and act upon the violation. Some examples of fraudulent claims which can result in prosecution are:

* Filing claims for non-existing injury/illness.
* Filing claims for an injury or illness that is not work related.
* Aiding a co-worker to file a false claim.
* Conspiring with a medical provider to fraudulently obtain benefits.
* A medical care provider over charging or submitting fraudulent billings.

APPENDIX

A

SAFETY ACTION REQUEST FORM
PYRAMID LAKE PAIUTE TRIBE

This form is for use by employees who wish to provide a safety suggestion or report an unsafe workplace condition or practice.

DESCRIPTION OF UNSAFE CONDITION OR PRACTICE:

CAUSES OR OTHER CONTRIBUTING FACTORS:

EMPLOYEE'S SUGGESTION FOR IMPROVING SAFETY:

Has this matter been reported to the Department Head or your Immediate Supervisor? _____YES _____NO

Employee Name (Optional)_____

Department_____ Date_____

The Tribe will investigate any suggestions or questions and will advise you and any other workers involved of our response. If this request is received anonymously, a response will be given verbally to the affected area employees and if appropriate, posted on the bulletin board.

This form can either be submitted to the Safety Committee or be placed in the safety suggestion box.

SAFETY ACTION REQUEST FORM
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The Tribe will investigate any suggestions or questions and will advise you and any other workers involved of our response. If this request is received anonymously, a response will be given verbally to the affected area employees and if appropriate, posted on the bulletin board.

This form can either be submitted to the Safety Committee or be placed in the safety suggestion box.

B

EMPLOYEE SAFETY ORIENTATION FORM
PYRAMID LAKE PAIUTE TRIBE

NAME _____
(Print) Last First Middle Date Employed

DEPARTMENT ASSIGNED _____ TYPE OF WORK _____

PAST WORK EXPERIENCE _____

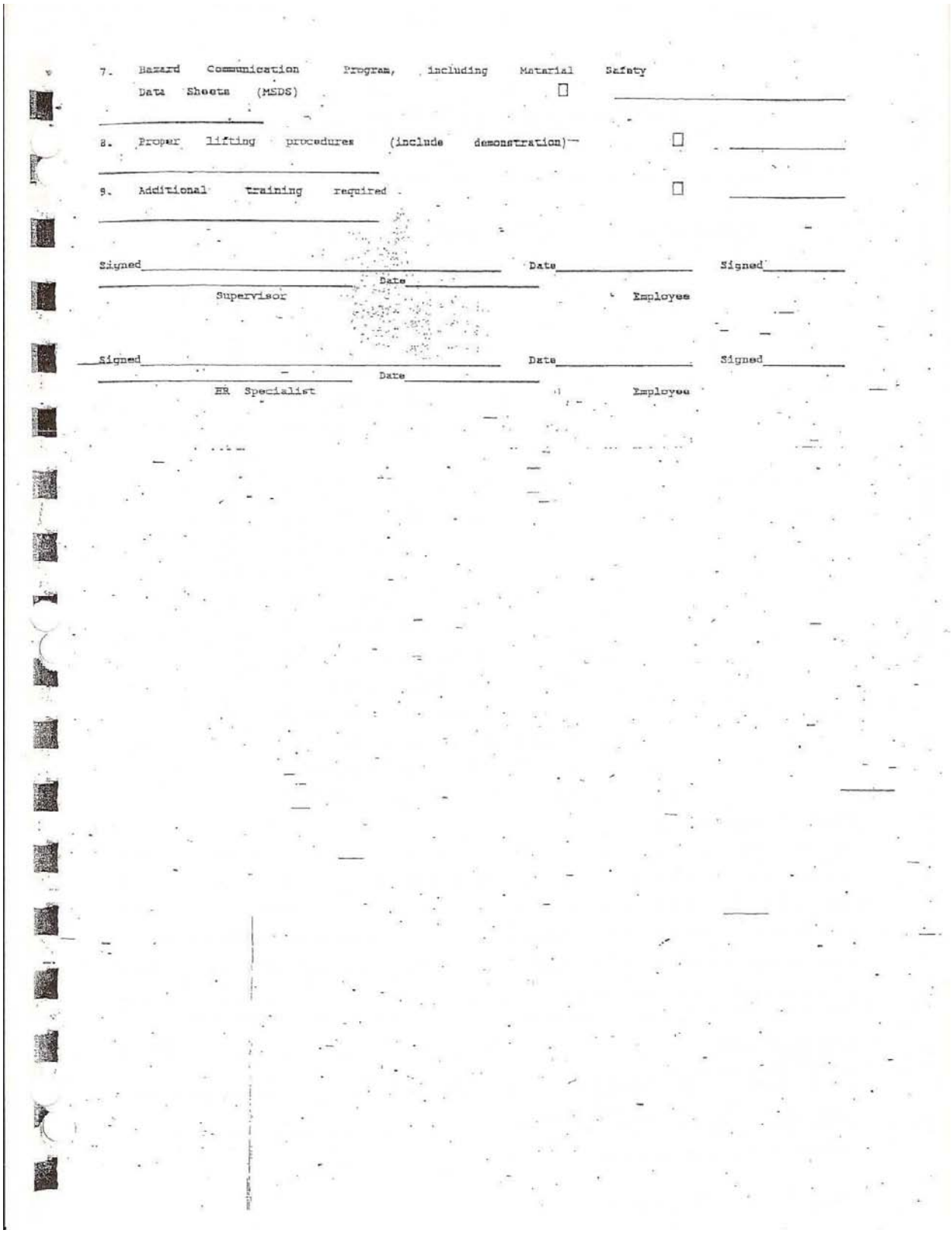
THE FIRST PART OF THIS REPORT SHALL BE COMPLETED BY AN HR SPECIALIST AND THE SECOND PART OF THIS REPORT SHALL BE COMPLETED BY THE EMPLOYEE'S SUPERVISOR FOR EVERY NEW OR TRANSFERRED EMPLOYEE ON THE FIRST DAY OF WORK. THE FOLLOWING ITEMS HAVE BEEN REVIEWED:

HR SPECIALIST

	Check here	COMMENTS
1. Company safety policies and programs	☐	
2. Safety rules (general)	☐	
3. Safety rule enforcement procedures	☐	
4. When, where and how to report injuries	☐	
5. How workers' compensation system works	☐	
6. A copy of the "Workplace Safety" pamphlet with a signed receipt	☐	
7. Notification of medical care providers under contract to treat employees injured on job (Form C-2)	☐	
8. When, where and how to report unsafe conditions	☐	

SUPERVISOR

1. Safety rules (specific to the job)	☐	
2. Review of fire and emergency evacuation plan	☐	
3. Location and use of fire extinguishers	☐	
4. Safety dress code	☐	
5. Assignment, use, and care of personal protective equipment	☐	
6. Importance of housekeeping, i.e., cleaning up spills, etc.	☐	



7. Hazard Communication Program, including Material Safety Data Sheets (MSDS) ☐ _____

8. Proper lifting procedures (include demonstration) ☐ _____

9. Additional training required ☐ _____

Signed _____ Date _____ Signed _____

Supervisor Date Employee

Signed _____ Date _____ Signed _____

HR Specialist Date Employee

C

PYRAMID LAKE PAIUTE TRIBE
SUPERVISOR'S DAILY INSPECTION FORM
TO BE DONE AT THE BEGINNING OF EACH SHIFT

Section:_____

	YES	NO
	☐	☐
All operators wearing proper	☐	☐
Clothing	☐	☐
Ear Plugs	☐	☐
Shoes	☐	☐
Wrist Bands	☐	☐
Back Supports	☐	☐
Safety Glasses		
Hair Length/Jewelry	☐	☐
	☐	☐
Unsafe Conditions Found	☐	☐
Loose Wiring	☐	☐
Purge Shield	☐	☐
Oily Floors	☐	☐
Slick Floors	☐	☐
Cluttered Work Station	☐	☐
Inventory Not Properly Stacked	☐	☐
Machine Safety Switches/Guards Not In Place	☐	☐
Covers Not In Place	☐	☐
Improper Chemical Labeling/Storage		
Tools Not Used Properly		
Repetitive Tasks Not Rotated		

Other Comments:

_____

_____

_____

_____

_____

_____

_____

Reported to:_____ B Y
(Sign):_____
Person/Department responsible for correction:_____

_____

Copy provided to this person/department on:_____

_____
Date

Copy provided to Tribal Chairman on:

PYRAMID LAKE PAIUTE TRIBE D

Date

SAFETY COMMITTEE'S MONTHLY INSPECTION CHECK OFF LIST

Name of person(s) conducting inspection_____

Date of inspection_____ Division_____

THE FOLLOWING LIST IS INTENDED AS A REMINDER. LOOK FOR ALL UNSAFE ACTS AND CONDITIONS AND REPORT THEM BELOW AND TO THE RESPONSIBLE SUPERVISOR.

MACHINERY

_____ Safety Guards in place
_____ Oil leaks
_____ Loose wiring
_____ Is there anything blocking machines?
_____ Is there any temporary wiring?
_____ Is there any exposed or loose wiring?
_____ Are hopper covers secured and do they have chains?

HOUSEKEEPING

_____ Aisles, stairs, and floors clear/clean
_____ Storage of material orderly with ample aisle space?
_____ Light and ventilation
_____ Disposal of waste
_____ Are yard and parking areas clear of debris, oil, or any big holes?

TOOLS

_____ Use of proper tools

FIRST AID

_____ Are first aid kits supplied and locked?
_____ Emergency showers operating

UNSAFE PRACTICES

_____ Excessive speed of vehicles
_____ Improper lifting
_____ Smoking violations
_____ Horseplay/running
_____ Removing/working machine or other safety guards
_____ Are grate covers on floor openings properly covered?

PERSONAL PROTECTIVE EQUIPMENT

_____ Goggles, glasses or face shields
_____ Safety shoes
_____ Hearing protection
_____ Gloves
_____ Respiratory or gas masks
_____ Protective clothing

PRESSURE EQUIPMENT

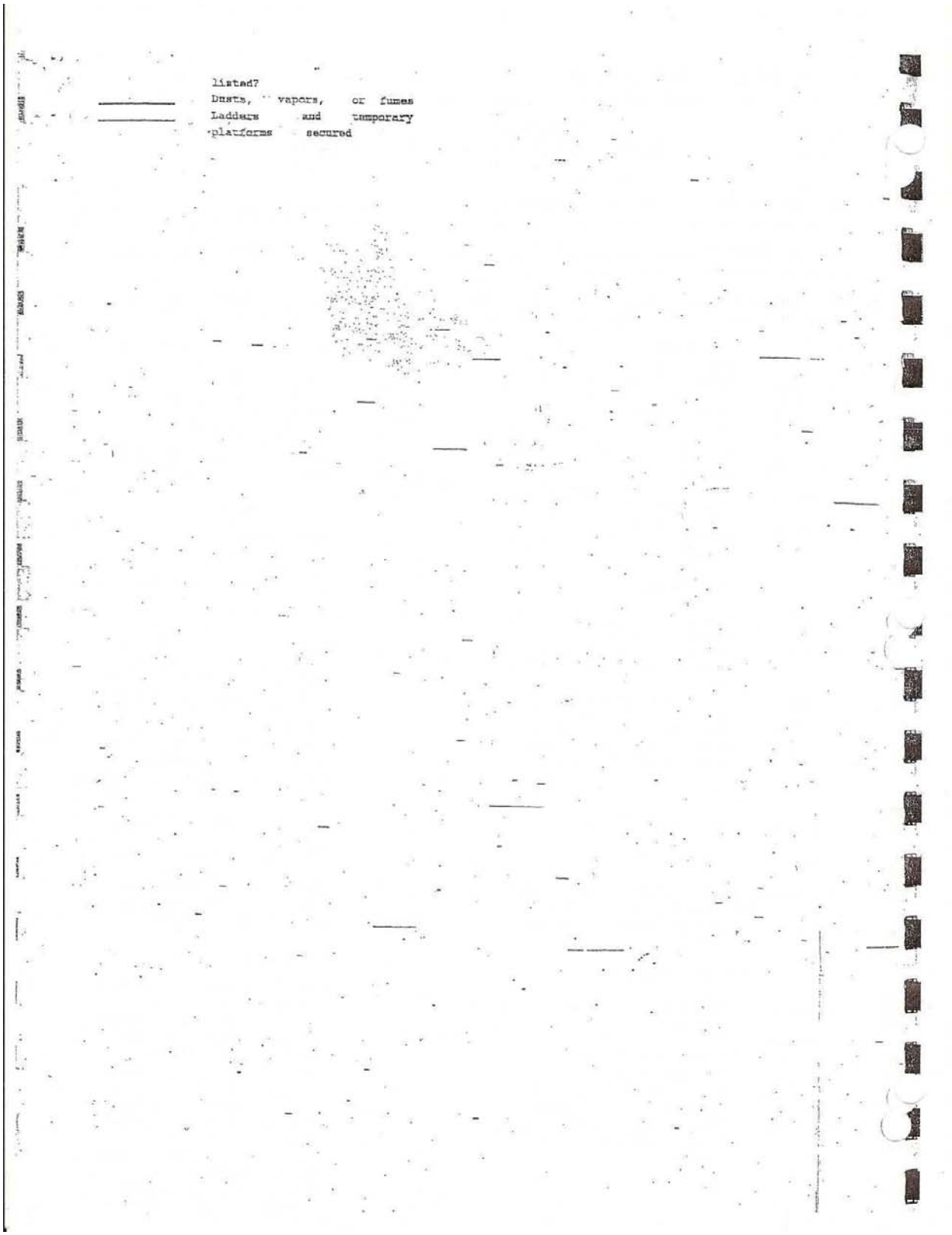
_____ Steam equipment
_____ Air receivers and compressors
_____ Gas cylinders and hose

FIRE PROTECTION

_____ Are all fire extinguishers properly charged, pressurized, in good condition, readily accessible, and numbered?
_____ Are there any fire extinguishers missing?
_____ Are all fire exits clearly marked and visible?
_____ Are maps posted, listing all exits?
_____ Are any sprinkler heads painted or corroded?
_____ Are any sprinklers obstructed by partitions or high piled storage?

MISCELLANEOUS

_____ Are outside lights working properly?
_____ Are all electrical boxes (panels) free and clear behind yellow lines?
_____ Are outside lights working properly?
_____ Are flammable liquids kept in properly marked containers?
_____ Material safety data sheets available
_____ Are new processes, chemicals and solvents


listed?
Dusts, vapors, or fumes
Ladders and temporary
platforms secured

PYRAMID LAKE PAIUTE TRIBE

E

SAFETY COMMITTEE'S MONTHLY
INSPECTION WRITTEN REPORT

UNSAFE CONDITION/ACT OBSERVED	CORRECTIVE ACTION SUGGESTED	SUGGESTION FORWARDED TO

Committee Chairman

Date

PYRAMID LAKE PAIUTE TRIBE F

ACCIDENT REPORT

This report should be completed so as to aid in eliminating the recurrance of similar accidents in the future. All accident reports are to be prepared during the shift on which the accident occurred.

INJURED EMPLOYEE _____ DATE AND TIME OF ACCIDENT _____

SHIFT AND GROUP _____ OCCUPATION _____

DEPT. _____

AGE _____ SEX _____ EMPLOYEE # _____ DATE OF THIS REPORT _____

EXACT LOCATION WHERE ACCIDENT OCCURRED _____

What task was employee performing when injured? (include tool, materials or machinery used) _____

How was the employee injured? _____

Nature and extent of injury: _____

Disposition of case: No medical treatment ☐ Sent to hospital/clinic ☐ Nurse called in ☐

Other ☐ (specify) _____

Check type of incident and fully explain your selection here. _____

INCIDENT CATEGORY

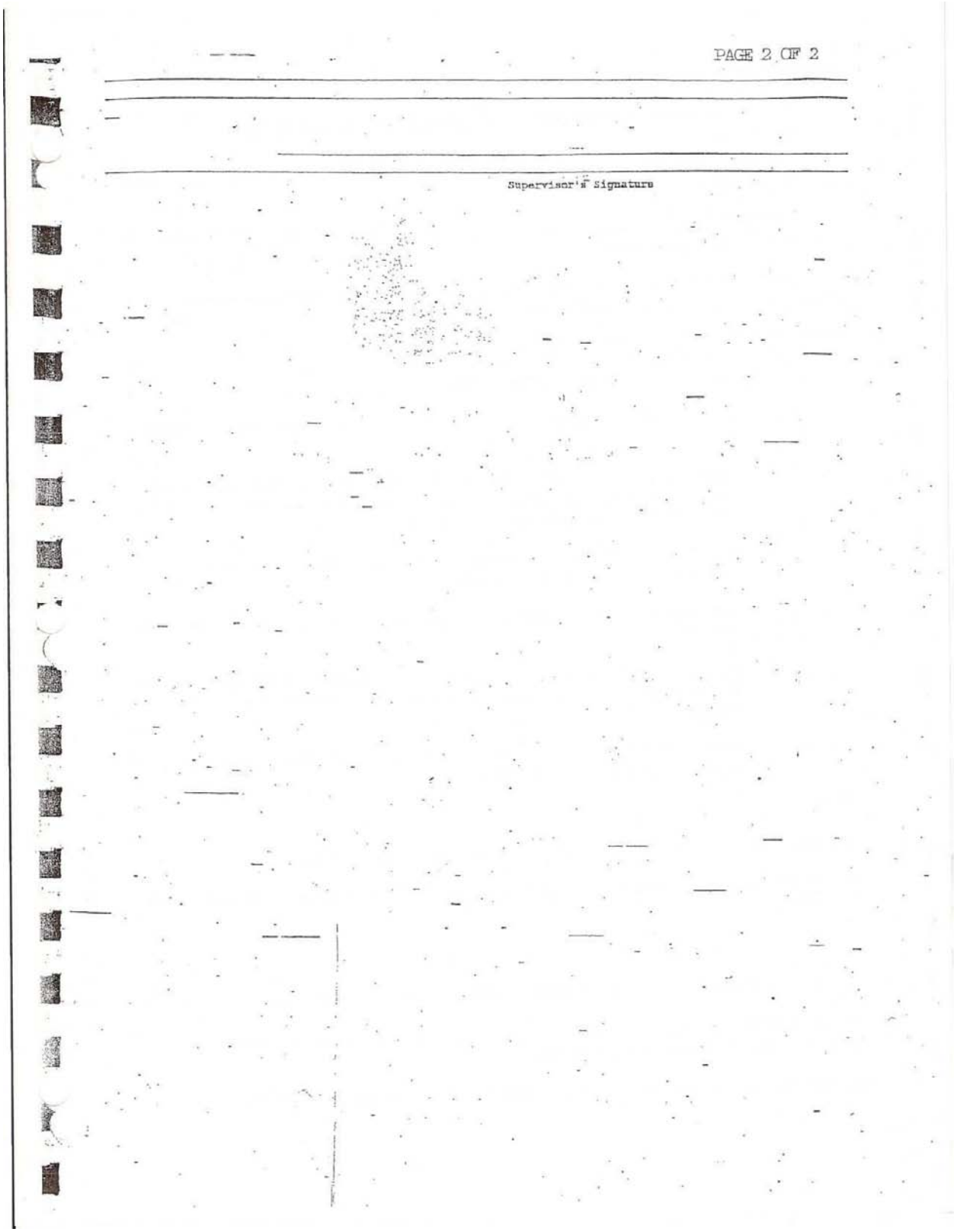
ENVIRONMENTAL		PERSONAL	
Inadequate safeguards	☐	Bodily conditions	☐
Defective equipment or tool	☐	Lack of skill or knowledge	☐
Improper tool or equipment	☐	Attitude/horseplay	☐
Poor housekeeping	☐	Improper apparel or lack of protective equipment	☐

Responsibility for accident Employee ☐ Supervisor ☐ Both ☐

Explain _____

What corrective action has been or will be taken to prevent a recurrence? _____

Does an unsafe condition still exist? Yes _____ No _____ If yes, explain fully _____



PAGE 2 OF 2

Supervisor's Signature

G

PYRAMID LAKE PAIUTE TRIBE

WORKER'S COMPENSATION INJURY CHECK-OFF LIST

EMPLOYEE NAME _____

DEPARTMENT _____ SHIFT _____

DATE OF INJURY _____

PLEASE COMPLETE THE FOLLOWING FOR EACH WORK INJURY.

_____ Fill out an ACCIDENT REPORT form.

_____ Have employee complete - NOTICE OF INJURY OR OCCUPATIONAL DISEASE form.

_____ ALWAYS ask the employee how he/she feels and if they need medical care. If they need medical care, send them immediately to the clinic with an authorization slip signed by you.

_____ Send all doctor's reports to the Worker's Compensation Representative in Human Resources.

_____ Make sure that your supervisor sees the reports after they have been completed.

_____ Make sure that everything is signed and completed correctly and send all originals to the Workers' Compensation Representative in Human Resources.

_____ Human Resources will schedule an injury investigation meeting - It is your responsibility to attend.

NOTE:

Maintain open, frequent communication with the injured worker.

Call Human Resources immediately with any pertinent information that may affect the claim.

IF THIS IS A LOST TIME INJURY, NOTIFY HUMAN RESOURCES IMMEDIATELY.

IF THE EMPLOYEE IS UNABLE TO WORK THE REMAINDER OF THE DAY OF THE INJURY, PAY THE EMPLOYEE FOR ANY HOURS LOST THAT DAY.

IF YOU RUN OUT OF SUPPLIES, PLEASE CONTACT HUMAN RESOURCES.

PYRAMID LAKE PAIUTE TRIBE H

SAFETY COMMITTEE MINUTES

HOLLY DECORATIONS INC., NEVADA				
DATE OF MEETING	DATE OF NEXT MEETING	# OF EMPLOYEES	# OF ACCIDENTS THIS PERIOD OR MONTH	MONTHLY SAFETY SUBJECT

AGENDA:

1. List members present
2. Safety Inspection
3. Read minutes of previous meeting
4. Review of accidents and possible preventive measures
5. Hazards observed or reported to committee
6. Discussion of training and educational activities
7. Report to management

Signed:

Committee Chairman

1. Use other side or blank sheet for extension of minutes.
2. Please attach a copy of the Safety Committee's monthly inspection report and return to Safety Director.

PYRAMID LAKE PAIUTE TRIBE I

SAFETY TRAINING DOCUMENTATION

Date_____

Name of Trainer_____

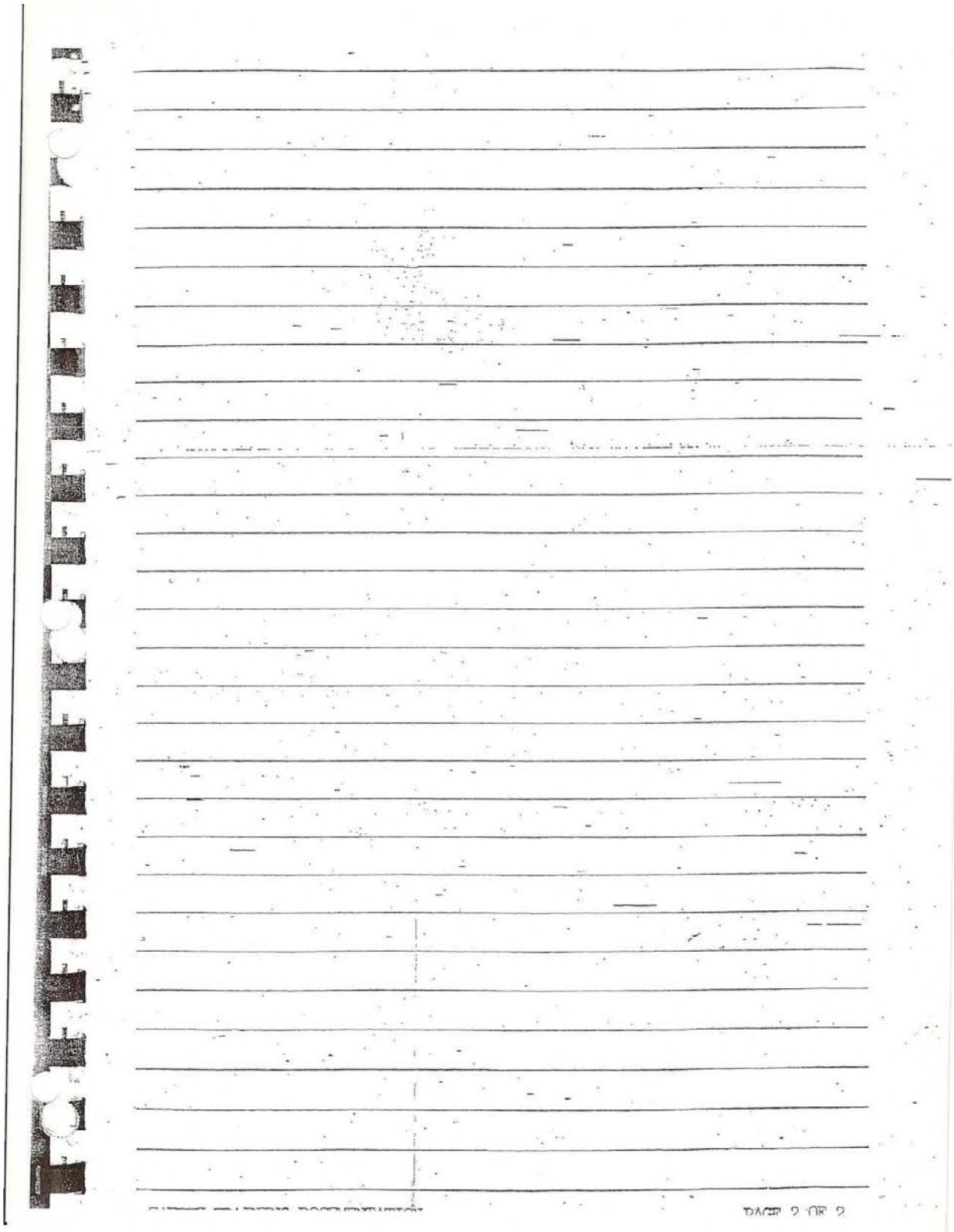
Subject(s) Covered

Training Aids Used

Work Location/Job Safety Class(es) Included

Attendees (Please print and sign your name legibly. Use additional sheets as necessary.)

Print Signature



Appendix C: Laboratory QA Manual

PYRAMID LAKE PAIUTE TRIBE

PYRAMID LAKE FISHERIES
QUALITY ASSURANCE PROJECT PLAN For

Water Quality Monitoring and Analysis

Received by EPA QA office March 10, 1999
Control# WATR233299VSF1

Currently under revision

The QA Plan for the PLPT Water Quality Laboratory is currently under revision. Data Quality Indicator tables have been developed for the water quality parameters measured for this project. The specifications in these DQI tables have been provided to the Water Quality laboratory for review and the laboratory agrees to meet the criteria specified in the DQI tables. Until the PLPT Water Quality Laboratory has updated its QA plan and analytical SOPs, the laboratory will consult the DQI tables provided in Appendix E.

Water Quality Laboratory – Sutcliffe, NV

TABLE OF CONTENTS

Appendix D: Laboratory DQI Tables

Table 1: Summary of Analytical Methods

Analytical Parameter	**PLPT Analytical Method- based on**
Total Phosphate	365.3 EPA
Orthophosphate	365.3 EPA
Nitrate+Nitrite	4500-NO_3^- E Standard Methods, 20th Edition
Ammonia-N	4500-NH_3 F Standard Methods, 20th Edition
Total Kjeldahl Nitrogen	4500-N_{org} C Standard Methods, 20th Edition

Table 2: Summary of Contract Required Detection Limits, Holding Times, and Preservation

Analytical Parameter	**Contract Required Detection Limit (CRDL)**	**Maximum Holding Times**	**Preservation**
Total Phosphorus	0.010 mg/L	28 days	Do Not filter samples. Add 2 ml H_2SO_4/L, Refrigerate[1] Or Freeze[2]
Ortho-Phosphate	0.010 mg/L	28 days	Filter Samples. Freeze[2]
Nitrate+Nitrite	0.010 mg/L	28 days	Filter samples. - w/in 48 hrs of collection: Refrigerate[1] - w/in 28 days of collection: Add 2 mL H_2SO_4/L, Refrigerate[1]
Ammonia-N	0.010 mg/L	28 days	Filter samples. - w/in 24 hrs of collection: Refrigerate[1] - w/in 28 days of collection: Freeze at -20°C or add H_2SO_4 to pH <2 and refrigerate[1]
Total Kjeldahl Nitrogen	0.010 mg/L	28 days	Add H_2SO_4 to pH 1.5 to 2.0 and refrigerate[1]

[1] Refrigerate = storage at 4°C ± 2°C, in the dark
[2] Freeze = storage at or below -10°C

Data Calculation and Reporting Units

Calculate the sample results according to PLPT Laboratory SOPs.

Report sample results in concentration units of milligram per liter (mg/L). Report concentrations that are less than 10 mg/L to 2 significant figures, and concentrations that are greater than or equal to 10 mg/L to 3 significant figures.
For rounding results, adhere to the following rules:

a) If the number following those to be retained is less than 5, round down;
b) If the number following those to be retained is greater than 5, round up; or
c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

Table 3: Summary of the Standard Curve Calibration Evaluation

Calibration Element	**Frequency**	**Acceptance Criteria**	**Corrective Action**
Standard Curve Calibration (blank + 3 points)	With each set of samples analyzed	$r \geq 0.995$	1. Terminate analysis 2. Recalibrate and verify before sample analysis
Blank Verification	With each set of samples analyzed	± 2 standard deviations	1. Terminate analysis 2. Recalibrate and verify before sample analysis
Slope of Standard Curve Verification	With each set of samples analyzed	± 2 standard deviations	1. Terminate analysis 2. Recalibrate and verify before sample analysis
Intercept of Standard Curve Verification	With each set of samples analyzed	|intercept -blank| /slope > Detection Limit	1. Terminate analysis 2. Recalibrate and verify before sample analysis

Prepare a standard curve with each set of samples analyzed.

Dilute and reanalyze samples with concentrations exceeding the range of the calibration curve. Results for such reanalyses should fall within the mid-range of the calibration curve. Report results and submit documentation for both analyses.

Table 4: Summary of Internal Quality Control Procedures for PLPT WQ Laboratory

QC Element	Frequency	Acceptance Criteria	Corrective Action
Duplicate Samples	35% of samples	# of Duplicates ≥ 35% of Samples	N/A
Duplicate Sample Variability	N/A	<20% of Duplicates exceeds maximum variability[1]	1. Flag associated data with an "*"
Matrix Spike	With each set of samples analyzed	± 15% from expected value	1. Flag associated data with an "N"

[1] Variability Limits
DL = Detection Limit
Calculation of Limits

Interval	RSD%
<2 · DL	100%
2-3 · DL	80%
3-4 · DL	60%
4-5 · DL	40

Appendix E: Laboratory Standard Operating Procedures

Standard Operating Procedure for:

Total Phosphorus Determination

1.0 Scope and Application

1.1 This method covers the determination of specified forms of phosphorus in drinking, surface and saline waters, domestic and industrial wastes.

1.2 The methods are based on reactions that are specific for the orthophosphate ion. Thus, depending on the prescribed pretreatment of the sample, the various forms may be determined.

1.3 The methods are usable in the 0.01 to 1.2 mg P/L range.

2.0 Summary of Method

2.1 Total Phosphorus (P) – all of the phosphorus present in the sample, regardless of form, as measured by the persulfate digestion procedure.

2.2 Ammonium molybdate and potassium antimonyl tartrate react in acid medium with dilute solutions of phosphorus to form an antimony-phospho-molybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid. The color is proportional to the phosphorus concentration.

2.3 Only orthophosphate forms a blue color in this test. Polyphosphates (and some organic phosphorus compounds) may be converted to the orthophosphate form by sulfuric acid hydrolysis. Organic phosphorus compounds may be converted to the orthophosphate form by persulfate digestion.

3.0 Interferences

3.1 Arsenate is determined similarly to phosphorus and should be considered when present. Concentrations as low as 0.1 mg As/L interfere with the phosphate determination. This interference may be eliminated by reducing the arsenic acid to arsenious acid with sodium bisulfite.

3.2 When high concentrations of iron are present low recovery of phosphorus will be obtained because it will use some of the reducing agent. The bisulfite treatment will also eliminate the interference.

4.0 Filtration

4.1 Do not filter samples.

5.0 Preservation and Storage

5.1 Pour samples into clean 250 mL bottles labeled with the sample ID, sample date, preservation, and analytical parameter.

5.2 If the analysis cannot be performed the day of collection, the sample should be preserved by the addition of 2 mL concentrated H_2SO_4 (Sulfuric Acid) per liter and refrigerate at 4°C or freeze at or below -10°C without any additions.

5.2.1 Example sample sizes and preservation:
200 mL sample use 0.4 mL conc. H_2SO_4
250 mL sample use 0.5 mL conc. H_2SO_4

6.0 Equipment

6.1 Spectrophotometer: Suitable for measurements at 900 nm with a light path of 1 cm or longer.

6.2 Acid-washed glassware: all glassware should be washed with 10% HCl and rinsed with deionized water. This glassware should only be used for the determination of phosphorus and after should be washed as stated above and kept covered until needed again. Commercial detergents should never be used.

Occasionally all glassware should be washed with hot 1:1 HCl and rinsed with deionized water. The acid-washed glassware should be filled with deionized water and treated with all the reagents to remove the last traces of phosphorus that might be absorbed on the glassware.

A set of 125 mL Erlenmeyer flasks should be dedicated to the determination of phosphorus.

6.3 Glassware: Class A volumetric flasks and pipets as required.

6.4 Waterbath: Capable of reaching a temperature of 95°C.

6.5 Balance: Analytical, capable of accurately weighing to the nearest 0.001 g.

6.6 Eppendorf dispenser: Manual hand dispenser with 50 mL volume tips. For use when adding reagents to samples. Use a different tip for each reagent added.

6.7 Hot plate: Use large hot plate within fume hood with safety shield.

7.0 Reagents

7.1 **Ammonium molybdate-antimony potassium tartrate solution:** Dissolve 8 g of ammonium molybdate and 0.2 antimony potassium tartrate in 800 mL of deionized water and dilute to 1 liter.

7.2 **Ascorbic acid solution:** Dissolve 30 g of ascorbic acid in 400 mL of deionized water and dilute to 500 mL. Add 1 mL of acetone. This solution is stable for two weeks. Store this solution in the refrigerator in an opaque container. Ascorbic acid needs to be at room temperature when it is

added to samples. Prior to running a test, pour about 100 ml into a beaker, and store in a dark place at room temperature.

7.3 **Sulfuric acid, 11N:** Slowly add 310 mL of concentrated H_2SO_4 to approximately 600 mL of deionized water. Cool and dilute to 1000 mL.

7.4 **Sodium bisulfite ($NaHSO_3$) solution:** 1N sulfuric acid (H_2SO_4) will be needed, make by adding 28 mL of concentrated sulfuric acid to about 600 mL of deionized water and dilute to 1 liter. Dissolve 52 g of sodium bisulfite ($NaHSO_3$) in the 1 liter of 1N H_2SO_4. Final volume will be slightly more than 1 liter.

7.5 **Ammonium persulfate.**

7.6 **Stock phosphorus solution:** Dissolve 0.4393 g of pre-dried (dry for 1 hour at 105℃) Potassium dihydrogen phosphate (KH_2PO_4) in deionized water and dilute to 1000 mL. 1.0 mL = 0.1 mg P This solution will contain 100 mg/L P.

7.7 **Standard phosphorus solution:** Dilute 100 mL of stock phosphorus solution to 1000 mL with deionized water. 1 mL = 0.01 mg P. This solution will contain 10 mg/L P.

Prepare an appropriate series of standards by diluting suitable volumes of standard or stock solutions to 1000 mL with deionized water.

Stock solution and standards should not be stored for more than 6 months.

Example: Diluting 5 mL of standard solution to 1000 mL makes a standard of 0.05 mg/L P.

8.0 Procedure

Follow all the steps in this procedure for those samples that may have arsenate interference. For samples that arsenate interference is not a factor, use the following procedure skipping steps 8.1.4 and 8.8 – 8.10.

8.1 Preparation

8.1.1 Retrieve the samples from refrigerator. The samples should be near room temperature when beginning test.

8.1.2 Retrieve the dedicated set of glassware (125 mL Erlenmeyer flasks).

8.1.3 All 125 mL Erlenmeyer flasks dedicated for the phosphorus determination should be labeled to avoid confusion between blanks, standards, spikes, and samples.

8.1.4 Prepare the waterbath. The water should be at least 1.5 – 2.0 inches deep. Add deionized water to increase depth, if necessary. Plug in the waterbath to begin heating; it will require at least 2 hours to reach the test temperature (95°C). This step is only for the analysis of samples that may have arsenate interference.

8.1.5 Start the hotplate just before beginning analysis to give it some time to warm up.

8.1.6 Retrieve all reagents needed. Make reagents before starting test if needed.

8.2 Prepare blanks, standards, spikes, and samples. Rinse the graduated cylinder or pipette used to measure blanks, standards, spikes, and samples in between measurements with deionized water.

8.2.1 Blanks: Measure 50 mL of deionized water and transfer to labeled flask. Blanks should be run in duplicate.

8.2.2 Standards: Measure 50 mL of appropriate standard and transfer to labeled flasks. Standards should be run in duplicate.

8.2.3 Samples: Mix the sample and then measure and transfer 50 mL to labeled flasks. At least 30% of the water samples should be run in duplicate, the actual number will depend on how many samples are in the batch.

An aliquot portion of the sample diluted to 50 mL can be used if the sample is suspected to be higher than the prepared standards. Note any dilutions on laboratory bench sheet and laboratory notebooks.

8.2.4 Spike: Using a pipette, measure 8 mL of the 1.0 mg/L standard solution into a 100 mL volumetric flask, then dilute to 100 mL with one of the samples. Cap and mix thoroughly. This results in a spike addition of 0.08 mg P.

Measure 50 mL of the spike solution and transfer to the labeled flask. Record which sample was used for the spike solution on laboratory bench sheets and in laboratory notebooks.

8.3 Add 1 mL of 11N sulfuric acid to each flask and mix.

8.4 Add 0.4 g of ammonium persulfate to each flask and mix.

8.5 Place all flasks on hotplate. Pull the vent hood down to about 4 inches above flasks. Turn on the vent fan, and ceiling exhaust fan. Let the flasks boil until the volume in each is reduced to about 10 mL. This normally takes about 30 to 60 minutes.

8.6 Remove flasks from hotplate when final volume of about 10 mL is reached. Turn off hotplate when all samples have been reduced in volume to the specified amount. Let samples cool.

8.7 Dilute volume of samples with deionized water using a graduated cylinder. Be sure to rinse cylinder between measurements.

8.7.1 Samples with arsenate interference: Dilute to 40 mL.
8.7.2 Samples without arsenate interference: Dilute to 50 mL, and skip to step 8.11.

8.8 Add 5 mL of sodium bisulfite to each flask and mix.

8.9 Place flasks in the waterbath, which should be at 95°C. Place circular weights around the necks of the flasks to prevent them from tipping over. Flasks should not be stoppered while in the waterbath. Cover the waterbath with the metal hood. Watch the waterbath temperature, which will fall after the flasks are put in place. The flasks need to set in the waterbath for 20 minutes after the temperature reaches 95°C.

8.10 After 20 minutes, remove flasks from the waterbath and unplug the power cord. When the samples have cooled, using a graduated cylinder dilute the volume to 50 mL with deionized water. Rinse the graduated cylinder between measurements.

8.11 Add 4 mL of ammonium molybdate-antimony potassium tartrate to each flask and mix.

8.12 Add 2 mL of ascorbic acid to each flask and mix. Let set for 5 minutes.

8.13 After 5 minutes, measure the absorbance of the samples in the spectrophotometer with the wavelength set at 900nm. The color of samples will remain stable for at least one hour. The 10 cm cell should be used, unless a reading cannot be obtained, in which case the 1 cm cell is used.

9.0 Calculation

9.1 Prepare a standard curve by plotting the absorbance values of standards versus the corresponding phosphorus concentrations using Excel.

9.2 Obtain concentration value of sample directly from prepared standard curve. Report results as P, mg/L.

10.0 References

11.0 U.S. EPA. “Methods for Chemical Analysis of Water and Wastes”. Method 365.3. EPA/600/4-79/020. US EPA, Office of Research and Development. Washington, DC 20460. March 1983.

Standard Operating Procedure for:

Orthophosphate (Dissolved Reactive Phosphorus) Analysis

1.0 Scope and Application

1.1 This method covers the determination of specified forms of phosphorus in drinking, surface and saline waters, domestic and industrial wastes.

1.2 The methods are based on reactions that are specific for the orthophosphate ion. Thus, depending on the prescribed pretreatment of the sample, the various forms may be determined.

1.3 The methods are usable in the 0.01 to 1.2 mg P/L range.

2.0 Summary of Method

2.1 Ammonium molybdate and potassium antimonyl tartrate react in acid medium with dilute solutions of phosphorus to form an antimony-phospho-molybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid. The color is proportional to the phosphorus concentration.

3.0 Interferences

3.1 Arsenate is determined similarly to phosphorus and should be considered when present. Concentrations as low as 0.1 mg As/L interfere with the phosphate determination. This interference may be eliminated by reducing the arsenic acid to arsenious acid with sodium bisulfite.

3.2 When high concentrations of iron are present low recovery of phosphorus will be obtained because it will use some of the reducing agent. The bisulfite treatment will also eliminate the interference.

4.0 Filtration

4.1 Filter samples immediately after collection, through a 0.45-µm membrane filter. Wash membrane filters by running 150-mL of deionized water through them before contact with sample water. A glass fiber filter may be used to pre-filter hard-to-filter-samples.

5.0 Preservation and Storage

5.1 Once filtered pour samples into clean 250 mL bottles labeled with the sample ID, sample date, preservation, and analytical parameter.

5.2 If the analysis cannot be performed the day of collection, the sample should be preserved by freezing at or below -10°C.

6.0 Equipment and Supplies

6.1 Spectrophotometer: Suitable for measurements at 900 nm with a light path of 1 cm or longer.

6.2 Acid-washed glassware: all glassware should be washed with 10% HCl and rinsed with deionized water. This glassware should only be used for the determination of phosphorus and after should be washed as stated above and kept covered until needed again. Commercial detergents should never be used.

Occasionally all glassware should be washed with hot 1:1 HCl and rinsed with deionized water. The acid-washed glassware should be filled with deionized water and treated with all the reagents to remove the last traces of phosphorus that might be absorbed on the glassware.

A set of 125 mL Erlenmeyer flasks should be dedicated to the determination of phosphorus.

6.3 Glassware: Class A volumetric flasks and pipets as required.

6.4 Waterbath: Capable of reaching a temperature of 95°C.

6.5 Balance: Analytical, capable of accurately weighing to the nearest 0.001 g.

6.6 Eppendorf dispenser: Manual hand dispenser with 50 mL volume tips. For use when adding reagents to samples. Use a different tip for each reagent added.

7.0 Reagents

7.1 **Ammonium molybdate-antimony potassium tartrate solution:** Dissolve 8 g of ammonium molybdate and 0.2 antimony potassium tartrate in 800 mL of deionized water and dilute to 1 liter.

7.2 **Ascorbic acid solution:** Dissolve 30 g of ascorbic acid in 400 mL of deionized water and dilute to 500 mL. Add 1 mL of acetone. This solution is stable for two weeks. Store this solution in the refrigerator in an opaque container. Ascorbic acid needs to be at room temperature when it is added to samples. Prior to running a test, pour about 100 ml into a beaker, and store in a dark place at room temperature.

7.3 **Sulfuric acid, 11N:** Slowly add 310 mL of concentrated H_2SO_4 to approximately 600 mL of deionized water. Cool and dilute to 1000 mL.

7.4 **Sodium bisulfite ($NaHSO_3$) solution:** 1N sulfuric acid (H_2SO_4) will be needed, make by adding 28 mL of concentrated sulfuric acid to about 600 mL of deionized water and dilute to 1 liter. Dissolve 52 g of sodium bisulfite ($NaHSO_3$) in the 1 liter of 1N H_2SO_4. Final volume will be slightly more than 1 liter.

7.5 **Stock phosphorus solution:** Dissolve 0.4393 g of pre-dried (dry for 1 hour at 105°C) Potassium dihydrogen phosphate (KH_2PO_4) in deionized water and dilute to 1000 mL. 1.0 mL = 0.1 mg P This solution will contain 100 mg/L P.

7.6 **Standard phosphorus solution:** Dilute 100 mL of stock phosphorus solution to 1000 mL with deionized water. 1 mL = 0.01 mg P. This solution will contain 10 mg/L P.

Prepare an appropriate series of standards by diluting suitable volumes of standard or stock solutions to 1000 mL with deionized water.

Stock solution and standards should not be stored for more than 6 months.

Example: Diluting 5 mL of standard solution to 1000 mL makes a standard of 0.05 mg/L P.

8.0 Procedure

Follow all the steps in this procedure for those samples that may have arsenate interference. For samples that arsenate interference is not a factor, use the following procedure skipping steps 8.1.4 and 8.3 – 8.5.

8.1 Preparation

8.1.1 Retrieve the samples from refrigerator. The samples should be near room temperature when beginning test.

8.1.2 Retrieve the dedicated set of glassware (125 mL Erlenmeyer flasks).

8.1.3 All 125 mL Erlenmeyer flasks dedicated for the phosphorus determination should be labeled to avoid confusion between blanks, standards, spikes, and samples.

8.1.4 Prepare the waterbath. The water should be at least 1.5 – 2.0 inches deep. Add deionized water to increase depth, if necessary. Plug in the waterbath to begin heating; it will require at least 2 hours to reach the test temperature (95°C). This step is only for the analysis of samples that may have arsenate interference.

8.1.5 Retrieve all reagents needed. Make reagents before starting test if needed.

8.2 Prepare blanks, standards, spikes, and samples. Rinse the graduated cylinder or pipette used to measure blanks, standards, spikes, and samples in between measurements with deionized water.

8.2.1 Blanks: Measure 50 mL of deionized water and transfer to labeled flask. Blanks should be run in duplicate.

8.2.2 Standards: Measure 50 mL of appropriate standard and transfer to labeled flasks. Standards should be run in duplicate.

8.2.3 Samples: Mix the sample and then measure and transfer 50 mL to labeled flasks. At least 30% of the water samples should be run in duplicate, the actual number will depend on how many samples are in the batch.

An aliquot portion of the sample diluted to 50 mL can be used if the sample is suspected to be higher than the prepared standards. Note any dilutions on laboratory bench sheet and laboratory notebooks.

8.2.4 Spike: Using a pipette, measure 8 mL of the 1.0 mg/L standard solution into a 100 mL volumetric flask, then dilute to 100 mL with one of the samples. Cap and mix thoroughly. This results in a spike addition of 0.08 mg P.

Measure 50 mL of the spike solution and transfer to the labeled flask. Record which sample was used for the spike solution on laboratory bench sheets and in laboratory notebooks.

8.3 Add 5 mL of sodium bisulfite to each flask and mix.

8.4 Place flasks in the waterbath, which should be at 95°C. Place circular weights around the necks of the flasks to prevent them from tipping over. Flasks should not be stoppered while in the waterbath. Cover the waterbath with the metal hood. Watch the waterbath temperature, which will gall after the flasks are put in place. The flasks need to set in the waterbath for 20 minutes after the temperature reaches 95°C.

8.5 After 20 minutes, remove flasks from the waterbath and unplug the power cord. When the samples have cooled, using a graduated cylinder dilute the volume to 55 mL with deionized water. Rinse the graduated cylinder between measurements.

8.6 Add 1 mL of 11N sulfuric acid to each flask and mix.

8.7 Add 4 mL of ammonium molybdate-antimony potassium tartrate to each flask and mix.

8.8 Add 2 mL of ascorbic acid to each flask and mix. Let set for 5 minutes.

8.9 After 5 minutes, measure the absorbance of the samples in the spectrophotometer with the wavelength set at 900 nm. The color of samples will remain stable for at least one hour. The 10 cm cell should be used, unless a reading cannot be obtained, in which case the 1 cm cell is used.

9.0 Calculation

9.1 Prepare a standard curve by plotting the absorbance values of standards versus the corresponding phosphorus concentrations using Excel.

9.2 Obtain concentration value of sample directly from prepared standard curve. Report results as P, mg/L.

10.0 References

U.S. EPA. “Methods for Chemical Analysis of Water and Wastes”. Method 365.3. EPA/600/4-79/020. US EPA, Office of Research and Development. Washington, DC 20460. March 1983.

Nitrite + Nitrate

1.0 Scope and Application

1.1 This method covers the determination of nitrite + nitrate in drinking, ground, surface and saline waters, domestic and industrial wastes.

1.2 The applicable range is 0.01 – 1.0 mg NO_3^--N/L. Higher concentrations can be determined by sample dilution.

2.0 Summary of Method

2.1 Nitrate (NO_3^-) is reduced almost quantitatively to nitrite (NO_2^-) in the presence of cadmium (Cd). This method uses commercially available Cd granules treated with copper sulfate ($CuSO_4$) and packed in a glass column.

2.2 The NO_2^- produced thus is determined by diazotizing with sulfanilamide and coupling with N-(1-naphthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye that is measured colorimetrically. A correction may be made for any NO_2^- present in the sample by analyzing without the reduction step.

3.0 Interferences

3.1 Suspended matter in the column will restrict sample flow. Colorimetric samples require an optically clear sample. Filter turbid samples through 0.45-µm-pore-diameter membrane filter. Test filters for nitrate contamination.

3.2 Concentrations of iron, copper, or other metals above several milligrams per liter lower reduction efficiency. Add EDTA to samples to eliminate this interference.

3.3 Oil and grease will coat the Cd surface. Remove by pre-extraction with an organic solvent (see Section 5520, Standard Methods).

3.4 Residual chlorine can interfere by oxidizing the Cd column, reducing its efficiency. Check samples for residual chlorine (see DPD method in Section 4500-Cl, Standard Methods). Remove residual chlorine by adding sodium thiosulfate ($Na_2S_2O_3$) solution (Section 4500-NH_3.B.3d, Standard Methods).

3.5 Sample color that absorbs at about 540 nm interferes.

4.0 Filtration

4.1 Filter samples immediately after collection, through a 0.45-µm membrane filter. Wash membrane filters by running 150-mL of deionized water through them before contact with sample water. A glass fiber filter may be used to pre-filter hard-to-filter-samples.

5.0 Preservation and Storage

5.1 Once filtered pour samples into clean 250 mL polypropylene bottles labeled with the sample ID, sample date, preservation, and analytical parameter.

5.2 Start NO_3^- determinations promptly after sampling. If storage is necessary, store for up to 48 h at 4°C; for longer storage, preserve with 2 mL of concentrated H_2SO_4/L and store at 4°C. Note: when sample is preserved with acid, NO_3^- and NO_2^- cannot be determined as individual species.

6.0 Equipment

6.1 Reduction columns (2): Purchased cadmium reduction columns.

6.2 Column stand.

6.3 Spectrophotometer: Suitable for use at 543 nm, providing a light path of 1 cm or longer.

6.4 Acid-washed glassware: all glassware should be washed with 10% HCl and rinsed with deionized water. This glassware should only be used for the determination of nitrite + nitrate and after should be washed as stated above and kept covered until needed again.

A set of 125 mL Erlenmeyer flasks should be dedicated to the determination of nitrite + nitrate.

6.5 Glassware: Class A volumetric flasks and pipettes as required.

6.6 Balance: Analytical, capable of accurately weighing to the nearest 0.001 g.

6.7 Adjustable volume pipettes: Manual hand dispenser with 5 mL volume tips. For use when adding reagents to samples. Use a different adjustable volume pipette and tip for each reagent added.

6.8 Bottle top dispenser: Dispenser with a 50 mL capacity. Used for adding Ammonium chloride-EDTA solution to samples.

7.0 Reagents

7.1 **Nitrate-free water:** Use deionized water of highest purity to prepare all solutions and dilutions. The absorbance of a reagent blank prepared with this water should not exceed 0.10.

7.2 **Cadmium granules:** 20 to 100-mesh Cd granules. Before storage wash granules with 6*N* HCl (note: limit the cadmium contact with acid to less than 15 minutes). Rinse with deionized water and store in a glass jar containing enough dilute NH_4Cl-EDTA to cover the granules.

Note: Occasionally it is necessary to re-sieve the Cd granules to remove the fines that can clog the columns. Run the Cd through a #25 sieve and return the retained granules to the storage bottle for use in testing. Store the fines in another bottle, under dilute Ammonium Chloride-EDTA solution and discard the fines with other hazardous waste.

7.3 **Color reagent:** To 800 mL deionized water add 100 mL 85% phosphoric acid and 10 g sulfanilamide. After dissolving sulfanilamide completely, add 1 g N-(1-naphthyl)-

ethylenediamine dihydrochloride. Mix to dissolve, then dilute to 1 L with deionized water. Solution is stable for about **1 month** when stored in a dark bottle in refrigerator.

7.4 **Ammonium chloride-EDTA solution:** Dissolve 26 g ammonium chloride (NH_4Cl) and 3.4 g EDTA (disodium ethylenediamine tetraacetate) in 1700 mL water. Adjust to pH 8.5 with concentrated ammonium hydroxide (NH_4OH) and dilute to 2L.

7.5 **Dilute ammonium chloride-EDTA solution:** Dilute 600 mL NH_4Cl-EDTA solution to 1000 mL with deionized water.

7.6 **Hydrochloric acid, HCl, 6N:** Fill a 100 mL graduated cylinder to the 40 mL mark with deionized water. Then fill to the 90 mL mark with concentrated hydrochloric acid. Let the solution cool then fill to 100 mL with deionized water. (HCl, 6N = 1:1 or 50% HCl)

7.7 **Copper sulfate solution, 2%:** Dissolve 20 g copper sulfate-pentahydrate ($CuSO_4.5H_2O$) in 500 mL deionized water and dilute to 1 L.

7.8 **Stock nitrate solution:** Dry potassium nitrate (KNO_3) in an oven at 105°C for 24 hours. Dissolve 7.218 g in deionized water and dilute to 1000 mL. Preserve with 2 mL trichloromethane ($CHCl_3$) per liter. This solution is stable for **6 months**. 1 mL = 1.0 mg or 1000 mg/L NO_3-N.

7.9 **Stock nitrite solution:** Dry potassium nitrite (KNO_2) in an oven at 105°C for 24 hours. Dissolve 6.072 g KNO_2 in 500 mL of deionized water and dilute to 1 L in a volumetric flask. Preserve with 2 mL of trichloromethane and keep under refrigeration. This solution is stable for **3 months** 1 mL = 1.0 mg or 1000 mg/L NO_2-N. This solution should then be diluted to 100 mg/L.

8.0 Procedure

8.1 Preparation

8.1.1 Retrieve the samples from refrigerator. The samples should be near room temperature when beginning the test.

8.1.2 Retrieve all reagents needed. Prepare reagents before starting test if needed.

8.1.3 Retrieve the dedicated set of glassware (125 mL Erlenmeyer flasks).

8.1.4 All 125 mL Erlenmeyer flasks dedicated for the nitrate + nitrite determination should be labeled to avoid confusion between blanks, standards, spikes, and samples.

8.2 Prepare cadmium

8.2.1 Pour enough cadmium granules to fill two reduction columns in a 400 mL beaker.

8.2.2 Wash cadmium granules with 6*N* (50%) HCl and rinse with deionized water. Note: limit the cadmium contact with acid to less than 15 minutes. The color of the cadmium so treated should be silver. Note: All acid that contacts cadmium should be discarded as hazardous waste.

8.2.3 Pour enough 2% copper sulfate solution in beaker for 5 minutes or until blue color partially fades. Pour out used solution and repeat with fresh copper sulfate solution until a brown colloidal precipitate begins to develop.

8.2.4 Gently flush with deionized water to remove all precipitated copper. Flush 10 - 20 times, until water comes out clear. If you don't rinse the granules thoroughly, the reduction columns will get plugged up and reduction efficiency will be reduced. The color of the cadmium so treated should be black.

8.3 Prepare reduction columns.

8.3.1 Insert glass wool plug into bottom of reduction column.

8.3.2 Assemble reduction columns on stand. Until this analysis is familiar, it is recommended to only use one column. Columns should be set at a sufficient height to easily place and remove 250 mL beakers beneath them.

8.3.3 Fill columns with deionized water.

8.3.4 Add sufficient copper-cadmium (Cu-Cd) granules to produce a column 18.5 cm long. Gently tap the columns so the granules settle, the "tightness" of packing should be the same in each column. Maintain water level above Cu-Cd granules to prevent entrapment of air.

8.3.5 Wash column with 200 mL dilute NH_4Cl-EDTA solution.

8.3.6 Activate column by passing through it, at 7 to 10 mL/min, at least 100 mL of solution composed of 25% 1.0 mg NO_3^-N/L standard and 75% NH_4Cl-EDTA solution. (15 mL : 45 mL)

8.3.7 Pour 50 mL dilute NH_4Cl-EDTA into column and close stopcock.

8.4 Prepare standards to be used for standard curve. The standards should bracket the sample concentrations. Use a minimum of three nitrate standards and one nitrite standard (concentration should be the same as one of the nitrate standards), in addition to three reagent blanks (deionized water) two for nitrate and one for nitrite.

8.4.1 Pour a small amount (3-5 mL) of the nitrite stock solution into a clean 50 mL beaker. Using a volumetric pipette, transfer exactly 1 mL of the nitrite stock solution from the beaker to a clean 100 mL volumetric flask. Dilute to 100 mL with deionized water. This makes 10 mg/L-NO_2 solution.

8.4.2 From the 10 mg/L nitrite solution, prepare a 1 mg/L nitrite solution. Using a volumetric pipette, transfer 10 mL of the 10 mg/L nitrite solution to a clean 100 mL volumetric flask. Dilute to 100 mL with deionized water. This makes a 1 mg/L-NO_2 solution.

8.4.3 From the 1 mg/L nitrite solution, prepare a 0.10 mg/L nitrite solution. Using a volumetric pipette, transfer 10 mL of the 1 mg/L nitrite solution to a clean 100 mL volumetric flask. Dilute to 100 mL with deionized water. This makes a 0.10 mg/L-NO_2 solution.

8.4.4 Pour a small amount (3-5 mL) of the nitrate stock solution into a clean 50 mL beaker. Using a volumetric pipette, transfer exactly 1 mL of stock solution from the beaker to a clean 100 mL volumetric flask. Dilute to 100 mL with deionized water. This makes 10 mg/L-NO_3 solution.

8.4.5 From the 10 mg/L nitrate solution, prepare a 1 mg/L nitrate solution. Using a volumetric pipette, transfer 10 mL of the 10 mg/L nitrate solution to a clean 100 mL volumetric flask. Dilute to 100 mL with deionized water. This makes a 1 mg/L-NO_3 solution.

8.4.6 From the 1 mg/L nitrate solution, prepare the nitrate standards to be used for the test. For example: Pipette 1, 5, 10, and 15 mL into clean 100 mL volumetric flasks. Dilute to 100 mL, cap and mix thoroughly. These flasks will contain 0.01, 0.05, 0.10 and 0.15 mg/L-NO_3, respectively.

8.5 Sample pH adjustment- Adjust pH to between 7 and 9, as necessary, using a pH meter and dilute HCl or NaOH. This insures a pH of 8.5 after adding ammonium chloride- EDTA solution.

8.6 Prepare blanks, standards, spikes, and samples for addition of reagents. Rinse the graduated cylinder or pipette used to measure blanks, standards, spikes, and samples in between measurements with deionized water.

8.6.1 Blanks: Measure 15 mL of deionized water and transfer to labeled flask. Blanks should be run in duplicate. Prepare one nitrite blank.

8.6.2 Standards: Measure 15 mL of appropriate standard and transfer to labeled flasks. Standards should be run in duplicate.

8.6.3 Samples: Mix the sample and then measure and transfer 15 mL to labeled flasks. At least 30% of the water samples should be run in duplicate, the actual number will depend on how many samples are in the batch.

An aliquot portion of the sample diluted to 15 mL can be used if the sample is suspected to be higher than the prepared standards. Note any dilutions on laboratory bench sheet and laboratory notebooks.

8.6.4 Spike: Prepare a 0.05 mg/L spike. Rinse a 100 mL volumetric flask with a small amount of the sample to be used. Pipette 5 mL from the 1 mg/L nitrate solution into the 100 mL flask, and dilute to 100 mL with the sample. Cap and mix thoroughly. This results in a spike addition of 0.05 mg/L nitrate.

Measure 15 mL of the spike solution and transfer to the labeled flasks. Record on laboratory bench sheets and in laboratory notebooks the sample used for the spike solution.

8.7 Measure 45 mL of ammonium chloride-EDTA into each flask and mix. Use the bottle top dispenser to add ammonium chloride-EDTA.

8.8 Rinse reduction columns. Open the stopcock and drain down to where the column constricts, just above the cadmium granules. Do not expose the cadmium granules. Fill columns with dilute ammonium chloride-EDTA, and rinse once again. The columns are now ready to run samples.

8.9 Check the reduction efficiency of the cadmium column.

8.9.1 Using the procedure outlined in Step 8.10, run the nitrate reagent blanks through the columns first. Then follow instructions in Step 8.11 for both the nitrate and nitrite blanks.

8.9.2 Run the 0.10 mg/L nitrate standards through the columns, following the procedure in Step 8.10. Then follow instruction in Step 8.11 for both for both the nitrate and nitrite 0.10 mg/L standards.

8.9.3 Check the absorbance values of both the nitrite and nitrate reagent blanks against the acceptable values in the QA/QC binder. If they are within the "control limits" (and preferably within the "warning limits"), proceed to the next step. If they are not within the limits, empty the cadmium granules from the columns and prepare cadmium again according to section 8.2.

8.9.4 Compare the absorbance values of the 0.10 mg/L nitrate standard to the 0.10 mg/L nitrite standard. If the reduction efficiency is greater than 75%, proceed to the next step. If the reduction efficiency is less than 75%, empty the cadmium granules from the columns and prepare the cadmium again according to section 8.2.

Reduction efficiency % = (NO_3 absorbance / NO_2 absorbance) X 100

8.10 Run samples.

8.10.1 Pour the entire contents of a sample flask into a column and collect at a rate of 7 to 10 mL/minute.

8.10.2 Drain 25 mL into a waste beaker, and stop. Discard contents of beaker.

8.10.3 Place the sample flask under the stopcock, and open. Drain down to just above the cadmium granules, and stop.

8.10.4 There is no need to wash columns between samples, but if columns are not to be reused for several hours or longer, pour 50 mL dilute NH_4Cl-EDTA solution on to the top and let it pass through the system. Store Cu-Cd column in this solution and never let it dry.

8.10.5 Repeat steps 8.10.1 – 8.10.3 for all the samples, except the nitrite standard and nitrite blank, which should not be run through the column. Immediately begin step 8.11.

8.11 Prepare samples to read in the spectrophotometer.

8.11.1 After each sample has been run through a column, a 30 mL sample needs to be measured from each flask. Mix the sample by swirling the flask, pour 30 mL of sample into a graduated cylinder, discard whatever is left in the flask, then pour the contents of the cylinder back into the flask.

8.11.2 As soon as possible, and not more than 15 minutes after reduction, add 1.2 mL color reagent to 30 mL sample and mix.

8.12 Between 10 minutes and 2 hours afterward, measure absorbance using the spectrophotometer with the wavelength set at 543 nm.

8.12.1 If all samples are <0.20 mg/L, use the 10 cm cell. If all samples are >0.20 mg/L, use the 1 cm cell. If both cells need to be used, you will need to read at least one blank and three standards in each cell, and calculate separate standard curves.

8.13 Clean up. The cadmium granules can be re-used. Rinse the columns with dilute ammonium chloride-EDTA and drain to a level between the lower mark and he top of the cadmium granules. Then rinse the cadmium into a beaker with deionized water. Rinse the granules with 1:1 hydrochloric acid, and store in dilute ammonium chloride-EDTA.

9.0 Calculations

9.1 Prepare a standard curve by plotting absorbance readings of standards against nitrate concentrations of standards. Compute sample concentration by comparing sample absorbance with the standard curve.

9.2 Obtain concentration of value of sample directly from prepared standard curve. Report results as NO_3^--N + NO_2^--N, mg/L.

10.0 References

APHA, AWWA, WEF. *Standard Methods for the Examination of Water and Wastewater, 20th Edition* (4500-NO_3 E. Cadmium Reduction Method). American Public Health Association, Washington DC. 1998.

Standard Operating Procedure for:

Ammonia as Nitrogen Determination

1.0 Scope and Application

1.1 This method covers the determination of ammonia in drinking, ground, surface and saline waters, domestic and industrial wastes.

1.2 The applicable range is 0.01 – 0.60 mg NH_3-N/L. Higher concentrations can be determined by sample dilution.

2.0 Summary of Method

2.1 An intensely blue compound, indophenol, is formed by the reaction of ammonia, hypochlorite, and phenol catalyzed by sodium nitroprusside.

3.0 Interferences

3.1 Complexing magnesium and calcium with citrate eliminates interference produced by precipitation of these ions at high pH. There is no interference from other trivalent forms of nitrogen.

3.2 Remove interfering turbidity by distillation or filtration.

3.3 If hydrogen sulfide is present, remove by acidifying samples to pH 3 with dilute HCl and aerating vigorously until sulfide odor no longer can be detected.

4.0 Filtration

4.1 Filter samples immediately after collection, through a 0.45-µm membrane filter. Wash membrane filters by running 150-mL of ammonia-free water through them before contact with sample water. A glass fiber filter may be used to pre-filter hard-to-filter-samples.

5.0 Preservation and Storage

5.1 Once filtered pour samples into clean 250 mL polypropylene bottles labeled with the sample ID, sample date, preservation, and analytical parameter.

5.2 Most reliable results are obtained on fresh samples. If samples are to be analyzed within 24 hours of collection, refrigerate unacidified at 4°C. For preservation for up to 28 days, freeze at -20°C unacidified, or preserve samples by acidifying to pH <2 and storing at 4°C. If acid preservation is used, neutralize samples with NaOH or KOH immediately before making the determination.

6.0 Equipment

6.1 Spectrophotometer: Suitable for measurements at 640 nm with a light path of 1 cm or greater.

6.2 Acid-washed glassware: all glassware should be washed with 10% HCl and rinsed with ammonia-free water. This glassware should only be used for the determination of ammonia as nitrogen and after should be washed as stated above and kept covered until needed again.

A set of 125 mL Erlenmeyer flasks should be dedicated to the determination of ammonia as nitrogen.

6.3 Glassware: Class A volumetric flasks and pipettes as required.

6.4 Balance: Analytical, capable of accurately weighing to the nearest 0.001 g.

6.5 Eppendorf dispenser: Manual hand dispenser with 50 mL volume tips. For use when adding reagents to samples. Use a different tip for each reagent added.

7.0 Reagents

7.1 **Phenol solution:** Dissolve 10 g of reagent-grade phenol in 100 ml of 95% denatured ethanol. Prepare weekly. Caution: Wear gloves and eye protection when handling phenol; use good ventilation to minimize all personnel exposure to this toxic volatile substance.

7.2 **Sodium nitroprusside, 0.5% w/v:** Dissolve 0.5 g sodium nitroprusside in ammonia-free water, and dilute to 100 ml. Store in amber bottle for up to 1 month.

7.3 **Alkaline citrate solution:** Dissolve 200 g of trisodium citrate and 10 g of sodium hydroxide in deionized water. Dilute to 1000 mL.

7.4 **Sodium hypochlorite:** Commercial solution, about 5%. This solution slowly decomposes once the seal on the bottle cap is broken. Replace about every 2 months.

7.5 **Oxidizing solution:** Make a 4:1 ratio of alkaline solution to sodium hypochlorite. Make fresh daily. For 25 samples, add 30 mL sodium hypochlorite to 120 mL alkaline solution.

7.6 **Stock ammonium solution:** Dissolve 3.819 g anhydrous NH_4Cl (dried at 100°C) in water and dilute to 1000 mL. Salts to be used should be dried at 100°C for 24 hours before being weighed. Shelf life = Six months. 1.00 mL = 1.00 mg N = 1.22 mg NH_3.

7.7 **Standard ammonium solution:** Use stock ammonium solution and deionized water to prepare a calibration curve in a range appropriate for the concentrations of the samples. Prepare daily.

7.8 **5% HCl:** Dilute 50 mL of 1:1 HCl with ammonia-free water to 500 mL (1:1 HCl is equal volumes of HCl and water). Fill cylinder with 300 mL of ammonia-free water before adding acid.

7.9 **Ammonia-free water:** Prepare ammonia-free deionized water by passing distilled water (or previously deionized water) through the ion-exchange columns. Check ammonia-free water for the possibility of a high blank value. It is very hard to store ammonia-free water in the laboratory without contamination from gaseous ammonia. Use fresh (made within a few hours) ammonia-free water for testing procedures.

8.0 Procedure

8.1 Preparation

8.1.1 Retrieve the samples from refrigerator. The samples should be near room temperature when beginning the test.

8.1.2 Retrieve ammonia stock solution from refrigerator. Allow time to reach room temperature.

8.1.3 Retrieve all reagents needed. Prepare reagents before starting test if needed.

8.1.4 Retrieve the dedicated set of glassware (125 mL Erlenmeyer flasks). Rinse all glassware used for the test with 5% HCl and three times with ammonia-free water.

8.1.5 All 125 mL Erlenmeyer flasks dedicated for the ammonia determination should be labeled to avoid confusion between blanks, standards, spikes, and samples.

8.2 Prepare standards to be used for standard curve. The standards should bracket the sample concentrations. Use a minimum of three standards in addition to a reagent blank (ammonia-free water).

8.2.1 Pour a small amount (3 - 5 mL) of the ammonia stock solution into a clean 50 mL beaker. Using a volumetric pipette, transfer exactly 1 mL of stock solution from the beaker to a clean 100 mL volumetric flask. Dilute to 100 mL with ammonia-free water. Place a cap on the flask and mix thoroughly by inverting at least 5 times. This solution is 10 mg/L ammonia.

8.2.2 Using the 10 mg/L ammonia solution, transfer exactly 10 mL to a clean 100 mL volumetric flask. Dilute to 100 mL with ammonia-free water. Place a cap on the flask and mix thoroughly by inverting at least 5 times. This solution is 1 mg/L ammonia.

8.2.3 Using the 1 mg/L ammonia solution, prepare the standards to be used for the test. For example: Pipette exactly 1, 3, 8 and 10 mL into separate clean 100 mL volumetric flasks. Dilute to 100 mL, cap and mix thoroughly. These flasks will contain 0.01, 0.03, 0.08 and 0.10 mg/L ammonia, respectively.

8.3 Prepare blanks, standards, spikes, and samples for addition of reagents. Rinse the graduated cylinder or pipette used to measure blanks, standards, spikes, and samples in between measurements with deionized water.

8.3.1 Blanks: Measure 50 mL of deionized water and transfer to labeled flask. Blanks should be run in duplicate.

8.3.2 Standards: Measure 50 mL of appropriate standard and transfer to labeled flasks. Standards should be run in duplicate.

8.3.3 Samples: Mix the sample and then measure and transfer 50 mL to labeled flasks. At least 30% of the water samples should be run in duplicate, the actual number will depend on how many samples are in the batch.

An aliquot portion of the sample diluted to 50 mL can be used if the sample is suspected to be higher than the prepared standards. Note any dilutions on laboratory bench sheet and laboratory notebooks.

8.3.4 Spike: Prepare a 0.05 mg/L spike. Rinse a 100 mL volumetric flask with a small amount of the sample to be used. Pipette 5 mL from the 1 mg/L ammonia solution into the 100 mL flask, and dilute to 100 mL with the sample. Cap and mix thoroughly. This results in a spike addition of 0.05 mg/L ammonia.

Measure 50 mL of the spike solution and transfer to the labeled flasks. Record on laboratory bench sheets and in laboratory notebooks the sample used for the spike solution.

8.4 Add 2 mL of phenol solution to each flask and mix.

8.5 Add 2 mL of sodium nitroprusside solution and mix.

8.6 Add 5 mL of oxidizing solution and mix.

8.7 Replace flask stoppers. Let color develop at room temperature (22 to 27°C) in subdued light (for best results keep samples in the dark by covering with an opaque cover) for at least 1 hour. Color is stable for 24 hours.

8.8 Measure absorbance of the samples in the spectrophotometer with the wavelength set at 640 nm.

9.0 Calculations

9.1 Prepare a standard curve by plotting absorbance readings of standards against ammonia concentrations of standards. Compute sample concentration by comparing sample absorbance with the standard curve.

9.2 Obtain concentration of value of sample directly from prepared standard curve. Report results as NH_3-N, mg/L.

10.0 References

APHA, AWWA, WEF. *Standard Methods for the Examination of Water and Wastewater, 20th Edition* (4500-NH_3 F. Phenate Method). American Public Health Association, Washington DC. 1998.

Standard Operating Procedure for:

Total Kjeldahl Nitrogen (TKN) Determination

1.0 Scope and Application

1.1 This method determines nitrogen in the trinegative state. The method fails to account for nitrogen in the form of azide, azine, azo, hydrazone, nitrate, nitrite, nitrile, nitro, nitroso, oxime, and semi-carbazone. "Kjeldahl nitrogen" is the sum of organic nitrogen and ammonia nitrogen.

1.2 The applicable range is 0.2 to 2 mg TKN-N/L.

2.0 Summary of Method

2.1 In the presence of H_2SO_4, potassium sulfate (K_2SO_4) , and cupric sulfate ($CuSO_4$) catalyst, amino nitrogen of many organic materials is converted to ammonium. Free ammonia also is converted to ammonium. After addition of base, the ammonia is distilled from an alkaline medium and absorbed in boric or sulfuric acid. The ammonia then may be determined colorimetrically.

3.0 Interferences

3.1 Nitrate: During Kjeldahl digestion, nitrate in excess of 10 mg/L can oxidize a portion of the ammonia released from the digested organic nitrogen, producing N_2O and resulting in a negative interference. When sufficient organic matter in a low state of oxidation is present, nitrate can be reduced to ammonia, resulting in a positive interference in conjunction with the kjeldahl methods described herein.

3.2 Inorganic salts and solids: The acid and salt content of the kjeldahl digestion reagent is intended to produce a digestion temperature of about 380°. If the sample contains a very large quantity of salt or inorganic solids that dissolve during digestion, the temperature may rise above 400°C, at which point pyrolytic loss of nitrogen begins to occur. To prevent an excessive digestion temperature, add more H_2SO_4 to maintain the acid-salt balance. Not all salts cause precisely the same temperature rise, but adding 4mL H_2SO_4/g salt in the sample gives reasonable results. Add the extra acid and the digestion reagent to both sample and reagent blank. Too much acid will lower the digestion temperature below 380°C and result in incomplete digestion and recovery. If necessary, add sodium hydroxide-sodium thiosulfate before the final distillation step to neutralize the excess acid.

Large amounts of salt or solids also may cause bumping during distillation. If this occurs, add more dilution water after digestion.

3.3 Organic matter: During kjeldahl digestion, H_2SO_4 oxidizes organic matter to CO_2 and H_2O. If a large amount of organic matter is present, a large amount of acid will be consumed, the ratio of salt to acid will increase, and the digestion temperature will increase. If enough organic matter is present, the temperature will rise above 400°C, resulting in pyrolytic loss of nitrogen. To prevent this, add to the digestion flask 10 mL concentrated H_2SO_4/3 g COD. Alternately, add 50 mL more digestion reagent/g COD. Additional sodium hydroxide-sodium thiosulfate reagent may

be necessary to keep the distillation pH high. Because reagents may contain traces of ammonia, treat the reagent blank identically with the samples.

4.0 Filtration

4.1 Do not filter samples.

5.0 Preservation and Storage

5.1 Pour samples into clean 250 mL polypropylene bottles labeled with the sample ID, sample date, preservation, and analytical parameter.

5.2 Most reliable results are obtained on fresh samples. If immediate analysis is not possible, preserve samples for kjeldahl digestion by acidifying to pH 1.5 to 2.0 with concentrated H_2SO_4 and storing at 4°C.

6.0 Equipment

6.1 Digestion apparatus: Labconco 25-place rapid digestor with Kjeldahl flasks with a capacity of 300 mL.

6.2 Distillation apparatus: Buchi 315 distillation unit.

6.3 Spectrophotometer: Suitable for measurements at 640 nm with a light path of 1 cm or greater.

6.4 Acid-washed glassware: all glassware should be washed with 10% HCl and rinsed with deionized water. This glassware should only be used for the determination of ammonia as nitrogen and after should be washed as stated above and kept covered until needed again.

A set of 125 mL Erlenmeyer flasks should be dedicated to the determination of TKN as nitrogen.

6.5 Glassware: Class A volumetric flasks and pipets as required.

6.6 Balance: Analytical, capable of accurately weighing to the nearest 0.001 g.

6.7 Eppendorf dispenser: Manual hand dispenser with 50 mL volume tips. For use when adding reagents to samples. Use a different tip for each reagent added.

7.0 Reagents

7.1 **Ammonia-free water:** Prepare all reagents and dilutions in ammonia-free water. Cap storage container tightly, and do not store for more than one day. Prepare ammonia-free water by passing distilled water through and ion-exchange columns. Check ammonia-free water for the possibility of a high blank value. It is very hard to store ammonia-free water in the laboratory without contamination from gaseous ammonia. Use fresh ammonia-free water for testing procedures.

7.2 **TKN digestion reagent:** Dissolve 134 g K_2SO_4 in 650 mL ammonia-free water. Slowly add 200 mL of concentrated sulfuric acid. Dilute to 1 liter with ammonia-free water (this produces a salt to acid ration of 0.67:1). Store above 20°C to prevent re-crystallization of K_2SO_4 – if crystals form, dissolve as much as possible before using.

7.3 **Copper sulfate catalyst:** Dissolve 25.115 g anhydrous copper sulfate in ammonia free water and dilute to 1 liter.

7.4 **$Na_2B_4O_7 \cdot 10H_2O$:** Dissolve 66 g of $Na_2B_4O_7 \cdot 10\ H_2O$ in 3 liters of ammonia-free water.

7.5 **Sodium hydroxide solution:** Dissolve 500 g of NaOH flakes or pellets in water and dilute to 1 liter. Heat is generated in this reaction. Fill the flask with about 700 mL and add the NaOH slowly. Put the beaker in a waterbath on the stir plate to help keep it cool.

7.6 **Phenol solution:** Dissolve 10 g of reagent-grade phenol in 100 ml of 95% denatured ethanol. Prepare weekly. Caution: Wear gloves and eye protection when handling phenol; use good ventilation to minimize all personnel exposure to this toxic volatile substance.

7.7 **Sodium nitroprusside, 0.5% w/v:** Dissolve 0.5 g sodium nitroprusside in ammonia-free water, and dilute to 100 ml. Store in amber bottle for up to 1 month.

7.8 **Alkaline citrate solution:** Dissolve 200 g of trisodium citrate and 10 g of sodium hydroxide in deionized water. Dilute to 1000 mL.

7.9 **Sodium hypochlorite:** Commercial solution, about 5%. This solution slowly decomposes once the seal on the bottle cap is broken. Replace about every 2 months.

7.10 **Oxidizing solution:** Make a 4:1 ratio of alkaline solution to sodium hypochlorite. Make fresh daily. For 25 samples, add 30 mL sodium hypochlorite to 120 mL alkaline solution.

7.11 **Stock ammonium solution:** Dissolve 3.819 g anhydrous NH_4Cl (dried at 100°C) in water and dilute to 1000 mL. Salts to be used should be dried at 100°C for 24 hours before being weighed. Shelf life = Six months. 1.00 mL = 1.00 mg N = 1.22 mg NH_3.

7.12 **Standard ammonium solution:** Use stock ammonium solution and deionized water to prepare a calibration curve in a range appropriate for the concentrations of the samples.

7.13 **5% HCl:** Dilute 50 mL of 1:1 HCl with ammonia-free water to 500 mL (1:1 HCl is equal volumes of HCl and water). Fill cylinder with 300 mL of ammonia-free water before adding acid.

8.0 Procedure

8.1 Preparation

8.1.1 Retrieve the samples from refrigerator. The samples should be near room temperature when beginning the test.

8.1.2 Retrieve ammonia stock solution from refrigerator. Allow time to reach room temperature.

8.1.3 Retrieve all reagents needed. Prepare reagents before starting test if needed.

8.1.4 Retrieve the dedicated set of glassware (125 mL Erlenmeyer flasks). Rinse all glassware used for the test with 5% HCl and three times with ammonia-free water.

8.1.5 All 125 mL Erlenmeyer flasks dedicated for the ammonia determination should be labeled to avoid confusion between blanks, standards, spikes, and samples.

8.1.6 Check the steam generator water supply. The square jug on top of the distiller should be filled with ammonia-free water and $Na_2B_4O_7 \cdot 10H_2O$ of solution. The purpose of the $Na_2B_4O_7 \cdot 10H_2O$ is to raise the conductance of the generator water so it will boil. Add to the square jug in a ratio of 40 mL $Na_2B_4O_7 \cdot 10H_2O$ solution for every one liter of ammonia-free water. The jug should be filled with 10-15 liters to run 25 samples.

8.1.7 Fill smaller reservoir on the lower right side of the distiller with sodium hydroxide solution.

8.1.8 Turn on the block rapid digestor. Set temperature to 360°C.

8.2 Prepare standards to be used for standard curve. The standards should bracket the sample concentrations. Use a minimum of three standards in addition to a reagent blank (ammonia-free water). Standards should be 0.2, 0.5, 1.0, and 1.5 mg/L ammonia.

8.2.1 Pour a small amount (3-5 mL) of the ammonia stock solution into a clean 50 mL beaker. Using a volumetric pipet, transfer exactly 1 mL of stock solution from the beaker to a clean 100 mL volumetric flask. Dilute to 100 mL with ammonia-free water. Place a cap on the flask and mix thoroughly by inverting at least 5 times. The solution is now 10 mg NH_3-N/L.

8.2.2 Using the 10 mg/L ammonia solution, prepare the standards to be used for the test. Pipet exactly 2, 5, 10, and 15 mL into separate clean 100 mL volumetric flasks. Dilute to 100 mL, cap and mix thoroughly. These flasks will contain 0.2, 0.5, 1.0, and 1.5 mg NH_3-N/L, respectively.

8.3 Prepare a reagent blank, spike and glycine pTSA standard.

8.3.1 Blank: Fill a 100 mL volumetric flask with ammonia-free water.

8.3.2 Spike: Prepare a 0.5 mg/L spike. Rinse a 100 mL volumetric flask with a small amount of the sample to be used for the spike. Pipet 5 mL from the 10 mg/L ammonia solution into the 100 mL flask, and dilute to 100 mL with the sample. Cap and mix thoroughly. This results in a spike addition of 0.5 mg/L ammonia. Record which sample was used for the spike solution on laboratory bench sheets and in laboratory notebooks.

8.3.3 Glycine pTSA standard: Pipet 10 mL of glycine pTSA into a 100 mL volumetric flask and dilute to 100 mL with ammonia-free water. Cap and mix thoroughly. Final ammonia concentration of this standard is 0.8 mg/L.

8.4 Measure standards and samples into digestion tubes. Tubes should be pre-rinsed with ammonia-free water and allowed to drain beforehand. Rinse the graduated cylinder or pipette used to measure blanks, standards, spikes, and samples in between measurements with ammonia-free water.

The metal frame that holds the digestion tubes is labeled A - F along one side and 1 - 5 along another, so that any position in the block can be referenced, for example A1, C2, or D5. Arrange standards in the order in which they will be placed into the metal frame. Start with the blanks in cells A1 and A2, the 0.5 standard in cell A3 and A4, and progress through the standards in an orderly fashion. Write down what sample each frame position contains on the backside of the laboratory bench sheet.

8.4.1 Blanks: Measure 50 mL of the reagent blank and transfer to a digestion tube and place in metal frame. Blanks should be run in duplicate.

8.4.2 Standards: Measure 50 mL of each standard and transfer to digestion tubes. Standards should be run in duplicate, except for the glycine pTSA standard.

8.4.3 Samples: Mix the sample and then measure and transfer 50 mL to digestion tubes. At least 30% of the water samples should be run in duplicate, the actual number will depend on how many samples are in the batch.

An aliquot portion of the sample diluted to 50 mL can be used if the sample is suspected to be higher than the prepared standards. Note any dilutions on laboratory bench sheet and laboratory notebooks.

8.4.4 Spike: Measure 50 mL of the spike solution and transfer to the labeled flasks. Record which sample was used for the spike solution on laboratory bench sheets and in laboratory notebooks.

8.5 Add digestion reagents to each of the tubes. An Eppendorf dispenser can be used to measure and dispense reagents. When dispensing reagent the “first shot” should be wasted to ensure that each shot afterward is the proper volume.

8.5.1 Add 2 mL of copper sulfate catalyst to each tube.

8.5.2 Add 15 mL of TKN digestion reagent to each tube.

8.6 Add a “pinch” of Teflon boiling chips to each tube (4-8 chips). Inspect tubes to make sure they all have sample, reagent and boiling chips added.

8.7 Digest samples in digestion apparatus. If samples dry out before digestion is complete during steps below, nitrogen will be lost (this requires some idea beforehand of how much nitrogen may be in the sample). Samples with high organic content may have to be diluted prior to

digestion; or additional sulfuric acid can be added to the tube. If extra acid is used, the amount of NaOH reagent added during Step 8.10.2 must be adjusted, because the NaOH reagent to total sulfuric acid ration must be 5:1. For example, 15 mL of TKN digestion reagent contains 3 mL of sulfuric acid, so 15 mL of NaOH is added in Step 8.10.2. Also, if an additional 1 mL of sulfuric acid is added to samples, it must also be added to all standards, blanks, spikes, etc. For each 1 mL of additional sulfuric acid added now, 5 mL of additional NaOH reagent must be added in Step 8.10.2, prior to distillation.

8.7.1 Place the metal side plates on the metal frame, and shake the frame (side to side) to that the contents of each tube become mixed.

8.7.2 Turn on the vent fans in the fume hood.

8.7.3 Place the tubes in the heating block. The metal frame sites on top of the hearing block. Record the time on the laboratory bench sheet.

8.7.4 The tubes need to heat until fumes are visible. This normally takes about one hour. First, the water heats, and steam will be visible. After the water steams off, acid will remain. The acid will then begin to fume (note: acid fumes are whiter than the steam).

8.7.5 Once observing acid fumes record the time on the laboratory bench sheet. The samples need to digest for an additional 30 minutes after acid fumes are observed.

8.7.6 After 30 minutes, remove the metal frame from the heater block, remove the metal side plates, and set the frame down in another part of the vent hood to cool. Do not breathe the fumes. The heating block now can be turned off.

8.7.7 Let the tubes cool for 7 minutes. Then begin adding ammonia-free water according to the directions in section 8.8.1. If the cooling time is too short, samples will splatter when water is added. If cooling time is too long, samples will solidify when water is added.

8.8 Prepare samples to run through the distillation unit.

8.8.1 After the 7 minute cooling period, add about 30 mL of ammonia-free water to each tube and mix. Some samples may solidify if so follow the steps below.

8.8.1.1. If the precipitate in the tube is soluble (dissolves when you add water), the sample is ready to be distilled.

8.8.1.2. If the precipitate is not soluble, put the tube back in the heating block, which should still be quite hot. Let it heat to boiling, and mix until the precipitate is dissolved. Let cool until no steam is visible. It is then ready for distillation.

8.9 Prepare distillation unit and the 250 mL Erlenmeyer receiving flasks.

8.9.1 Rinse the 250 mL Erlenmeyer receiving flasks to be used for samples; once with 5% HCl, then twice with ammonia-free water.

8.9.2 Add 5 mL of 0.04N sulfuric acid to each flask and put in rubber stoppers.

8.9.3 Loosen the lid on the square jug on top of the distillation unit. Hook up the jug to the steam generator, and open the valve on the jug. The clamp on the hose should be adjusted to minimize overflow from the steam generator, and to keep the distillate temperature <29°C.

8.9.4 Close the glass valve located at the bottom of the opening on the left side of the unit. This will fill the generator. Fill once, drain and then refill.

8.9.5 Turn the cooling water on. The valve is located on the wall behind and to the left of the distillation unit. There are two black-handled valves; the cooling water is the one on the right.

8.9.6 Turn the distillation unit on. This is the red power switch on the upper left corner of the front panel. The air pump inside will begin to hum. If the power fails to come on, check the fuse directly below the power switch. Replace if necessary. Instruction manual, spare parts, and fuses are located in the drawer below the distillation unit.

8.9.7 Put about 15 mL of ammonia-free water in a tube and place the tube in the tube holder on the distillation unit. Twisting the tube slightly against the rubber stopper ensures a good seal. Place a 500 mL beaker under the white plastic tube to the right of the digestion tube holder, on top of the large rubber stopper (the rubber stopper acts as a spacer to make moving the beaker and flasks easier).

8.9.8 Add 15 mL of NaOH to the digestion tube. A chart on the wall near the generator describes how the valve system works. To add 15 mL, operate the hydroxide valve (top valve) while observing the level of liquid in the tube against the orange tape on the plate behind the tube; each mark on the tape equals 5 mL.

8.9.9 Turn the steam on by opening the middle valve, and close the drain by closing the bottom valve. Allow to boil until there is about 100 mL of liquid in the 500 mL beaker. Then turn the steam off by closing the middle valve. After the distillation unit sucks the sample out of the tube, open the drain (bottom valve).

8.9.10 Repeat steps 8.9.7 – 8.9.9 two more times. The steam generator is now ready to run samples.

8.9.10.1. If necessary adjust the flow on the steam generator supply line such that the steam generator is kept just full, not too much overflow, and the receiving solution temperature remains 29°C.

8.10 Run all samples through the distillation unit.

8.10.1 Select a tube to run through the unit. Unstopper the corresponding 250 mL Erlenmeyer flaks and place it on top of the large rubber stopper, so that the white

plastic tube is submerged in the acid. Then put the digestion tube in the digestion tube holder, a slight twist will help ensure a good seal.

8.10.2 Add 15 mL of NaOH (three marks on the tape behind the tube: see chart for proper valve operation). Don't forget to add an additional 5 mL of NaOH for each additional 1 mL of sulfuric acid that might have been added during Step 8.7.

8.10.3 Turn steam on and immediately close drain.

8.10.4 When the Erlenmeyer flask has been filled to the 75 mL mark, remove the large rubber stopper and the flask. Immediately replace it with the 500 mL beaker. Keep the steam on.

Note: The distiller temperature should remain <29°C. If it is too hot, tighten the clamp on the tube from the square jug on top of the unit.

8.10.5 Pour the contents of the Erlenmeyer flask into a 100 mL graduated cylinder. Fill to 100 mL with ammonia-free water, then pour back into the flask and mix (the flask now contains 100 mL of sample).

8.10.6 Now measure out 50 mL of sample back into the graduated cylinder. Discard what is left in the flask. Then pour the contents of the graduated cylinder back into the flask and stopper. Set aside.

8.10.7 When the 500 mL beaker has filled to about 75 mL, turn the steam off. This will cause the remaining contents in the digestion tube to be sucked up into the distillation unit.

Note: If the condenser tube is below the distillate level when the steam is turned off, the distillate will be sucked up into the condenser. Lower the beaker and open the drain valve to drain it back out.

8.10.8 When the contents of the distillation tube have all been sucked up into the distillation unit, open the drain.

8.10.9 Using thick black rubber gloves unseat the digestion tube. Set it down beside the holder, so that the long white tube is still in it.

8.10.10 Open the steam valve for 2 or 3 seconds. This drains the steam generator.

8.10.11 Using the glove remove the digestion tube. Rinse it out twice (inside and out) with tepid tapwater, then once with the water in the 500 mL beaker and then a final rinse with ammonia-free water.

8.10.12 Repeat steps 8.10.1 – 8.10.11 until all samples have been run.

8.10.13 Clean the steam generator by repeating steps 8.10.7 – 8.10.10.

8.10.14 Turn the power switch off. Open the glass valve to drain the steam generator. Close the cooling water valve. Close the square jug valve and disconnect the hose. Turn the jug around on top of the unit so that the valve is over the sink.

8.10.15 Partially fill a digestion tube with ammonia-free water and place it in the holder, so that the end of the tube is submerged. Also partially fill the 500 mL beaker with water, and place it on the large rubber stopper so that the white tube is submerged. Make sure the NaOH valve and steam valve are closed, and the drain valve is open.

8.11 Prepare samples to read in spectrophotometer.

8.11.1 Add 2 mL of phenol solution to each flask and mix.

8.11.2 Add 2 mL of sodium nitroprusside solution and mix.

8.11.3 Add 5 mL of oxidizing solution and mix.

8.11.4 Replace flask stoppers. Let color develop at room temperature (22 to 27°C) in subdued light for at least 1 hour. Color is stable for 24 hours.

8.11.5 Measure absorbance of the samples in the spectrophotometer with the wavelength set at 640 nm.

9.0 Additional Information

9.1 The boiling point of fuming sulfuric acid is 330°C. The K_2SO_4 increases the boiling point, thereby reducing digestion time.

9.2 Acid is consumed during the digestion process. If the sulfate: acid ratio exceeds 0.8 mg/L, the mixture will solidify upon cooling. Place the tube back in the digester until the solid is dissolved. If all acid is consumed (i.e., the sample dries during digestion), ammonia will be lost and the sample must be run again.

9.3 If the sulfate: acid ratio exceeds 1.3 mg/L, the digestion temperature will exceed 400°C and ammonia may be lost by volatilization.

9.4 The catalyst speeds the reaction between sulfuric acid and the sample. When copper sulfate ($CuSO_4$) is used instead of HgO there is no need to add sodium thiosulfate to the NaOH reagent.

9.5 During digestion, the sample will turn black and large amounts of sulfur dioxide fumes will be produced (be careful not to breathe these fumes; they are extremely irritating); then the sample will turn “clear”. Ideally, digestion should continue for an additional length of time equal to the length of time that the sample took to reach the clearing phase.

10.0 Calculations

10.1 Prepare a standard curve by plotting absorbance readings of standards against ammonia concentrations of standards. Compute sample concentration by comparing sample absorbance with the standard curve.

10.2 Obtain concentration of value of sample directly from prepared standard curve. Report results as TKN-N, mg/L.

11.0 References

APHA, AWWA, WEF. *Standard Methods for the Examination of Water and Wastewater, 20th Edition* (4500-N_{org} C. Semi-Micro-Kjeldahl Method). American Public Health Association, Washington DC. 1998.

Standard Operating Procedure for:

Zooplankton Sample Analysis

8.0 Scope and Purpose

8.1 This SOP applies to the analysis of zooplankton samples collected from Pyramid Lake.

9.0 Sample Preservation and Storage

9.1 Add 3-5 mL of Lugols solution to preserve the sample.

9.2 Store sample in refrigerator until analysis.

10.0 Equipment

10.1 Bottom of Zooplakton net- collection chamber only.

10.2 600 mL beaker.

10.3 1 – 2 mL Hensen-Stempel pipette.

10.4 Sedgewick-Rafter cell.

10.5 Clear glass slides.

10.6 Lab counter with 9 counting keys, pre-labeled counter with expected species.

10.7 Squirt bottle filled with deionized water.

10.8 Microscope.

10.9 Identification key.

11.0 Procedure

11.1 Take samples out of refrigerator.

11.2 Pour sample into zooplankton collection chamber, with 240 µm mesh size. Take care to keep bottom clamp closed on the collection chamber so none of the zooplankton sample is escapes.

11.3 Using the squirt bottle rinse the zooplankton collection chamber to rinse preservative from the sample.

11.4 Unclamp the bottom of the zooplankton collection chamber and rinse the zooplankton sample into a 600 mL beaker. Dilute to 300 mL with deionized water.

11.5 Stir sample to mix evenly and use the Hensen-Stempel pipette to extract a 1 mL portion of the sample.

11.6 Place the extracted portion of the sample onto a Sedgewick-Rafter cell and cover with a clear glass slide.

11.7 Place slide on microscope.

11.8 Identify specimens on the slide using the identification key, as zooplankton are identified use the lab counter labeled with expected species to count each zooplankton.

11.9 Count using a grid pattern (left to right, back and forth) until all zooplankton on slide are identified and counted.

11.10 Do 5 repetitions for each sample (steps 4.5 – 4.9).

11.11 Record the final counts on a zooplankton bench sheet.

11.12 Enter final counts into the excel spreadsheet on the PLF server, the path is: My Computer/Resource/Water Quality/PLWQ/Zooplankton Calculation Sheets/PLFZoopCalcForm.xls.

11.13 The form will automatically do the calculations. Enter all information including dates, sample station, etc. Print a copy when done to file.