

Appendix B

A1. TITLE AND APPROVAL SHEETS

Quality Assurance Project Plan

for

Upper Owens River Water Quality Project
(Mammoth Lakes Basin Nonpoint Source Project)

May, 2012



California Trout

QAPP Revision Number: 0.0

Group A: Project Management

APPROVAL SIGNATURES

GRANT ORGANIZATION:

<u>Title:</u>	<u>Name:</u>	<u>Signature:</u>	<u>Date*:</u>
CalTrout Project Director	W. Mark Drew, Ph.D.		5/9/2012
CalTrout QA Officer	W. Mark Drew, Ph.D.		5/9/2012

STATE WATER RESOURCES CONTROL BOARD:

<u>Title:</u>	<u>Name:</u>	<u>Signature:</u>	<u>Date*:</u>
Grant Manager	Cindy Wise		
QA Officer			

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Quality Assurance Project Plan
 Upper Owens River Water Quality Project
 (Mammoth Lakes Basin NPS Project)
 Date:

THE FOLLOWING QUALITY ASSURANCE PROJECT PLAN (QAPP) IS BASED ON THE 2008 SWAMP QUALITY ASSURANCE PROGRAM PLAN (QAPRP) FOR THE STATE OF CALIFORNIA'S SURFACE WATER AMBIENT MONITORING PROGRAM (SWAMP) [HTTP://WWW.WATERBOARDS.CA.GOV/WATER_ISSUES/PROGRAMS/SWAMP/DOCS/QAPP/SWAMP_QAPP_MASTER090108A.PDF](http://www.waterboards.ca.gov/water_issues/programs/swamp/docs/qapp/swamp_qapp_master090108a.pdf), AND WAS CREATED USING THE ELECTRONIC TEMPLATE FOR SWAMP- COMPARABLE QUALITY ASSURANCE PROJECT PLANS ACCESSED AT [HTTP://WWW.WATERBOARDS.CA.GOV/WATER_ISSUES/PROGRAMS/SWAMP/QAPP.SHTML](http://www.waterboards.ca.gov/water_issues/programs/swamp/qapp.shtml) WHICH IS IN ACCORDANCE WITH THE U.S. EPA QAPP GUIDANCE, EPA AQ/R5, 3/01.

DISTRIBUTION LIST

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Moss Landing Marine Laboratory Marine Pollution Studies Laboratory (MLML-MPSL) Project Manager	Autumn Bonnema	831-771-4175	1
Mammoth Community Water Department (MCWD) Laboratory, Laboratory Director	Blair Hafner	760-934-2596 ext. 249	1

A4. PROJECT/TASK ORGANIZATION

A4.1 Involved parties and roles.

California Trout, Inc. (CalTrout) is a non-profit organization with a mission to protect and restore wild trout and steelhead salmon and their waters throughout California. As such, CalTrout is interested in monitoring and maintaining or improving the quality of the water in the Upper Owens River Watershed, including the Mammoth Lakes and Hot Creek Basins, and has entered into a California Nonpoint Source (NPS) Pollution Control Program, Federal Clean Water Act Section 319(h) grant agreement with the State Water Resources Control Board (SWRCB) (Agreement Number: 11-108-556) to carry out a project entitled, Upper Owens River Water Quality Project, which shall henceforth be referred to in this document as the Mammoth Lakes Basin Nonpoint Source (NPS) Project or MLB-NPS Project or Project.

CalTrout will play the lead role in general project administration and management, including quarterly and final fiscal and performance reporting, managing day-to-day operations and activities, and assuring compliance with applicable state requirements. CalTrout will work with contractors from American Rivers, the Owens Valley Indian Water Commission, and the Inyo-Mono Integrated Regional Water Management Program to accomplish this project and will convene and manage a Technical Advisory Committee (TAC) to consult with advisors from the Inyo National Forest, Town of Mammoth Lakes, Mammoth Community Water District, and Sierra Nevada Aquatic Research Laboratory. Figure 1 is a project organizational flow chart listing key members of the project team. Table 1 lists the people involved with this project including personnel and members of the TAC.

Grant Manager (SWRCB). The SWRCB Grant Manager (GM) will act as the SWRCB point of contact for this project. The GM will assure that QAPP development is coordinated between the SWRCB and CalTrout). The GM will also assure that the nonpoint source project is completed in accordance with the terms and conditions of the funding agreement between the SWRCB and CalTrout.

Quality Assurance Manager (SWRCB). The SWRCB Quality Assurance Manager (QAM) has overall responsibility for the review and approval of the QAPP. The QAM reviews the laboratory procedures to be followed. The QAM reviews the procedures and QA Plans of commercial laboratories employed for the project through the QAPP approval process. The QA Office will then provide assessment of the implementation of the laboratory's QA program, including review of analytical data, corrective actions, etc., as requested by the SWRCB Grant Manager.

Project Director (CalTrout). W. Mark Drew, Ph.D. is CalTrout's Eastern Sierra Program Manager and is the Project Director (PD) for the Mammoth Lakes Basin NPS Project. The CalTrout PD has overall responsibility for all phases of the MBL-NPS Project, with oversight by the SWRCB. The PD will ensure that project participation, lines of communication, data quality objectives, field activities, interactions with the contract laboratories, data management, quality assurance, documentation, and reporting are fulfilled in accordance with this project plan. Together with the Water Quality Specialist the CalTrout PD will define project Quality Assurance/Quality Control (QA/QC) objectives and establish project policy and procedures to address the specific objectives of each task and the project as a whole; apply the technical resources necessary to ensure QA/QC performance within budget and schedule constraints; orient the field technicians and support staff concerning the project's special considerations; may take part in field activities; review the work performed on each task to ensure its quality, responsiveness, and timeliness; ensure that all personnel are aware of project QA/QC objectives; ensure that chain-of-custody requirements are met; prepare and assure the quality of all external QA/QC reports prior to their submission to EPA.

Field Personnel:

Field Manager/Water Quality Specialist/ (CalTrout Contractor). Darla Heil is an Environmental Specialist with the Owens Valley Indian Water Commission (Commission) which is contracting her services to CalTrout to serve as Field Manager (FM) and Water Quality Specialist (WQS) for this project. The Commission is also contracted by CalTrout to provide a multiparameter water quality Sonde to obtain the field measurements that will be recorded as a part of this project. The FM/WQS has overall responsibility for overseeing and/or carrying out the field sampling aspects of the project so that the field activities are accomplished in accordance with this QAPP, including water quality sample collection, proper sample handling and shipment within sample holding times, bottle ordering and coordination with contracting analytical laboratories, and chain-of-custody preparation. The FM/WQS will direct and monitor field activities and identify problems at the field level and discuss resolutions with the CalTrout Program Director, the Field Technician, the CalTrout QA Officer, or the SWRCB. The FM/WQS will also oversee and or carry out the maintenance, calibration, and operation of the multiparameter Sonde in accordance with the procedures outlined in this document. FM/WQS oversight will assure that the sampling quality assurance and quality control procedures of this project plan are implemented satisfactorily.

Field Technician/Data Manager (CalTrout Contractor). Janet Hatfield is the Program Assistant for the Inyo-Mono Integrated Regional Water Management Program (IRWMP) and is contracting with CalTrout to serve as Field Technician (FT) and Data Manager for the Mammoth Lakes Basin NPS Project. The FT will carry out the field sampling aspects of this project along with the FM following the procedures included in this QAPP, assuring that the sampling quality assurance and quality control procedures of this project plan are implemented satisfactorily including water quality sample collection, proper sample handling and shipment within sample holding times, bottle ordering and coordination with contracting analytical laboratories, and chain-of-custody preparation.

Analytical Laboratory Activities

Nutrients: The California Department of Fish and Game-Water Pollution Control Laboratory (CDFG-WPCL) is located at 2005 Nimbus Road, Rancho Cordova, CA 95670. The CDFG-WPCL will analyze the nutrient parameters including total nitrogen, nitrate + nitrite, and total phosphorus for the Mammoth Lakes Basin NPS Project. The project contact at CDFG-WPCL is Patricia Bucknell, Project Manager.

Trace Metals: The Moss Landing Marine Laboratory-Marine Pollution Studies Laboratory (MLML-MPSL) is located at 7544 Sandholdt Road, Moss Landing, CA 95039. The MLML-MPSL will analyze the trace metals parameters for the project including total mercury in water and total manganese. The project contact at MLML-MPSL is Autumn Bonnema, Program Manager.

Solids: The solids parameters for the Mammoth Lakes Basin NPS Project, including total dissolved solids (TDS) and total suspended solids (TSS), will be analyzed locally by the Mammoth Community Water District (MCWD) Laboratory, which is located at 1315 Meridian Boulevard, Mammoth Lakes, CA 93546. The MCWD Laboratory contact is Blair Hafner, Lab Director.

The CDFG-WPCL and MCWD Laboratory are certified analytical laboratories, and the MLML-MPSL is approved by the SWRCB for the analyses to be performed through this project; data quality assessments will be conducted by each lab according to the methods defined in each laboratory's Quality Assurance Project Plan and/or in their QA Manuals (see Appendix B). The analytical laboratories will supply the project field staff with appropriate sampling kits for each set of parameters which will include: coolers; sterilized bottles of appropriate type and size for the parameters to be sampled and containing appropriate preservatives, if any are needed; and laboratory chain-of-custody forms. The labs will be responsible for analyzing the samples within sample holding times, performing proper QA/QC procedures, and reporting the results to CalTrout.

Data Management Activities: Janet Hatfield, Program Assistant for the Inyo-Mono Integrated Regional Water Management Program, is contracted by CalTrout to serve as Data Manager (DM) for the Mammoth Lakes Basin NPS Project. The DM will be responsible for data management duties including checking over the laboratory water quality results upon their receipt from the analytical laboratories, performing the data verification procedures set out in this QAPP, requesting stream flow data from the Mammoth Community Water District following sampling events in a timely manner, setting up and maintaining an in-house CalTrout database, entering the hand-measured water quality and stream flow data into the database, requesting electronic spreadsheet data files from the analytical laboratories and loading the lab data into the database, backing up the data files, and storing the hardcopy and electronic data in a secure location.

Consulting Scientist(s): Luke Hunt, Ph.D. is an Associate Director of Conservation for American Rivers, who is contracted by CalTrout as a consultant.

A4.2 Quality Assurance Officer Role.

Quality Assurance (QA) Officer (CalTrout). W. Mark Drew, Ph.D., the Project Director for the Mammoth Lakes Basin NPS Project, will also serve as the QA Officer for the project. The QA Officer is responsible for oversight of the implementation of the project activities undertaken with respect to this QAPP, in coordination with CalTrout and the SWRCB. The QA Officer is responsible for maintaining QA for the Mammoth Lakes Basin NPS Project. The CalTrout QA Officer will:

- Remain independent of direct job involvement and day to day operations
- Have direct access to the SWRCB GM and QAM as necessary to resolve any QA dispute
- Have direct access to the Contracting Analytical Laboratories to resolve any QA dispute
- Coordinate with CalTrout to ensure that the QA objectives of the project are met
- Perform a QA audit on various phases of field operations, as needed
- Require and review actions taken in the event of QA failure and document any factors affecting data usability for the historical record.

A4.3 Persons responsible for QAPP update and maintenance.

Changes and updates to this QAPP may be made after review of evidence for the needed change by CalTrout's Project Director/QA Officer and Field Manager/Water Quality Specialist with the concurrence of both the SWRCB's Grant Manager and Quality Assurance Officer. The CalTrout Field Manager/Water Quality Specialist will be responsible for making the changes, submitting drafts for review, preparing a final copy, and submitting the final for signature.

Figure 1: Project Organizational Chart

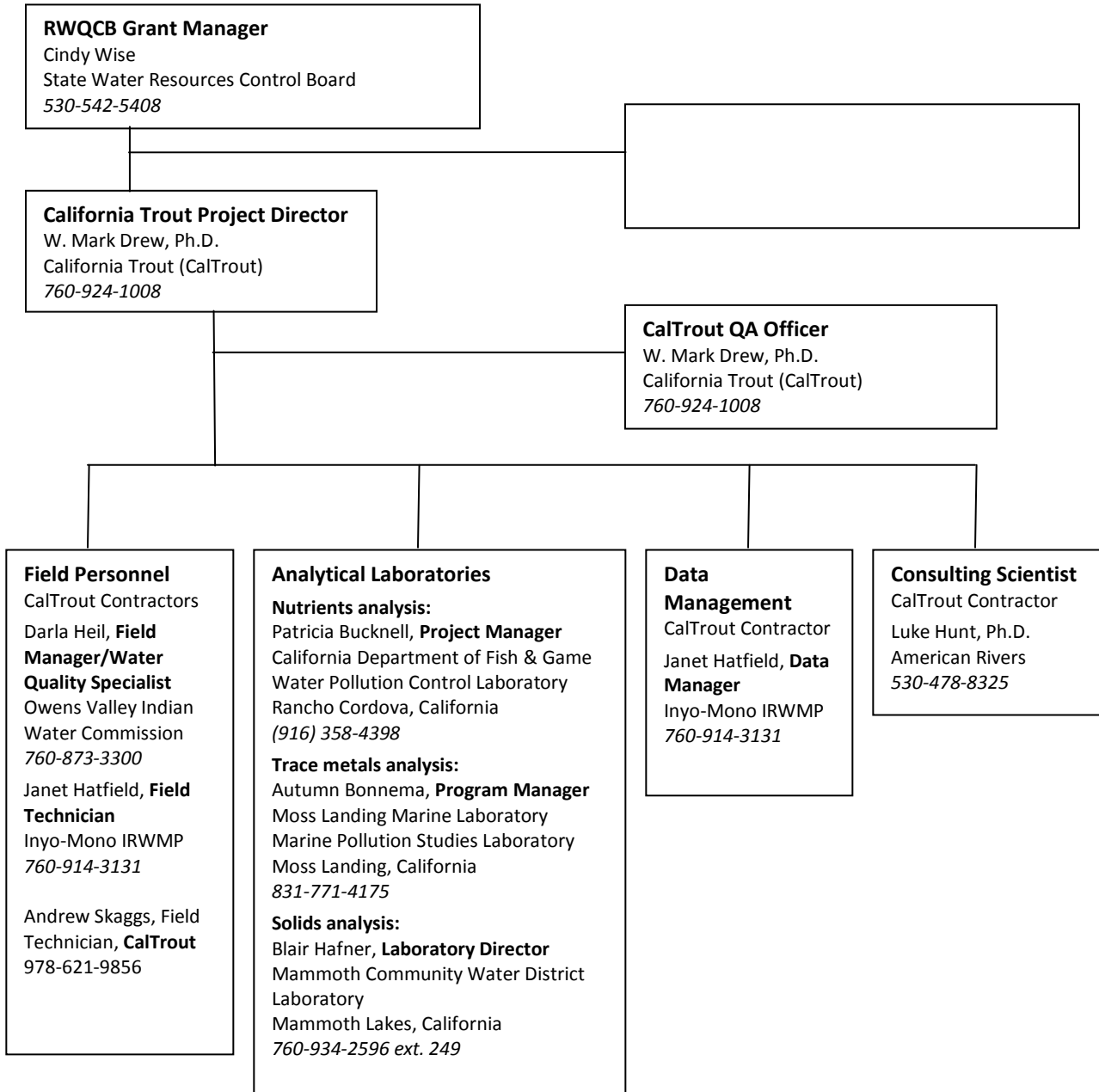


Table 1: Project Personnel and Technical Advisory Committee Members – Contact Information.

Name	Organizational Affiliation	Title	Contact Information (Telephone number, email address)
Project Personnel			
W. Mark Drew, Ph.D.	CalTrout	Project Director/QA Officer	760-924-1008 mdrew@caltrout.org
Janet Hatfield	Inyo-Mono Integrated Regional Water Management Group, CalTrout Contractor	Field Technician/ Data Manager	760-914-3131 Janet@inyomonowater.org
Andrew Skaggs	California Trout AmeriCorps	Field Technician/ Outreach Coordinator	978-621-9856 askaggs@caltrout.org
Darla Heil	Owens Valley Indian Water Commission, CalTrout Contractor	Field Manager/Water Quality Specialist	760-873-3300 darlah@oviwc.com
Luke Hunt, Ph.D.	American Rivers, CalTrout Contractor	Consulting Scientist	530-478-8325 lhunt@americanrivers.org
Technical Advisory Committee (TAC) Members			
Todd Ellsworth	Inyo National Forest, Forest Hydrologist	Advisory	tellsworth@fs.fed.us
Clay Murray	Mammoth Community Water District, Resource Monitoring Specialist/Water Treatment Operator III	Advisory	(760) 934-2596 cmurray@mcwd.dst.ca.us
Peter Bernasconi, P.E.	Town of Mammoth Lakes, Senior Civil Engineer	Advisory	pbernasconi@ci.mammothlakes.ca.us
Dan Dawson	Sierra Nevada Aquatic Research Laboratory, Director	Advisory	dawson@eri.ucsb.edu
David Parker	Professor of Environmental Chemistry at UC Riverside	Advisory	david.parker@ucr.edu

A5. PROBLEM DEFINITION/BACKGROUND

A5.1 Problem statement.

Background: The Mammoth Lakes Basin is near the Town of Mammoth Lakes (TOML) and is surrounded by the 11,000-foot Mammoth Crest (Sierra Crest) to the west and the Sherwins to the south. The lakes in the upper basin include Barney Lake, Skelton Lakes, and a number of other lakes, (see Appendix A, Maps 1 & 2, Upper Owens River Water Quality Project – Land Ownership & Sampling Sites) and in the lower basin include Lake George, Horseshoe Lake, Lake Mary, Lake Mamie and Twin Lakes. These lakes eventually drain into Mammoth Creek, which drops into the valley flowing east, changing its name into Hot Creek east of Highway 395 and eventually flowing into the Owens River. This project will focus on the Mammoth Lakes Basin and the Mammoth Creek and Hot Creek watersheds.

The United States Forest Service (USFS), Bureau of Land Management (BLM), and the City of Los Angeles, Department of Water and Power (LADWP) are the major landowners within the watershed and manage over 90 percent of the land area (see Appendix A, Map 1, Upper Owens River Water Quality Project – Land Ownership). Livestock grazing occurs extensively on both public and private lands in the watershed. The TOML which sits on the western boundary of the Mammoth Lakes Basin serves as a gateway for recreational use of federal lands in the area. Visitor use in the TOML has grown rapidly, accounting for 3.7 million visitor-days annually. The population of this resort town is about 7,500 year-round residents (2004 data) but can reach 35,000 during peak weekends, and this is projected to increase in the future. The primary winter activities in and around Mammoth Lakes are downhill and cross-country skiing, and snowmobiling. In summer, visitors take advantage of a myriad of recreational opportunities such as hiking, rock climbing, cycling, fishing, boating, and off-road vehicle use. The Mammoth Lakes Basin flows into the Owens Valley watershed, a primary source of water for Los Angeles.

The water quality of the area's snowmelt runoff is assumed to be excellent (Watershed Management Plan 2007 - http://monocounty.ca.gov/cdd%20site/Planning/Documents/ManagementPlan_UpperOwensRiver.pdf). However, Mammoth Creek above Highway 395 is listed as impaired for total mercury, total manganese, and total dissolved solids; monitoring of these constituents will be included in this project. It is suspected that a spring flowing from a mine site that forms a tributary of Mammoth Creek may be the source of trace metal contamination in Mammoth Creek; as a part of this project the Mine Spring will be sampled and analyzed for total mercury and total manganese on a one-time basis. The grant agreement between CalTrout and the SWRCB specifies monitoring nutrient loads in the Mammoth Lakes Basin. Nitrogen and phosphorus were chosen because they often control biological productivity, which impairs dissolved oxygen levels in streams and overall ecosystem health. In addition, members of the TAC, which has been set up as a part of this project, have identified total suspended solids (TSS) as a constituent of concern in the Mammoth Lakes Basin and have requested that TSS be monitored as a part of this project.

Project Purpose: The overall goal of the Upper Owens River Water Quality Project is to reduce non-point source pollutant loads and restore beneficial uses. This project will establish a monitoring plan (see Appendix B) to assess and prioritize pollutant sources within the project area, as well as identify and prioritize measures to address the identified issues. The proposed project has the following specific objectives, which are consistent with United States Environmental Protection Agency's (U.S. EPA) Nine Key Elements of a Watershed Plan:

- Conduct a scientific assessment in the Mammoth Lakes Basin (including Mammoth Creek and Hot Creek watersheds), which is part of the Upper Owens River watershed, to identify causes and sources of nutrient (and other contaminant) loading;
- Identify and prioritize implementation measures that are necessary for reducing nutrient (and other contaminant) loads and pertinent constituents;
- Specify measurable pollutant load reductions associated with each priority implementation measure, and develop a monitoring plan to evaluate the effectiveness of the implementation efforts;
- Describe costs, technical needs, and schedule for implementing priority management measures; and
- Convene an Advisory Team to enhance public understanding of the project and encourage early and continued participation by a diverse group of stakeholders in selecting, designing and implementing NPS management measures.

This Quality Assurance Project Plan (QAPP) is being written to specifically to address the scientific assessment to be conducted in the Mammoth Lakes Basin to identify causes and sources of contaminant loading.

A5.2 Decisions or outcomes.

This project will provide information about the water quality of Mammoth Creek for the following parameters:

- *Constituents identified in the grant agreement with the SWRCB:* nutrients (total nitrogen, combined nitrate and nitrite (nitrate + nitrite), and total phosphorus).
- *TMDL constituents:* trace metals (total mercury in water (mercury), total manganese (manganese)) and total dissolved solids (TDS).
- *A constituent of concern to the TAC:* total suspended sediments (TSS).
- Field measurements of physical stream parameters: temperature, conductivity, pH, dissolved oxygen, and turbidity.

During the first field season of the two-year project (June – November, 2012), samples will be collected and measurements taken at eight to nine monitoring sites on Mammoth Creek during six sampling events. The data will be analyzed to identify water quality problems and possible sources. If pollutant concentrations approach or exceed California water quality objectives or standards during the first year of this project, additional sampling sites or parameters may be identified to investigate the contributing reach. The location of any additional sampling sites will be determined with the help of a detailed GIS map and in consultation with the TAC and SWRCB. During the second year of the project some of the initial sampling sites and parameters may be abandoned so that the sampling effort can become focused on more detailed investigation into any high pollutant source subwatersheds within the Mammoth Lakes Basin that were identified during the first sampling season. Building on the water quality information gathered, the focus of the second year of the project will be to identify, prioritize, and describe implementation measures designed to reduce nutrient loads and TMDL constituents in the Mammoth Lakes Basin. This task will be led by CalTrout, with assistance from American Rivers and Inyo National Forest and with substantial input from the TAC. Criteria will be developed and applied to prioritize implementation measures. Goals will be to specify measurable pollutant load reductions associated with each priority implementation measure and to develop a long-term monitoring plan to evaluate the effectiveness of the implementation efforts and to ensure that additional degradation of water quality due to nutrient or TMDL constituent loading is not occurring in the watershed.

A5.3 Water quality or regulatory criteria.

Regulatory thresholds for this project are summarized below in Tables 2 & 3.

Table 2: Laboratory analyzed pollutants, method citations and regulatory thresholds

Parameter	Method	Laboratory Reporting Limit	Regulatory Threshold
Total Nitrogen	QC 10107044B	0.0500 mg/L	See Table 3
Nitrate + Nitrite as N	QC 10107041B	0.0100 mg/L	See Table 3
Total Phosphorus as P	QC 10115012B	0.0100 mg/L	See Table 3
Total Dissolved Solids (TDS)	SM 2540 C	7 mg/L	See Table 3
Total Suspended Solids (TSS)	SM 2540 D	0.4 mg/L	n/a
Total Mercury in water	EPA 1631E Modified	0.0002 µg/L (0.200 ng/L)	0.05 µg/L (ppb)
Total Manganese in water	EPA 1638 Modified	0.05 µg/L	50 µg/L (ppb)

Table 3: Water Quality Objectives from Lahontan Basin Plan

from: http://www.waterboards.ca.gov/lahontan/water_issues/programs/basin_plan/docs/ch3_wqobjectives.pdf

Ch. 3. WATER QUALITY OBJECTIVES									
Table 3-17 WATER QUALITY OBJECTIVES FOR CERTAIN WATER BODIES OWENS HYDROLOGIC UNIT									
See Fig. 3-10	Surface Waters	Objective (mg/L) ^{1,2}							
		TDS	Cl	SO ₄	F	B	NO ₃ -N	Total N	PO ₄
1	Owens River (above East Portal)	110	11.0	5.0	0.40	0.40	0.1	0.2	0.90
		200	16.0	8.0	0.80	0.80	0.1	0.5	3.75
2	Owens River (below East Portal)	100	6.0	6.0	0.30	0.20	0.5	0.6	0.73
		150	12.0	16.0	0.60	0.40	1.0	1.5	0.94
3	Coldwater Creek	35	0.7	-	-	-	0.5	0.5	0.02
		40	1.4	-	-	-	1.0	1.0	0.03
4	Mammoth Creek (Twin Lakes Bridge)	60	0.6	-	-	-	0.4	0.5	0.03
		90	1.0	-	-	-	0.8	1.0	0.05
5	Mammoth Creek (Old Mammoth Road)	85	0.8	-	-	-	0.4	0.6	0.27
		115	1.4	-	-	-	0.8	1.0	0.50
6	Mammoth Creek (at Hwy. 395)	75	1.0	6.0	0.10	0.03	0.4	0.6	0.11
		100	1.4	11.0	0.30	0.05	0.8	1.0	0.22
7	Sherwin Creek	22	0.5	-	-	-	0.4	0.5	0.05
		26	0.7	-	-	-	0.6	0.7	0.08
8	Hot Creek (at County Rd)	275	41.0	24.0	1.80	1.80	0.2	0.3	0.65
		380	60.0	35.0	2.80	2.60	0.4	1.5	1.22

A6. PROJECT/TASK DESCRIPTION

A6.1 Work statement and produced products.

Monitoring during the first year of this two-year project will include approximately six stream sampling events (on an approximate monthly basis from June through October or November) for nutrients (total nitrogen, nitrate + nitrite, total phosphorus) and solids (TDS, TSS), and three lower frequency sampling events (approximately bi-monthly) for metals (total mercury and total manganese) at eight to nine sites to determine event-based loads and annual export. Initially nine sampling locations have been designated by the TAC to sample surface water from accessible sites that are within the six distinct major watersheds comprising the Mammoth Lakes Basin as identified by the MCWD in the Mammoth Creek Environmental Impact Report (<http://www.mcwd.dst.ca.us/ProjectsReports/MamCrkEIR.htm>) (see Appendix A, Map 2, Upper Owens River Water Quality Project – Sampling Sites). One of the nine sites, Site MC-7 – “Murphy Gulch Confluence”, will be sampled only when the ephemeral tributary to Mammoth Creek, Murphy Gulch, flows. In addition, an effort will be made to collect samples within 24 hours after one storm event (with over 1.0 inch of rainfall, as measured at one of the weather stations in the Mammoth Lakes area described below, in Section B18) at a subset of three sampling sites that have been identified by the TAC (see Table 6 in Section B10, below). These post-storm samples will be analyzed for total nitrogen, nitrate + nitrite, total phosphorus, TDS, and TSS. One additional sampling site, Site MC-4A, “Mine Spring”, has been identified for a one time sampling that will be carried out during mid-summer for analysis of total mercury and total manganese; Mine Spring has been identified by the SWRCB as a possible source of mercury and manganese pollution in Mammoth Creek.

As a part of each sampling event field measurements will be recorded at each of the sampling sites using a YSI 6820V2 Sonde for water temperature, conductivity, pH, dissolved oxygen, and turbidity as well as specific conductance, total dissolved solids (TDS), and salinity (which are calculated by the recording instrument from conductivity and temperature readings). In addition stream flow averages and cfs data during sample collection will be provided by the Mammoth Community Water District (MCWD) from the gauging stations they use for monitoring stream flow on Mammoth Creek. Stream flow data will also be acquired from the Los Angeles Department of Water and Power “395” gauge to ensure accurate information is available for the lower sampling sites (sites 7, 8, & 9).

If pollutant concentrations near to or exceeding California water quality objectives or standards are found during the first year of this project, additional sampling sites or parameters may be identified to investigate the contributing reach. The location of any additional sampling sites will be determined with the help of a detailed GIS map and in consultation with the TAC and SWRCB. During the second year of the project some of the initial sampling sites and parameters may be abandoned so that the sampling effort can become focused on more detailed investigation into any high pollutant source watersheds within the Mammoth Lakes Basin that were identified during the first sampling season.

A6.2 Constituents to be monitored and measurement techniques.

Monitoring will consist of field measurements for temperature, conductivity, pH, turbidity and dissolved oxygen. Samples will be collected for total nitrogen, nitrate + nitrite, total phosphorus, TDS, TSS, total mercury, and total manganese and analyzed by a certified analytical laboratory. For laboratory analysis methods, please refer to Table 2 (in Section A5.3, above).

A6.3 Project schedule.

A project schedule follows.

Table 4: Project schedule.

Activity	Date (MM/DD/YY)		Deliverable	Deliverable Due Date
	Anticipated Date of Initiation	Anticipated Date of Completion		
Start Project	2/1/2012	4/1/2014	Final Report	4/1/2014
Field Sampling	6/5/2012	9/7/2013	Monitoring Plan	Approval needed before implementation of monitoring program
			Quality Assurance Project Plan	Approval needed before implementation of water quality sampling
			Quarterly Progress Reports	Before the 20 th day of the month following the end of each quarter
			Proof of water quality data submission to CEDEN	Before final invoice

A6.4 Geographical setting.

The upper reaches of the Owens River system lies in the Eastern Sierra Nevada Mountains and includes the Mammoth Lakes Basin and Mammoth Creek and Hot Creek watersheds (see Appendix A, Maps 1-3); it is known as the Long Hydrologic Area which encompasses approximately 380 square miles. The Mammoth Lakes Basin is a large glacial cirque that was carved by the grinding movement of glacial ice and rocks entrained within the ice. The lakes of Mammoth Lakes Basin eventually drain into Mammoth Creek, which drops into the valley flowing east, changing its name into Hot Creek east of Highway 395 and eventually flowing into the Owens River. This project will focus on the Mammoth Lakes Basin and the Mammoth Creek and Hot Creek watersheds covering an area of approximately 86 square miles (drainage area) and comprising roughly 25 percent of the Upper Owens River Watershed (based on HUC 5 scale). Mammoth Lakes Basin, Mammoth Creek and Hot Creek are particularly vulnerable to impairments associated with stream chemistry and hydrologic characteristics given the rapid development in the area and the amount of agricultural, grazing, and recreation activities occurring there.

A6.5 Constraints.

The length of the sampling season for this project could be constrained by weather and access to the sampling sites, as well as by the time when the agreement is signed and necessary documents (monitoring plan and QAPP) are prepared by CalTrout and approved by the SWRCB so that the monitoring program can begin. The Mammoth Lakes Basin is a region that has experienced heavy early and late season snow fall in the past. Since the elevation of the sampling sites for this project ranges from approximately 2,807 to 2,165 meters of elevation, the upper sites could become inaccessible early in the scheduled sampling season or before the end of the scheduled sampling season due to late and early season snowstorms, which could curtail the sampling season at inaccessible sites.

A7. QUALITY OBJECTIVES & CRITERIA FOR MEASUREMENT DATA

Data quality indicators for this project will consist of the following:

- Nutrients (total nitrogen, nitrate + nitrite, total phosphorus) – Accuracy, Completeness, Precision, Representativeness, Sensitivity
- Solids (total dissolved solids, total suspended solids) - Completeness, Precision, Representativeness, Sensitivity
- Metals (total manganese, total mercury) – Accuracy, Completeness, Precision, Representativeness, Sensitivity
- Field measurements (temperature, specific conductivity, pH, turbidity, dissolved oxygen) - Accuracy, Completeness, Precision

The fundamental QA objective with respect to accuracy, precision, bias, and sensitivity of laboratory analytical data is to achieve the QC acceptance criteria of the analytical protocols. Each of the laboratories that will analyze the water quality data for this project adheres to Standard Methods or Standard Operating Procedures (SOPs) for the laboratory analyses performed. See Appendix B “List of Associated QAPPs, QA Documents, Methods and/or Standard Operating Procedures (SOPs)” for the reference to the laboratory QA Documents, Methods and SOPs that will be employed for this project. Precision measurements will be determined on both field and laboratory replicates. Field duplicates will be collected for analysis for 5% of the total project sample count for each analyte. Each laboratory will analyze sample replicates as described in their QA Documents and SOPs (Appendix B).

Precision is a measure of the degree by which two or more measurements are in agreement. Accuracy is the degree to which an observed value and an accepted reference value agree. The precision and accuracy of the field measurements will be maintained by adhering to the calibration procedures and measurement SOPs included in Appendix B and described in Element B16: “Instrument/Equipment Calibration and Frequency” of this document. Method sensitivity is dealt with by the inclusion of the required SWAMP Target Reporting Limits, where such values exist.

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or parameter which is dependent upon the proper design of the sampling program and proper laboratory protocol. More critically, for this project, representativeness is a measure of how well samples reflect environmental conditions and the areas targeted for assessment. The project design factors (as reflected in sampling locations, sampling schedule, and sampling frequency) are key factors contributing to representativeness. The sampling network was designed to provide data representative of site conditions. During development of this network, consideration was given to existing analytical data, physical setting and processes, regulatory boundaries, areas of interest, and constraints. The rationale of the sampling network is described in detail in Section B10: “Sampling Process Design”, below. Representativeness will be satisfied by ensuring that proper sampling techniques are used, proper analytical procedures are followed, and holding times of the samples are not exceeded in transit or at the laboratory.

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. It is expected that the CalTrout contracting analytical laboratories will provide data meeting QC acceptance criteria for 90 percent or more for all samples tested using the laboratory standard operating procedures.

The measurement quality objectives for accuracy and precision for each of the parameter groups, and the target reporting limits for each analyte to be analyzed for this project are included below, in Table 5.

Table 5: Data Quality Objectives Table.

Laboratory Analysis Data Quality Objectives (DQOs) are SWAMP requirements taken from Appendix A, Tables A-1 and A-5, of the "Electronic Template for SWAMP- Comparable Quality Assurance Project Plans" accessed at: http://www.waterboards.ca.gov/water_issues/programs/swamp/qapp.shtml. Field Measurement DQOs are taken from Appendix A, Table A-10, in the Template document referenced above, and from the YSI 6-Series Multiparameter Water Quality Sonde User Manual, October 2006, Revision D, Appendix O, Sensor Specifications.

Analyte Group	Parameter	Accuracy	Precision	Target Reporting Limits	Completeness
Laboratory Analyses					
Nutrients	Total Nitrogen	± 20%	Laboratory duplicates: RPD<25% (n/a if concentration of either sample < RL)	0.05 mg/L	100%
	Nitrate + Nitrite	± 20%		0.01 mg/L	100%
	Total Phosphorus	± 20%		0.01 mg/L	100%
Solids	Total Dissolved Solids	N/A		10 mg/L	100%
	Total Suspended Solids	N/A		0.5 mg/L	100%
Trace Metals	Total Mercury	± 25%		0.0002 µg/L	100%
	Total Manganese	± 25%	0.05 µg/L	100%	
Field Measurements - YSI 6820V2 Sonde					
	Temperature	± 0.15°C	± 0.15°C		100%
	pH	± 0.2 pH units	± 0.2 pH units		100%
	Specific Conductivity	± 5% of reading + 0.001 mS/cm	± 5% of reading + 0.001 mS/cm		100%
	Turbidity	± 2% of the reading or 0.3 NTU, whichever is greater	± 2% of the reading or 0.3 NTU, whichever is greater		100%
	Dissolved Oxygen	0 to 20 mg/L: ± 1% of the reading or 0.1 mg/L, whichever is greater	0 to 20 mg/L: ± 1% of the reading or 0.1 mg/L, whichever is greater		100%

Comparability expresses the confidence with which one data set can be compared with another. The extent to which the analytical data will be comparable depends on the similarity of sampling and analytical methods. The procedures used to obtain the project analytical data, as documented in this QAPP, are expected to provide comparable data within this project. These new analytical data, however, may not be directly comparable to older data from other sources because of differences in procedures and QA objectives.

A8. SPECIAL TRAINING NEEDS/CERTIFICATION

A8.1 Specialized training or certifications.

No specialized training requirements or certifications are required of field personnel working on this project. The Field Manager/Water Quality Specialist, Darla Heil, holds an M.S. degree in Earth Sciences from the University of California, Santa Cruz and has worked in water quality and resource issues programs for the past twenty-two years, having undergone sampling technique training courses covering surface, and groundwater sampling, QA/QC protocol and methodology, field analysis, and recording. Ms. Heil is currently the Project Manager for groundwater and surface water monitoring and sampling programs being conducted on the Big Pine Paiute Reservation and the Lone Pine Paiute-Shoshone Reservation through her position as Environmental Specialist at the Owens Valley Indian Water Commission. The Field Technician/Data Manager, Janet Hatfield, holds a B.S. in Forestry from Colorado State University, and has recently completed her first year of coursework in the Masters of GIS program at Penn State University. Ms. Hatfield worked for the National Park Service for 12 years where she led a number of resources related field projects, all of which required data capture and management. Though no specialized training is required for this project, both Ms. Heil and Ms. Hatfield will review the SWAMP Field Methods Course materials accessible at:

http://water101.waterboards.ca.gov/swamp/qapp_advisor/FieldMethods/start.html as well as sampling protocol training videos available on the internet such as the video, "Collecting Water Quality Samples Using the Clean Hands Dirty Hands Technique" which is accessible at: <http://www.youtube.com/watch?v=BIHJFO4pfpl>.

Laboratory certification is not required for State Grant Projects, but the CDFG-WPCL and the MCWD Laboratory are both certified by the Environmental Laboratory Accreditation Program (ELAP) for the analyses they will perform for this project and the MLML-MPSL has State Water Resources Board approval for the analyses that they will perform.

A8.2 Training and certification documentation.

The project field personnel will document the time that each of them spends on field method training activities, and will report these activities to the Project Director. Documentation will consist of a record of the training name, date, and time spent.

The contracting analytical laboratories maintain records of their training activities. Those records can be obtained if needed from the laboratories.

A8.3 Training personnel.

The MLB-NPS Project Quality Assurance Officer and Field Manager/Water Quality Specialist will provide training to participating field personnel. Each analytical laboratory's Quality Assurance Officer will provide training to lab personnel.

A9. DOCUMENTS AND RECORDS

Janet Hatfield will serve as the CalTrout Data Manager for this project. All data generated for the MLB-NPS Project will be computerized in a format organized to facilitate data review and evaluation. All data will be backed up following each sampling event or any data input event. The field data recorded during

water quality measurement activities will be entered into spreadsheet database maintained by CalTrout. Other documentation to be maintained by the CalTrout Data Manager and project personnel includes field logbooks, chain-of-custody forms, hard copy analytical reports (after they are received from the contracting analytical laboratories), and field equipment maintenance and calibration records. CalTrout will maintain the electronic records for a period of at least one year following completion of this project on office computers and/or backup devices. CalTrout's contracting laboratories will maintain analytical equipment maintenance and calibration records, chain-of-custody forms, analytical reports, and QA/QC information. The water quality monitoring laboratory and field measured data will be uploaded to the California Environmental Data Exchange Network (CEDEN) through the appropriate SWAMP Data Center by the Data Manager before the grant is completed and after the final laboratory results are received from contracting laboratories. The Data Manager will use data formats and data management processes provided by SWAMP and CEDEN to ensure that all data collected as part of this project will be compatible with the database. CalTrout will provide the State Water Board with a receipt of successful data submission, which is generated by CEDEN. The laboratory and field measurement activities will be reported to the SWRCB in Quarterly Monitoring/Progress Reports and the results will be summarized in a Final Project Report before the project is completed. Copies of this QAPP and succeeding revisions of this QAPP will be submitted to the individuals identified in Section A3 in electronic format by the Project Director following approval of the document by the SWRCB.

GROUP B: DATA GENERATION AND ACQUISITION

B10. SAMPLING PROCESS DESIGN

Sample collection points and a justification for selection sites are described in the Monitoring Plan which is included in this QAPP in Appendix C. After consultation with the Technical Advisory Committee (TAC) nine water quality sampling points were selected on Mammoth Creek or Hot Creek meeting these criteria: publically accessible sites that are located in key spots in the six distinct sub-watersheds comprising the Mammoth Lakes Basin as identified by the Mammoth Community Water District in the Mammoth Creek Environmental Impact Review (<http://www.mcwd.dst.ca.us/ProjectsReports/MamCrkEIR.htm>). A tenth sampling point (MC-4A, Mine Spring), which is on a tributary to Mammoth Creek, was chosen for a one time sampling event for total manganese and mercury because it is suspected by the SWRCB that the Mammoth Creek tributary flowing from an abandoned mine spring upstream from that site may be the source of the total mercury and total manganese contamination in Mammoth Creek. The proposed sampling sites with their I.D. numbers, site names, approximate locations, and special sampling events are listed in Table 6, below, and shown on Map 2 (Appendix A, Upper Owens River Water Quality Project, Sampling Sites). The latitude, longitude and elevation locations listed in Table 6 are preliminary; the actual sampling sites will be field checked for accessibility for proper sample collection methods, and their locations recorded with a GPS prior to the beginning of the implementation of the sampling program. Refer to Appendix C: "Monitoring Plan" and Section A6: "Project/Task Description" of this document for more detailed discussion on the monitoring plan, planned sampling sites, sampling schedule, and parameters to be sampled and measured as a part of the MLB-NPS Project.

Table 6: Sample Sites Information Table.

Sample Sites		Approximate (Preliminary) Locations			Special Circumstance Sampling Events		
Sample I.D.	Site Name	Elevation (m)	Latitude	Longitude	One-time mid-summer sampling (total Mn, total Hg)	Sampled only if Murphy Gulch is flowing (nutrients, TDS, TSS)	Post-storm flow sampling sites (nutrients, TDS, TSS)
MC-1	Coldwater Creek	2807	37.591	-118.986			
MC-2	Lake Mary Bridge	2726	37.606	-119.008			
MC-3	Twin Lakes Bridge	2627	37.624	-119.005			
MC-4	Sherwin St. Bridge	2438	37.631	-118.987			X
MC-4a	Mine Spring	2533	37.623	-118.993	X		
MC-5	Mammoth Creek Park	2384	37.635	-118.962			X
MC-6	Sherwin Creek Campground	2269	37.634	-118.926			
MC-7	Murphy Gulch Confluence	2212	37.637	-118.913		X	X
MC-8	395 Flow Gauge	2195	37.641	-118.9			
MC-9	Hot Creek Confluence	2165	37.644	-118.853			

B11. SAMPLING METHODS

B11.1 Field Measurements.

Field measurements will be recorded at each site sampled during water quality sampling events using a YSI 6820V2 Multiparameter Sonde. The parameters measured will consist of water temperature, pH, dissolved oxygen, turbidity, and electrical conductivity. Specific conductance, salinity, and total dissolved solids (TDS), which are calculated by the instrument from the conductivity and temperature measurements, will also be recorded. The multi-probe Sonde will be placed in the body of water to be sampled and allowed to equilibrate while water samples are collected. Field measurements will be made as near the midpoint in the stream as safely possible and at a depth of approximately 0.2 m (8 in), following recommendation outlined in the California Department of Fish and Game - Marine Pollution Studies Laboratory (DFG-MPSL) Standard Operating Procedure (SOP), *Conducting Field Measurements and Field Collections of Water and Bed Sediment Samples in the Surface Water Ambient Monitoring Program* (http://swamp.mpsl.mlml.calstate.edu/wp-content/uploads/2009/04/swamp_sop_field_measures_water_sediment_collection_v1_0.pdf). The field measurements will be recorded after the water quality samples are collected on project Field Measurement Data Sheets (see Appendix D).

B11.2 Water Quality Analysis Samples.

The field personnel will collect sub-surface grab samples for the surface water quality analysis samples for conventional (nutrients and solids) and trace metals (total mercury and total manganese) constituents. Water samples will be collected from a location in the stream where the stream visually appears to be completely mixed, but where excess turbulence is not present. The samples will be taken from as near as is safely possible to the middle of the stream and at approximately 0.1 m (4 in.) depth, following recommendations outlined in the DFG-MPSL SOP referenced in the paragraph above. Sampling

from the shoreline of the stream (meaning standing on shore and sampling from there) is the least acceptable method, but in high water cases may be necessary. Water quality samples will be collected following established procedures explained in detail in the following SOPs that are fully referenced in Appendix D of this document DFG-MPSL SOP *“Conducting Field Measurements and Field Collections of Water and Bed Sediment Samples in the Surface Water Ambient Monitoring Program”* (Version 1, October 15, 2007) and U.S. EPA Method 1669 *“Sampling Ambient Water for Trace Metals at EPA Water quality Criteria Levels”* (July 1996) and incorporating modifications to EPA Method 1669 by MLML-MPSL which are included in Appendix D. Following are synopses of the above referenced procedures.

B11.3 Collection of Water Samples for Analysis of Conventional Constituents.

Water samples for analysis of conventional constituents will be collected following the DFG-MPSL SOP's cited in the paragraph above, using pre-cleaned, sterilized, inert (polyethylene or glass) sample bottles with necessary preservatives added that will be supplied by the analytical laboratory. To collect samples, the capped bottles will be held under the water surface to a depth of approximately 0.1 m below the surface at a safely accessible location approximately midway across the stream in a spot that is well mixed, but without excessive turbulence. The bottle's cap will be removed under water with the bottle pointed upstream away from the sample collector, and the bottle will be filled before the cap is replaced under water. Each sample bottle will be properly labeled with the station ID, sample identification number, matrix type, analysis type, project ID, and date and time of collection.

B11.4 Collection of Water Samples for Analysis of Trace Metals Constituents.

Metals-in-water samples should **not** be collected during periods of abnormally high turbidity if at all possible. Samples with high turbidity are unstable in terms of soluble metals, and it is difficult to collect a representative sub-surface grab sample. The total mercury and total manganese samples will be taken following clean hands/dirty hands methodology as outlined in the DFG-MPSL SOPs and U.S. EPA Method 1669 referenced above and in Appendix D and as summarized in *“EPA Methods 1669 Field Sampling Quick Reference”* provided by Brooks Rand Laboratories and included in Appendix D. The personnel involved in trace metals field sample collection will wear an outer pair of polyethylene gloves and will minimize the chance of contamination of the samples by removing all metal jewelry and avoiding breathing directly on the sample. The laboratory pre-cleaned glass or Teflon™ 250 mL (for mercury) or polyethylene 60 mL (for manganese) sample bottles are taken from the double-wrapped plastic bags using *“Clean Hands/Dirty Hands”* techniques. The dirty hands person opens the first bag, and the clean hands person opens the inner bag around the bottle. The clean hands person then removes the bottle from the inner bag. The clean hands person dips the bottle into the ambient water, with the cap on and the bottle pointed upstream away from the clean hands person, to approximately 0.1 m depth (avoiding disturbing surface scums), removes the cap to fill the bottle, placing the cap back on the bottle before being removed from the water, rinses the bottle five times with ambient water, making sure the threads of the bottle get rinsed as well, and fills the bottle to the top. The lid is secured and the bottle is put back into the inner clean bag and sealed by the clean collector. The dirty hands collector then seals the outer bag. It is very critical that all the acid is rinsed out of the bottles before the samples are taken and the bottles are cleaned according to laboratory protocol. After collecting the sample, the double-bagged container is placed in another plastic bag for shipping, and placed on ice in the ice chest, cooled to 4°C. This is to prevent possible contamination from other samples in the ice chest. Metals-in-water samples must be acid-preserved in the lab per the laboratory's SOPs within 48 hours of collection. Each outer sample-bag will be labeled with the station ID, sample identification number, matrix type, analysis type, project ID, and date and time of collection.

B11.5 Special Considerations and Field Duplicates.

No filtered samples should be required for this project. Decontamination of sampling equipment will not be necessary at any of these surface water sampling sites because only dedicated sampling equipment will be used to obtain water samples. No field equipment blanks should be needed for this project, since only dedicated sampling bottles will be used to obtain water samples, allowing no cross contamination.

Field duplicates will be submitted at an annual rate of 5%. When collecting duplicate water samples, the duplicate bottles will alternate in the filling sequence. Note that both duplicate bottles for one type of analysis will be filled separately from those for other analytes. Duplicate samples will be preserved, packaged, and sealed in the same manner as other samples of the same matrix and constituent type. A separate Station ID number will be assigned to each duplicate, and they will be submitted blind to the laboratory. The Station ID's of the duplicates will be numbered as in the following examples: Site MC-12 (if collected at site 1) or Site MC-52 (if collected at Site 5), and so on.

B11.6 Sample Preservation.

Sample preservation techniques will follow EPA and Standard Methods protocols (Table 2, page 12). All samples to be collected will be collected in pre-cleaned, sterilized bottles of the appropriate type and size for the analyses to be performed provided by the analytical laboratories. Analytical grade sulfuric acid will be added to the nutrient sample bottles by the CDFG WPCL so that samples collected for nutrient analyses will be acidified to a pH of less than 2 (Table 7, below, page 22). The pH of the acid preserved nutrient samples will be verified at the time of collection by the use of a pH strip. After sample collection, the containers will be agitated slightly to assure uniform mixing of the preservative and the sample. A small amount of the mixed sample and preservative will be poured into the sample container lid. The pH will be checked by dipping a pH strip into the aliquot of sample in the lid, and comparing it to the color chart on the pH strip container. The aliquot of sample in the lid will then be discarded before using the lid to tightly recap the sample bottle. If necessary the pH will be adjusted by adding extra aliquots of preservative, which will be provided by the analytical laboratories, and the sample pH will be rechecked following the procedure described above. Trace metals samples will be acidified with analytical grade nitric acid at the analytical laboratory, MLML MPSL, within the 48 hour holding period per the laboratory's methods and requirements.

B11.7 Corrective Actions.

In the event that failure occurs in sampling or measurement, the Field Manager shall record details of the nature of the problem or failure in the field logbook and notify the Project Director and QA Officer. The Field Manager together with the QA Officer shall be responsible for determining whether re-sampling or measurement is required and maintain documentation of the decision to accept or reject the suspect sampling or measurement data.

Table 7: Sample Volumes, Containers, Preservatives, Holding Times, & Maximum Number Samples.

Analytical Parameter	Volume Required (ml)	Sample Container	Preservative	Holding Time	Maximum # Samples 2012 – All Sites, (including 5% field duplicates)
Total Nitrogen	150-250	Polyethylene	H ₂ SO ₄ to pH≤2; cool to 6°C and store in the dark	28 days	60
Nitrate + nitrite	150-250	Polyethylene	H ₂ SO ₄ to pH≤2; cool to 6°C and store in the dark	28 days	60
Total phosphorus	300-500	Polyethylene	H ₂ SO ₄ to pH≤2; cool to 6°C and store in the dark	28 days	60
Total dissolved solids	100	Polyethylene	Cool to 6°C and store in the dark	7 days	60
Total suspended solids	1000	Polyethylene	Cool to 6°C	7 days	60
Total mercury in water	250-500	Acid cleaned Glass or Teflon™	Cool to 6°C in the dark	Within 48 hours acidify with pretested HCl to 0.5% at lab; then analyze within 6 months	30
Total manganese in water	60-100	Acid Cleaned Polyethylene	Cool to 6°C in the dark	Within 48 hours acidify with pretested HNO ₃ to pH≤2 at lab, then analyze within 6 months	30

B12. SAMPLE HANDLING AND CUSTODY

All samples will be placed in a cooler of blue ice or ice cubes at ≤6° Celsius for transport from the field site and maintained at that temperature until received at the laboratory. Samples will be kept upright during transport and tightly capped. Prior arrangement with the analytical laboratory and shipping by appropriate means will ensure that holding times are not violated. Refer to Table 7, above for sample volumes, containers, preservation, and holding times. Trace metals samples with short holding times (48 hours before the samples must be acidified at the analytical laboratory) will be shipped via a carrier using overnight delivery service in coolers provided by the MLML MPSSL. The sulfuric acid preserved nutrient samples, which have a longer holding time (28 days) will also be shipped to CDFG WPCL in ice packed coolers that will be provided by the lab; the samples will be shipped via a next day or second day delivery service with enough ice to maintain the sample temperatures. The solids samples will be packed in coolers with adequate ice to cool the samples to ≤6°C and will be hand-delivered to the MCWD Laboratory before the end of the day of the sampling event or stored in a refrigerator until the next morning when they will be delivered to the lab.

B12.1 Field Specific Custody Procedures.

The sample packaging and shipment procedures summarized below will insure that the samples will arrive at the laboratory with the chain-of-custody intact.

- The field personnel are personally responsible for the care and custody of the samples until they are transferred or properly dispatched.
- All bottles will be tagged with sample numbers and locations. The sample tags are typically adhesive backed paper or plastic and will be provided by the analytical laboratories. Care will be taken to assure that sample tags are securely affixed to the sample bottles. In the case of the trace metals samples collected using clean hands/dirty hands procedures, the outside of the outer sealed ziplock baggie will be labeled with the appropriate information with permanent markers.
- Sample tags are to be completed for each sample using waterproof ink, unless prohibited by weather conditions. For example, a logbook notation will explain that a pencil was used to fill out the sample tag because the ball point pen would not function in the wet weather. Sample labels should also indicate the analysis for each sample, the preservative used, the time the sample was collected, and preferably the sampler's name or initials.
- The Field Manager and/or QA/QC Officer will review all field activities to determine whether proper custody procedures were followed during the field work and decide if additional samples are required.

B12.2 Field Logbooks/Data Sheets/Documentation.

Field logbooks and/or datasheets will provide the means of recording the data collecting activities performed. As such, entries will be described in as much detail as possible so that persons going to the site could re-construct a particular situation without reliance on memory.

Field logbooks will be bound notebooks. Logbooks will be assigned to field personnel, but will be stored in the document control location when not in use. The project-specific document number or title will identify each logbook. The title page of each logbook will contain the following:

- Person to whom the logbook is assigned;
- Project name;
- Project start date, and;
- Project end date.

The logbook will contain a variety of information about each water quality sampling event. Entries for each event will include the date, start and end times, weather, level of personal protection being used, any significant observations, a detailed description of the location of the sampling site if it is different from the regular sampling site. The names of the visitors to the site and the purpose of their visit will also be recorded in the field logbook.

Field Measurement Data Sheets and Water Quality Sampling Data Sheets will be printed loose leaf forms, examples of which are included in Appendix D. Measurements made and samples collected will be recorded on these loose leaf forms or the same data will be recorded in the field notebook. All entries will be made in ink or a permanent marker and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark. Whenever a sample is collected, or a measurement is made, a detailed description of the location of the station shall be recorded if it is different from the regular sampling site. The number of the photographs taken of the station, if any, may also be noted. All equipment used to make measurements or to collect samples will be identified. The date and time a sample is taken or a measurement is recorded will be noted, along with the depth at which the sample was collected and the number of containers. Sample identification numbers will be

assigned prior to sample collection. The sample identification numbers will consist of: Site ID-Date, with the date in the format yymmdd (i.e. MC-1-120715 indicates a sample taken at Site MC-1 on July 15, 2012). Collection of any field duplicate samples, which will receive an entirely separate sample identification number, will be noted on the form. Duplicate identification numbers will consist of the Site ID+2-Date with the date in the same format as for a sample (i.e. MC-22-121015 indicates a duplicate sample taken at Site MC-2 on October 15, 2012).

B12.3 Transfer of Custody and Shipment Procedures.

The procedures are as follows:

- a) A properly completed chain-of-custody form accompanies samples. The sample ID numbers, site ID number and site name will be listed on the chain-of-custody form. Each analytical lab will supply chain-of-custody forms for the samples they will analyze. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents the transfer of custody of samples from the sampler to another person, to the sample shipper, to the analytical laboratory, or to/from a secure storage area.
- b) Samples will be properly packaged for shipment or hand delivery and will be dispatched to the contracting laboratories for analysis, with a separate signed custody record enclosed in each sample box or cooler. Shipping containers will be secured with shipping tape and custody seals for shipment to the laboratories unless they are hand delivered. The preferred procedure includes use of a custody seal attached to the front right and back left of the cooler. The cooler is strapped shut with strapping tape in at least two locations.
- c) The Chain-of-Custody Record identifying the contents will accompany all shipments. The original record will accompany the shipment, and one copy will be retained by the sampler and stored in the secure document area.
- d) If the samples are sent by common carrier, a bill of lading or an air bill should be used. Receipts of bills of lading or air bills will be retained as part of the permanent documentation. Commercial carriers are not required to sign off on the custody form as long as the custody forms are sealed inside the sample cooler and the custody seals remain intact.

It will be the responsibility of the Field Manager and/or Field Technician to ensure these procedures are followed.

B12.4 Laboratory Chain-of-Custody Procedures.

The chain-of-custody procedures for CalTrout's analytical laboratories are described in each laboratory's QA documents. These custody procedures along with holding time requirements for laboratory program samples are described in each laboratory's QA documents.

B12.5 Final Evidence File Custody Procedure.

The evidence files for analytical data will be maintained by CalTrout under the care of the Project Director and Data Manager. The content of the evidence file will include all relevant records, reports, correspondence, logs, field logbooks, data package, pictures, reports, chain-of-custody records/forms, data review reports, etc. The evidence file will be under custody of the Project Director and Data Manager at the CalTrout office in Mammoth Lakes, California.

B13. Analytical Methods

Analytical methods and equipment required are identified in Tables 8 & 9, below. Analytical methods have been selected through discussion of data quality objectives with the project team, the State Water Board, and the analytical laboratories. Analytical methods will be performed in accordance with the analytical laboratories' standard protocols as described in their laboratory QA Manuals which are either website referenced or included in Appendix B.

Table 8: Field Measurement Analytical Methods.

YSI 6820V2 - Analytical Methods							
Analyte	Laboratory / Organization	Project Action Limit (units, wet or dry weight)	Project Reporting Limit (units, wet or dry weight)	Analytical Method		Achievable Laboratory Limits	
				Analytical Method/ SOP	Modified for Method yes/no	MDLs	Method
Temperature	Field monitoring by CalTrout field staff	None	NA	SM ⁽¹⁾ 2550 <i>(YSI field measurement)</i>	No		
Conductivity	Same	None	0.001 mS/cm	SM 2510B <i>(YSI field measurement)</i>	No		
Specific Conductance	Same	None	0.001 mS/cm	SM 2510B <i>(YSI 6820V2 calculation)</i>	No		
Total Dissolved Solids	Same	None	0.001 g/L	SM 1030F <i>(YSI 6820V2 calculation)</i>	No		
Salinity	Same	None	0.01 <i>(dimensionless reported as ppt)</i>	SM 2520B <i>(YSI 6820V2 calculation)</i>	No		
pH	Same	None	NA	SM 4500 H+B <i>(YSI field measurement)</i>	No		
Optical Turbidity	Same	None	0.1 NTU	SM 2130B <i>(YSI field measurement)</i>	No		
Optical Dissolved Oxygen	Same	None	0.01 mg/L	ASTM ⁽²⁾ D888-09C <i>(YSI field measurement)</i>	No		

⁽¹⁾ *Standard Methods (SM) for the Examination of Water and Wastewater*, 20th edition.

⁽²⁾ American Society for Testing and Materials (ASTM) Method

Table 9: Laboratory Analytical Methods.

Analyte	Laboratory / Organization	Project Action Limit (units, wet or dry weight)	Project Reporting Limit (units, wet or dry weight)	Analytical Method		Achievable Laboratory Limits	
				Analytical Method	Modified for Method yes/no	MDLs	Method
Nitrate + Nitrite	CDFG Water Pollution Control Laboratory	None	0.0100 mg/L	QC ⁽¹⁾ 10107041B (SOP: WPCL-AA-018)	No	0.0050 mg/L	NA
Total Nitrogen	CDFG Water Pollution Control Laboratory	None	0.0500 mg/L	QC 10107044B (SOP: WPCL-AA-069)	No	0.0208 mg/L	NA
Total Phosphorus	CDFG Water Pollution Control Laboratory	None	0.0100 mg/L	QC 10115012B	No	0.0041 mg/L	NA
Total Dissolved Solids	Mammoth Community Water District Laboratory	None	7 mg/L	SM ⁽²⁾ 2540C	No	1 mg/L	NA
Total Suspended Solids	Mammoth Community Water District Laboratory	None	0.4 mg/L	SM 2540D	No	0.1 mg/L	NA
Total Mercury in Water	MLML - Marine Pollution Studies Laboratory	None	0.0002 µg/L (0.200 ng/L)	EPA 1631E	Yes	0.200 ng/L	NA
Total Manganese in Water	MLML - Marine Pollution Studies Laboratory	None	0.05 µg/L	EPA 1638	Yes	0.03 µg/L	NA

⁽¹⁾ QuikChem (QC) Method by Lachat Instruments

⁽²⁾ *Standard Methods (SM) for the Examination of Water and Wastewater*, 20th edition.

B14. Quality Control

B14.1 Field Measurements.

Field measurements will be taken with a YSI 6820V2 Multi-Parameter Sonde. QC procedures for measurements of water temperature, conductivity, pH, turbidity, and dissolved oxygen are limited to checking the reproducibility of the measurement by obtaining multiple readings; by calibrating the probes prior to the measurement event; and by checking the instrument’s calibration by recording the readings of the calibration standards after the measurements are taken. Calibration SOPs are included in Appendix E and discussed in Section B16: “Instrument/Equipment Calibration and Frequency” of this document. Two or more measurements will be recorded at each site. Each measurement will be recorded along with the average of the results for each parameter. The difference between the largest and smallest reading will be calculated. The percent difference between the largest and smallest result will also be calculated using the following formula:

$$\text{Percent difference} = 100 * (\text{largest} - \text{smallest}) / \text{average}$$

The difference or percent difference, as appropriate, will be compared against the Precision criteria established for field measurements in Section A7, Table 5 (page 22) . Since surface water streams can

present a heterogeneous matrix, observations of stream conditions will be recorded and included with the record of results.

B14.2 Laboratory Analysis.

The California Department of Fish and Game-Water Pollution Control Laboratory (CDFG-WPCL) of Rancho Cordova, CA; Mammoth Community Water District (MCWD) Laboratory of Mammoth Lakes, CA; and Moss Landing Marine Laboratory-Marine Pollution Studies Laboratory (MLML-MPSL) of Moss Landing, CA employ laboratory quality assurance and control to ensure the production of analytical data of known and documented usable quality. The CDFG-WPCL, MCWD Lab, and MLML-MPSL have Quality Assurance/Quality Control (QA/QC) Programs and Quality Management Plans, which provide rules and guidelines to ensure the reliability and validity of work conducted at the laboratories. Compliance with the QA/QC Programs are coordinated and monitored by the laboratories' Quality Assurance Managers who are independent of the operating departments.

The stated objectives of the laboratory QA/QC Programs are to:

- Ensure that all procedures are documented, including any changes in administrative and/or technical procedures.
- Ensure that all analytical procedures are conducted according to sound scientific principles and have been validated.
- Monitor the performance of the laboratory by a systemic inspection program and provide for a corrective action as necessary.
- Collaborate with other laboratories in establishing quality levels, as appropriate.
- Ensure that all data are properly recorded and archived.

All laboratory procedures are documented in writing as either Standard Operating Procedures (SOPs) or Method Procedures and are included or web referenced in Appendix B of this document. Internal quality control procedures for analytical services will be conducted by the contracting laboratories in accordance with standard operating procedures and the individual method requirements in a manner consistent with appropriate analytical methods. The laboratories will reanalyze any samples analyzed in non-conformance with the QC criteria, if sufficient sample volume is available and sample holding times are not exceeded. It is expected that sufficient volume of samples will be collected for reanalysis. The analyses will be conducted in accordance with data quality objectives and routine analytical processing of such samples. The laboratories will calculate data quality indicators such as accuracy and bias, precision, and sensitivity as appropriate for their methods and practices. The laboratories are expected to follow the appropriate SWAMP required corrective actions contained in Appendix D of the 2008 revision of the SWAMP guidance "Electronic Template for SWAMP-Comparable Quality Assurance Project Plans" http://www.waterboards.ca.gov/water_issues/programs/swamp/qapp.shtml.

B14.3 Other Quality Control Requirements.

Field duplicate samples will be analyzed to assess the quality of the data resulting from the field sampling program at a rate of 5% of total project sample count per year for each of the parameters measured, as recommended in the 2008 SWAMP QAPP Guidelines, Appendix D. Duplicate samples are analyzed to check for sampling and analytical reproducibility and the precision of all steps after acquisition of the samples. It is planned that field duplicate water samples will be collected for selected analytes during each sampling event at as many sites and for as many parameters, as are needed to attain the required number of duplicates during the project sampling season. The sites chosen for collecting duplicate samples will be ones that are most likely to have detectable concentrations of the

parameters selected for duplicate analysis. For duplicates with a heterogeneous matrix or ambient levels below the reporting limit, failed results may be qualified.

Field blanks samples will analyzed for the trace metals, only, at a rate of 5% of total project sample count per year. If contamination of the field blanks and associated samples is known or suspected, the laboratory should qualify the affected data, and notify the project coordinator and CalTrout Project Director.

Accuracy, the degree to which an observed value and an accepted reference value agree, will be assessed in the field through the adherence to all sample handling, preservation, and holding times. *Precision*, a measure of the degree by which two or more measurements are in agreement, will be assessed in field measurement by assuring proper operation and calibration of field equipment in accordance with manufacturer’s directions. SOPs for field equipment to measure pH, conductivity, DO, turbidity, and temperature are outlined in Section B.11.1: “Field Measurements”.

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. As stated in Section A7: “Quality/Objectives and Criteria for Measurement Data”, it is expected that the CalTrout’s contracting analytical laboratories will provide data meeting QC acceptance criteria for 90 percent or more for all samples tested using the laboratory standard operating procedures. Following completion of the analytical testing, the percent completeness may be calculated by the following equations:

$$\text{Completeness (\%)} = \frac{(\text{Number of valid data}) \times 100}{(\text{Number of samples collected for each parameter analyzed})}$$

All samples for this project are considered. Resampling will be considered if problems are encountered with any parameter associated with the project. It will be the responsibility of the QA/QC Officer to perform sufficient oversight of the project sampling and analytical work to determine whether such action is deemed necessary.

B15. Instrument/Equipment Testing, Inspection, and Maintenance

B15.1 Field Instruments and Equipment.

The field equipment for this project includes meters capable of reading temperature, pH, turbidity, dissolved oxygen, and conductivity and calculating specific conductance, total dissolved solids, and salinity from the instrument’s conductivity and temperature readings. Specific preventative maintenance procedures to be followed for field equipment are those recommended by the manufacturer.

Field water quality measurement instruments will be tested, inspected, and calibrated by the CalTrout Field Manager/Water Quality Specialist at the office of the Owens Valley Indian Water Commission (Commission or OVIWC), which owns the YSI 6820V2 Sonde, before it is carried to the field following procedures detailed in Appendix E and described in Section B16: “Instrument/Equipment Calibration and Frequency” (below). The Calibration Record sheet contained in Appendix E will be filled out during each instrument calibration event. Calibration checks will be performed after each measurement event, and will also be documented on the Calibration Record sheets. The Field Manager will also be responsible for the Sonde’s maintenance. Critical spare parts such as tape, o-rings, and batteries will be kept at the Commission office or with the instrument to minimize instrument down time.

B15.1 Laboratory Instruments.

Each of the three project analytical laboratories (CDFG-WPCL, MCWD Laboratory, and MLML-MPSL) maintains its equipment in accordance with its SOPs, which include those specified by the manufacturer and those specified by the method. These SOPs are attached in Appendix B and have been reviewed by CalTrout's Quality Assurance Officer and found to be in compliance with SWAMP criteria. Any deficiencies found in the equipment will be resolved, equipment re-inspected, and evaluation of the effectiveness of the corrective actions performed according to the laboratory SOPs. All spare parts are stored by the analytical laboratories and are replaced on an as needed basis in case corrective action is required.

Each of the three project analytical laboratories' (CDFG-WPCL, MCWD Laboratory, and MLML-MPSL) laboratory instruments will be maintained according to SOPs outlined in the laboratory QA manuals. As part of their QA/QC Program, routine preventative maintenance programs are conducted by the analytical laboratories to minimize the occurrence of instrument failure and other system malfunctions. Each laboratory has personnel to perform routine scheduled maintenance, and to repair or to coordinate with vendors for the repair of all instruments. All laboratory instruments are maintained in accordance with manufacturer's specifications and the requirements of the specific method employed. This maintenance is carried out on a regular, scheduled basis, and is documented in the laboratory instrument service logbook for each instrument.

B16. Instrument/Equipment Calibration and Frequency

This section describes procedures for maintaining the accuracy of all the instruments and measuring equipment, which are used for conducting field tests and laboratory analyses. These instruments and equipment should be calibrated prior to each use or scheduled on a periodic basis following manufacturer's instructions.

B16.1 Field Instruments/Equipment.

For the Mammoth Lakes Basin NPS project it is planned to use a YSI 6829V2 Sonde and a YSI 650 MDS Display/Logger to measure or generate water quality data in the field, which will be calibrated with sufficient frequency and in such a manner that accuracy and reproducibility of results are consistent with the manufacturer's specifications. The YSI system is capable of determining the following measurements: temperature, pH, optical dissolved oxygen, optical turbidity, and electrical conductivity and using the conductivity and temperature reading to calculate specific conductance, salinity, and total dissolved solids.

Prior to each field event the instrument will be examined to certify that it is in proper operating condition. This includes checking the manufacturer's operating manual and the instructions to ensure that all maintenance requirements are being observed. Field notes from previous sampling trips will be reviewed so that the notation on any prior equipment problem is not overlooked, and all necessary repairs to the equipment have been carried out.

Calibration of field instruments will be performed at the intervals specified by the manufacturer or more frequently as conditions dictate. For this project the YSI probes will be calibrated before each field event and the instrument's calibrations will be checked after each field event. Calibration standard operating procedures (SOPs) for the YSI 6820V2 Sonde are included in Appendix E, which also contains an example of the Calibration Record that is filled out each time a calibration is performed. Calibration Records are filed and stored in the YSI Sonde Maintenance Record notebook maintained in the Bishop,

CA office of the OVIWC, which owns the YSI 6820V2 Sonde. A copy of each Calibration Record generated for a MLB-NPS Project field event will be made and submitted to the CalTrout Project Director for storage with other project documents in the CalTrout office in Mammoth Lakes, CA.

Following SWAMP requirements contained in Appendix D of the 2008 revision of the SWAMP guidance “Electronic Template for SWAMP-Comparable Quality Assurance Project Plans”

(http://www.waterboards.ca.gov/water_issues/programs/swamp/qapp.shtml) if measurements from the YSI 6820V2 fail measurement quality objectives, the instrument will be recalibrated following its manufacturer’s cleaning and maintenance procedures. If measurements continue to fail measurement quality objectives, affected data will not be reported and the instrument manufacturer will be consulted for trouble shooting advice and the instrument will either be repaired in-house or will be returned to the manufacturer for maintenance/repair. All troubleshooting and corrective actions will be recorded in the calibration and field data logbooks.

B16.2 Laboratory Instruments.

Laboratory instrument calibration will be conducted in accordance with the analytical laboratories’ standard operating procedures (Appendix B).

B17. Inspection/Acceptance of Supplies and Consumables

Analytical laboratory supplies and consumables shall be inspected and accepted for use in accordance with the protocol set forth in the contracting analytical laboratories’ QA Manuals and/or QA Plans.

The CalTrout Field Manager or Field Technician shall be responsible for inspection and acceptance of measurement, sampling, and instrument calibration supplies and consumables for use in this project. New supplies and consumables will be acquired for use in this project. Storage and handling of such items will be conducted so as to prohibit contamination.

B18. Non-Direct Measurements

Because this project will generate a data set on water quality in the study area, water quality data from non-direct measurement sources will not be necessary for project implementation or decision making. However, in order to analyze whether existing uses of the subject waters are supported by existing water quality, CalTrout will compare data from this project to existing data sources, such as: the Lahontan Regional Water Quality Control Board (LRWQCB) water quality objectives as set forth in the Basin Plan for Mammoth Creek and Hot Creek. Stream flow data will be acquired from the MCWD from their stream flow gauging sites on Mammoth Creek to support the interpretation of the water quality data. Stream flow data will also be acquired from the Los Angeles Department of Water and Power “395” gauge to ensure accurate information is available for the lower sampling sites (sites 7, 8, & 9). Twenty-four hour precipitation data will be acquired from various weather stations in the Mammoth Lakes Basin, so that field team can accomplish the post-storm sampling event described in Section A6.1. The precipitation data sources will include the National Weather Service, the National Oceanic and Atmospheric Administration’s (NOAA) Meteorological Assimilation Data Ingest System (MODIS), and Mammoth Mountain Ski Area which will be accessed through the MesoWest database website: <http://mesowest.utah.edu/index.html>. The locations of the stream flow gauging stations and the meteorological stations are included in Map 2 (Appendix A, Upper Owens River Water Quality Project, Sampling Sites). The QA/QC measures employed by the data sources are acceptable for the data uses

for this project. The data from sources other than from this project will not be entered into the official SWAMP Information Management System.

B19. Data Management

Data will be maintained as established in Section A9: “Documents and Records”, above. CalTrout will maintain an inventory of data and its forms and will periodically check the inventory against the records in their possession. Raw data from field measurements and sample collection activities will be appropriately recorded either in the field logbook or on loose leaf Field Measurement Data Sheet forms and Water Quality Sampling Data Sheet forms, examples of which are included in Appendix D. The loose leaf forms will be scanned and archived electronically and will be stored in binders in the CalTrout office by the CalTrout Project Data Manager. If the data are to be used in the project reports, they will be reduced or summarized and the method of reduction will be documented in the report.

Data from analytical laboratory analysis of samples will be submitted to CalTrout in hard-copy form and in electronic data files by the laboratories. All data management and handling by the CDFG-WPCL, MLML-MPSL, and MCWD Laboratory will take place in accordance with standard procedures identified in the laboratories’ QA Manuals and SOPs (Appendix B).

Data generated for the Mammoth Lakes Basin NPS project that is delivered in a hard copy format will be organized to facilitate data review and evaluation. The data set will include the data flags provided by the analytical laboratories, as well as additional comments of the Laboratory QA Officer, QA/QC Officer, and/or Project Manager. The laboratory-provided data flags will include such items as: (1) concentration below required detection limit, or (2) estimated concentration due to results below required detection limit. Comments will indicate that the data are: (1) usable as a quantitative concentration, (2) usable with caution as an estimated concentration, or (3) unusable due to out-of-control QC results. The hard copy data reports will be scanned soon after they are received by CalTrout and archived electronically on CalTrout computers and electronic backup devices. The hard copy data will be stored in a secure location in the CalTrout Office for at least one year following the completion of the project

All involved laboratories are specifically requested to flag all data where there is a problem that might impact the usability of the data. Full data packages are not required for this project. As with data flagging, CalTrout will specifically request that the laboratory report quality control data with the sample results.

The laboratory data from the contracting analytical laboratories will also be requested to be delivered to the CalTrout in electronic data files that can be uploaded to CalTrout’s water quality database for storage and for ease in querying for data analysis and reporting to the SWRCB. Project data will also be formatted for upload into the California Environmental Data Exchange Network (CEDEN). Before the end of this project the CalTrout Data Manager will upload all water quality data obtained through implementation of the Mammoth Lakes Basin NPS Project Monitoring Plan (Appendix C) to the CEDEN following guidance for submitting data, including required minimum data elements and data formats, which is available at <http://www.ceden.org> or from the Regional Data Centers identified on the CEDEN website. CalTrout computer data files will be backed up on a regular basis and the backups stored in a secure location for at least five years following the completion of this project.

GROUP C: ASSESSMENT AND OVERSIGHT

C20. Assessments and Response Actions

Assessment of the environmental data collected by CalTrout for the Mammoth Lakes Basin NPS Project will be completed by the CalTrout Project Director/QA Officer in cooperation and collaboration with the Field Manager/Water Quality Specialist after the data are received from the analytical laboratories. The data assessment by the Project Director/QA Officer will be based on the criteria that the sample was properly collected and handled according to the Group B sections of this QAPP. The Project Director/QA Officer will conduct a systematic review of the data for compliance with the established QC criteria based on the duplicate results provided by the laboratory. An evaluation of data will be presented to the SWRCB in report form as necessary for quarterly reporting requirements.

The data review will identify any out-of-control data points and data omissions and interact with the laboratory to correct data deficiencies. Decisions to repeat sample collection and analyses may be made by the QA Officer based on the extent of the deficiencies and their importance in the overall context of the project.

C21. Reports to Management

Quarterly progress reports will be provided by the CalTrout Project Director to the SWRCB Grant Manager by the 20th of the month following the end of each quarter year of the project. The progress reports will provide a brief description of the work performed, accomplishments during the quarter, milestones achieved, monitoring results (if applicable), and any problems encountered in the performance of the work under the grant agreement. Following quarters when sampling occurs or when laboratory results are received, the quarterly progress report will address all quality assurance issues. Before the end of the project a draft and a final project report will be prepared by the CalTrout Project Director and submitted to the State Board Grant Manager. The draft and final reports will contain QA sections that summarize data quality information collected during the project including identification of the stream reach affected by these activities.

Copies of analytical laboratory reports will also be provided for review by the SWRCB Grant Manager upon request.

GROUP D: DATA VALIDATION AND USABILITY

D22. Data Review, Verification, and Validation

Analytical Laboratory data will be reviewed, validated and verified according to standard procedures outlined in the analytical laboratories' QA Manuals. Analytical laboratory data review and validation will be performed by the analyst and Laboratory QA Officer prior to release to CalTrout. The data set will include the data flags provided by the analytical laboratories, as well as additional comments of the Laboratory QA Officer, QA/QC Officer, and/or Project Manager. The laboratory-provided data flags will

include such items as: (1) concentration below required detection limit, or (2) estimated concentration due to results below required detection limit. Comments will indicate that the data are: (1) usable as a quantitative concentration, (2) usable with caution as an estimated concentration, or (3) unusable due to out-of-control QC results.

Data generated by project activities will be reviewed against the data quality objectives cited in Section A7 and the quality assurance/quality control practices cited in Sections B14, B15, B16, and B17. Data will be separated into three categories: 1) data meeting all measurement quality objectives, 2) data meeting measurement quality objectives but failing quality assurance/quality control practices, and 3) data failing to meet accuracy criteria. Data meeting all measurement quality objectives, but with failures of quality assurance/quality control practices will be set aside until the impact of the failure on data quality is determined. Once determined, the data will be moved into either the first category or the last category.

Data falling in the first category are considered usable by the project. Data falling in the last category are considered not usable. Data falling in the second category will have all aspects assessed. If sufficient evidence is found supporting data quality for use in this project, the data will be moved to the first category, but will be flagged with a "J" as per EPA specifications.

D23. Verification and Validation Methods

Raw data from field measurements and sample collection activities will be appropriately recorded in the field logbook or on data forms as described in Section B12.2: "Field Logbooks/Data Sheets/Documentation", and these data records will be checked visually by the Field Manager. As the data are entered into the CalTrout water quality database all data records will be checked visually by the Field Manager or the Data Manager. The CalTrout Project Director/QA Officer will perform a check of at least 10% of the data. If the data are to be used in the project reports, they will be reduced or summarized and the method of reduction will be documented in the report.

The analytical laboratories will perform in-house analytical data reduction and validation under the direction of their Laboratory QA Manager and/or Laboratory Manager. The Laboratory QA Officer/Laboratory Manager is responsible for assessing data quality and advising of any data that were rated "preliminary" or "unacceptable" or other notations, which would caution the data user of possible unreliability. Data reduction, validation, and reporting by the laboratory will be conducted as follows:

- Raw data produced by the analyst is turned over to the analytical laboratory's Project Manager.
- The analytical laboratory Project Manager and CalTrout Field Manager in conjunction with the CalTrout QA Officer will decide whether any sample reanalysis is required.
- Upon acceptance of the preliminary reports by the Laboratory QA Officer, final reports will be generated and signed by the Laboratory Project Manager. The laboratory package shall be presented in the same order in which the samples were analyzed.

The laboratory will report the data in the same chronological order in which it was analyzed along with QC data. The laboratory will provide the following information to the CalTrout Project Director in each analytical data package submitted:

- Cover sheets listing the samples included in the report and narrative comments describing problems encountered in analysis.
- Tabulated results of inorganic and organic compounds identified and quantified.

The CalTrout Project Director/QA Officer and Field Manager will conduct a systematic review of the data for compliance with the established QC criteria based on the duplicate results provided by the laboratory. An evaluation of data accuracy, precision, sensitivity and completeness, based on criteria in Section B, may be performed and presented to the SWRCB Grant Manager in report form.

Issues will be noted. Reconciliation and correction will be done by a committee composed of CalTrout's Project Director/QA Officer and Field Manager, and the analytical laboratory's QA Officer and Laboratory Director. Any corrections require a unanimous agreement that the correction is appropriate

The Data Review will identify any out-of-control data points and data omissions and interact with the laboratory to correct data deficiencies. Decisions to repeat sample collection and analyses may be made by the CalTrout QA Officer based on the extent of the deficiencies and their importance in the overall context of the project.

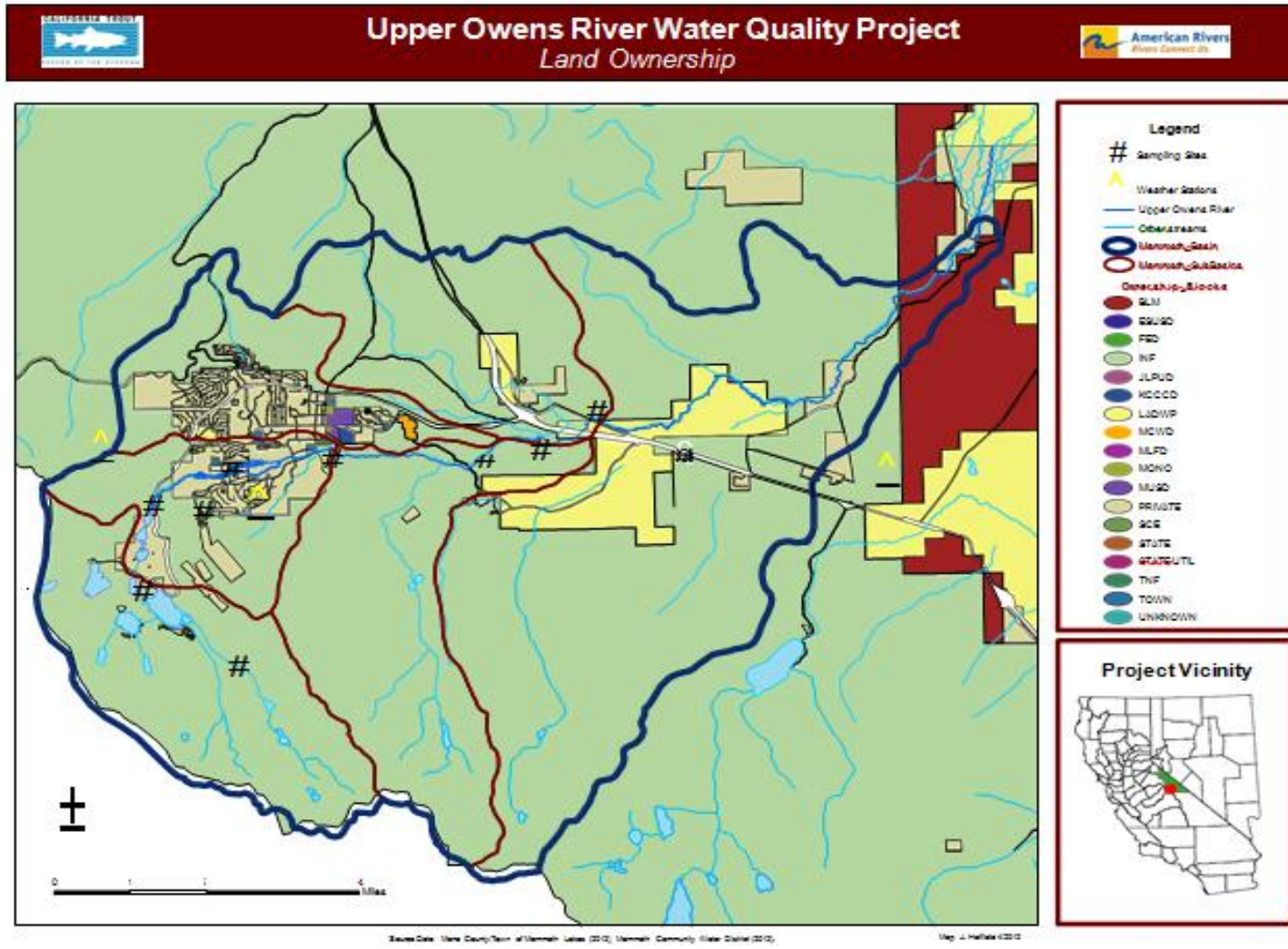
D24. Reconciliation with User Requirements

Results obtained from the project will be summarized, after data review, validation and verification, for use by CalTrout and the SWRCB. Monitoring data will be analyzed with respect to Lahontan Regional Water Quality Control Board water quality objectives, which can be found at (http://www.waterboards.ca.gov/lahontan/water_issues/programs/basin_plan/docs/ch3_wqobjectives.pdf). The water quality monitoring data will be analyzed via tables and charts to look for data anomalies or relationships that indicate possible contamination from either nonpoint sources of pollution, or in the case of the total mercury and total manganese, possible point sources of pollution. Trends will be evaluated whenever sufficient baseline data exist. If nonpoint pollution is identified by this project, a companion document produced in collaboration with the TAC will recommend control measures for reducing loads. The companion document will include calculations of expected load reductions from each suggested management practice. If a point source of pollution is identified by this study the results will be reported to the SWRCB by the Project Director for SWRCB regulation. Validated data collected as part of this project will be submitted to the CEDEN through the appropriate SWAMP Data Center as described in Sections A9: "Documents and Records" and B19: "Data Management".

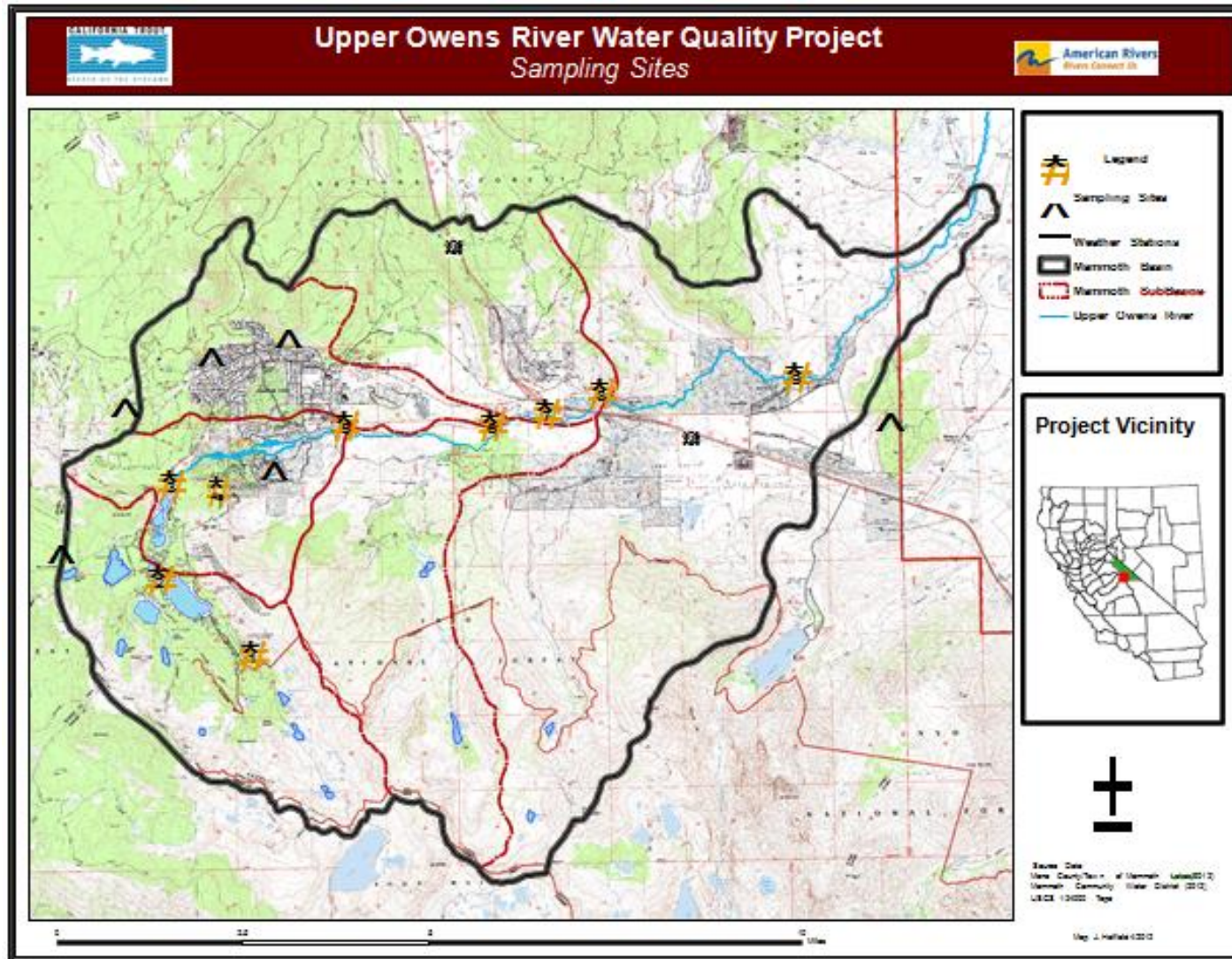
After the first few sampling events have occurred and the water quality data are received from the analytical laboratories it is possible that additional sampling sites or parameters may be identified to investigate the nonpoint pollution contributing reach. The location of any additional sampling sites will be determined in consultation with the TAC and SWRCB and with the help of specific GIS analysis using flowline data from the National Hydrography dataset. During the later part of the project some of the initial sampling sites and parameters may be abandoned so that the sampling effort can become focused on more detailed investigation into any high pollutant source watersheds within the Mammoth Lakes Basin that were identified during the first sampling events. If such changes in monitoring are indicated the Monitoring Plan and QAPP will be revised to reflect necessary changes in appropriate sections and elements of the documents and resubmitted to the SWRCB for approval before the changes are made.

Appendix B1

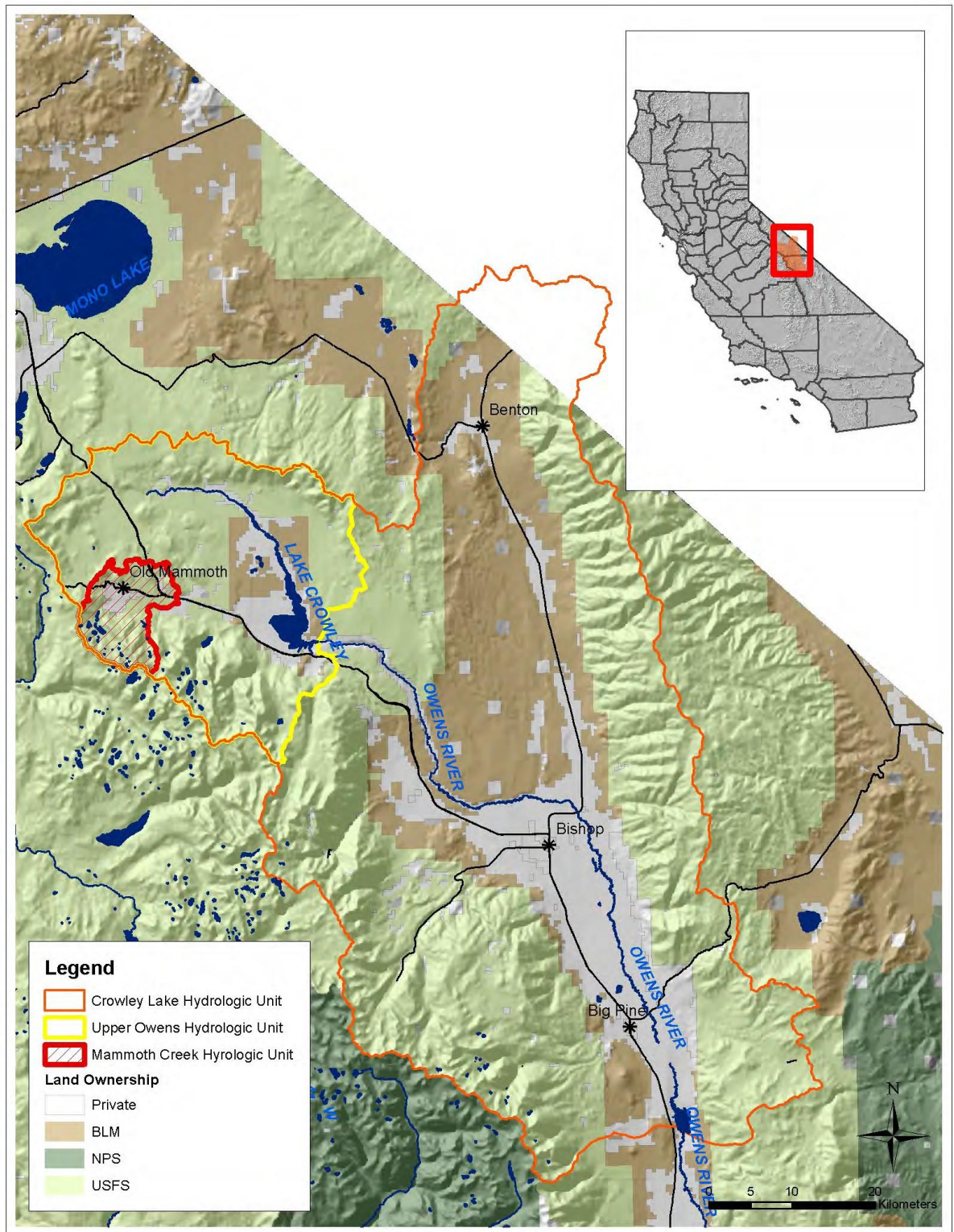
Map 1 – Upper Owens River Water Quality Project, Land Ownership



Map 2 – Upper Owens River Water Quality Project, Sampling Sites



Map 3 – Project Area in Watershed Context



Appendix B2

Water Quality Laboratory Associated QAPPs, QA Documents, Methods and/or Standard Operating Procedures (SOPs)

California Department of Fish and Game – Water Pollution Control Laboratory (CDFG-WPCL)

Laboratory Quality Assurance Document:

Surface Water Ambient Monitoring Program Quality Assurance Program Plan 09/01/08 (see http://www.waterboards.ca.gov/water_issues/programs/swamp/docs/qapp/swamp_qapp_master090108a.pdf)

Laboratory Analysis Methods:

SOP: WPCL-AA-069, Total Nitrogen in Water, FIA, Revision:3, Revision Date: 09/02/10 (see Appendix B)

SOP: WPCL-AA-018, N, NO3-NO2 in Water, FIA, Revision:4, Revision Date: 12/15/09 (see Appendix B)

CDFG Fish and Wildlife Water Pollution Control, Laboratory Standard Operating Procedure for the Determination of Total Phosphorus in Water Using Persulfate Digestion by Flow Injection Analysis Colorimetry, Revision # 8, Date: 02/13/09 (see Appendix B)

Mammoth Community Water District Laboratory (MCWD Lab)

Laboratory Quality Assurance Document:

Mammoth Community Water District, Mammoth Lakes, CA, Laboratory Quality Assurance Document, 2011: (see Appendix B)

Laboratory Analysis Methods:

MCWD Lab – Standard Operating Procedures – Total Dissolved Solids (see Appendix B) MCWD Lab – Standard Operating Procedures – Total Suspended Solids (see Appendix B)

Moss Landing Marine Laboratory – Marine Pollution Studies Laboratory (MLML-MPSL)

Laboratory Quality Assurance Document:

MPSL MLML Laboratory QAP, Revision 5. February, 2006

Laboratory Analysis Methods:

EPA Method 1631, Revision E: Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry, August 2002 (see http://water.epa.gov/scitech/methods/cwa/metals/mercury/upload/2007_07_10_methods_method_mercury_1631.pdf)

Modification of EPA Method 1631e, Marine Pollution Studies Lab (see Appendix B)

EPA Method 1638, Determination of Trace Elements in Ambient Waters by Inductively Coupled Plasma — Mass Spectrometry, January 1996 (see http://water.epa.gov/scitech/methods/cwa/bioindicators/upload/2007_07_10_methods_method_1638.pdf)

Modification of EPA Method 1638, Marine Pollution Studies Lab (see Appendix B)

STANDARD OPERATING PROCEDURE
TITLE: Total Nitrogen in Water by Persulfate Digests by Colorimetric Flow Injection Analysis
(Block Digester Method)

REVISION HISTORY		
Revision #	Summary of Changes	Date
3	<p>Changed preparation interval for the stock check standard 1 (nicotinic acid, 6.12.1) from 6 months to monthly and the working check standard (6.12.2) from monthly to bi-monthly.</p> <p>Changed the preparation interval for the mid-point standard CRM stock (6.12.3) to bi-weekly and the working CRM to weekly.</p> <p>Added definition of calibration blanks (8.1.2).</p>	09/02/10
2	<p>Reformatted and renumbered SOP. Added criteria and corrective actions for calibration and QC samples. Added QC summary attachments for enforcement testing vs. non-enforcement testing. Changed reporting limit from 0.030 to 0.050 mg/L as N and updated MDL. Midrange standard was changed from 0.25 to 0.20. Glassware cleaning procedure, valve timing, storage time to analysis changes. Removed residual criteria.</p>	01//13/10
1	<p>Lower reporting limit; changed from 0.2 mg/L to 0.030 mg/L as N.</p>	10/21/09
0	<p>Initial release as Method 69.</p>	06/03/09

Author:	<i>Patricia Bucknell</i>	Date:	09/02/10
Approved:	Laboratory Director <i>David B. Crane</i>	Date:	
Approved:	Section Lead <i>Kendall Penney</i>	Date:	
Approved:	Quality Assurance <i>Gail Cho</i>	Date:	
Approved:	Health and Safety <i>Thomas Lew</i>	Date:	

STANDARD OPERATING PROCEDURE

TITLE: Total Nitrogen in Water by Persulfate Digests by Colorimetric Flow Injection Analysis (Block Digester Method)

1.0 Scope and Application

- 1.1 This method covers the determination of total nitrogen in surface, ground, and waste waters after an acidic persulfate digestion.
- 1.2 The applicable range is 0.050 to 1.00 mg N/L. The method detection limit (MDL) is 0.020 mg N/L. Approximately 60 samples per hour can be analyzed.

2.0 Summary of Method.

- 2.1 This method utilizes an off-line digestion to convert all forms of nitrogen into nitrate using an alkaline and acidic persulfate digestion. In this digest, the potassium persulfate in an alkaline environment converts all forms of nitrogen-containing compounds to the nitrate form. Nitrate is quantitatively reduced to nitrite by passage of the sample through a copperized cadmium column. The nitrite thus produced is then determined by diazotizing with sulfanilamide followed by coupling with a buffer solution. The resulting water-soluble dye has a magenta color which is read at 520 nm.

3.0 Interferences and Comments.

- 3.1 Residual chlorine can interfere by oxidizing the reactor column.
- 3.2 Glassware contamination is a problem in low level nitrogen determinations. Soak digestion tubes in phosphate-free soap, and then scrub the inside before loading them in the dishwasher. After being washed, digestion tubes should be filled with TraceClean® HCl, covered and allowed to sit overnight. After soaking overnight, rinse with de-ionized water and then drain. Put the tubes open side up in a wire rack and put in a 103°C oven for one hour. Tubes are ready to use after cooling.
- 3.3 Samples containing bromide can lead to errors in the phosphate measurement. The bromine formed in the digestion may cause double peaks in low-level samples. Soaking these samples with helium for approximately 10 minutes will remove this interference.
- 3.4 Reagent grade chemicals may contain nitrogen. Use the best quality reagents possible to avoid large blank peaks, which can affect detection limits.
- 3.5 Samples and standards must be carried through the entire digestion procedure for this method.
- 3.6 Review batch quality controls and analytical sequence quality controls within a time frame that allows for any necessary sample reanalysis within holding times.

4.0 Safety

- 4.1 Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. At a minimum, wear gloves, lab coats, and safety eyewear to minimize contact with chemicals.
- 4.2 The following chemicals have the potential to be highly toxic or hazardous; for detailed explanation consult the MSDS.
 - 4.2.1 Cadmium granules.
 - 4.2.2 Ammonium hydroxide
 - 4.2.3 Sodium hydroxide.
 - 4.2.4 Phosphoric acid
 - 4.2.5 Sulfanilamide
 - 4.2.6 Sulfuric acid.
 - 4.2.7 Potassium persulfate
- 4.3 Dispose of wastes and reagents according to vwp L-EH-049 "Disposal of Hazardous Wastes."

5.0 Equipment and Supplies

- 5.1 Balance -- analytical, capable of accurately weighing to the nearest 0.0001 g, calibrated prior to use.
- 5.2 Glassware -- Class A volumetric flasks and pipettes or plastic containers as required.
- 5.3 Eppendorf pipettes and pipet tips.
- 5.4 Flow injection analysis equipment designed to deliver and react sample and reagents in the required order and ratios.
 - 5.4.1 Sampler.
 - 5.4.2 Multichannel proportioning pump.
 - 5.4.3 Reaction unit or manifold.
 - 5.4.4 Colorimetric detector.
 - 5.4.5 Data system
 - 5.4.6 10 mm, 80 uL, glass flow cell.
 - 5.4.7 520 nm interference filter.
- 5.5 Special Apparatus.
 - 5.5.1 Heating unit.
 - 5.5.2 Block Digester.
 - 5.5.3 Labware for digestion. For the BD-46, 25 x 150 mm Corning® Screw-capped tubes. Caps should have rubber liners
 - 5.5.4 Vortex mixer.
 - 5.5.5 Glass calibration vials must be used. Lachat per no. 21304, XYZ samplers.

6.0 Reagents and Standards.

Label all reagents and standards with identity, tracking number, concentration, preparation date, expiration date, preparers' initials.

6.1 Reagent water.

6.1.1 Use ASTM Type II water for all solutions. (See Standard Specification for Reagent Water D1193-77 for more information).

6.2 Reagent 1: 15N Sodium Hydroxide.

6.2.1 In a 250 mL volumetric flask add 150 g NaOH to 50 mL Milli-Q (MQ) water.. CAUTION: The solution will get very hot! Swirl until dissolved. Dilute to mark. Label, cool and store in a plastic bottle.

6.3 Reagent 2: Ammonium Chloride Buffer, pH 8.5.

6.3.1 **CAUTION: FUMES!!!** In a hood, using a 4 liter beaker add 1 liter Milli-Q water, 210 mL concentrated hydrochloric acid (HCL), 190 mL ammonium hydroxide (NH₄OH), and 2.0 g disodium EDTA. Let solution mix and cool. Adjust pH to 8.5 with concentrated HCL or 15 N sodium hydroxide solution. Transfer to a 2 liter volumetric flask and dilute to mark. Invert to mix. This solution does not have an expiration date; therefore, it is good for 6 months per Dave Crane.

6.4 Reagent 3: Sulfanilamide Color Reagent

6.4.1 To a 1 liter volumetric flask, add about 500 mL Milli-Q water. then add 100 mL of 85 % phosphoric acid (H₃PO₄), 40.0 g sulfanilamide, and 1.0 g N-(1-naphthyl)ethylenediamine dihydrochloride (NED). Shake to mix, and stir for 30 min. to dissolve. Dilute to the mark, and invert to mix. Store in an amber glass bottle and refrigerate. This solution is stable for one month.

6.5 Reagent 4: Stock 11N Sulfuric Acid

6.5.1 To an acid washed 1 liter volumetric flask , add 500 mL of Milli-Q water and place in a tub that has cold water in it. Gradually add 305 mL of concentrated sulfuric acid. CAUTION, solution will be hot! Stir to mix, cool to room temperature and dilute to volume. Label. Do not degas this reagent! Prepare monthly.

6.6 Reagent 5: Carrier, Sulfuric Acid, 0.231M

6.6.1 In an acid-washed 500 mL volumetric flask, add 200 mL water and 21 mL Reagent 4 (11N H₂SO₄). Dilute to the mark with DI water and invert to mix. Degas daily. Prepare fresh weekly.

- 6.7 Reagent 6: Basic Digestion Reagent (Digestion Reagent 1).
- 6.7.1 In a 500 mL acid washed volumetric flask dissolve 5.24 g sodium hydroxide (NaOH) and 21 g potassium persulfate ($K_2S_2O_8$), in approximately 800 mL DI water. Dilute to the mark and invert to mix. Prepare fresh monthly and store in plastic. Do not degas this reagent!
- 6.8 Reagent 7: Acidic Digestion Reagent (Digestion Reagent 2)
- 6.8.1 In a 250 ml volumetric flask, add 100 mL Reagent 4 (11N H_2SO_4) and 5.75 g potassium persulfate ($K_2S_2O_8$). Dilute to the mark with Reagent 4 (11N H_2SO_4) and invert to mix. Prepare fresh weekly. Do not degas this reagent!
- 6.9 Reagent 8: 0.5N Sodium Hydroxide.
- 6.9.1 To a 1 L plastic bottle containing 500 mL of DI water, add 20 g NaOH. Swirl until dissolved. Dilute to the mark. Prepare monthly.
- 6.10 Reagent Ultra Pure 1M HCl (manifold rinse). Approximately 10 mL.
- 6.11 Digested Standards.
- 6.11.1 Standard 1: Stock Nitrate Standard 10.0 mg/L NO_3 as N.
- 6.11.1.1 In a 50 mL FIA standard tube dilute 0.50 mL of 1000 mg-N/L Nitrate as N purchased standard with Milli-Q water. Dilute to the mark with Milli-Q water and invert to mix. This standard is stable for one month. Note: This standard must also be digested in case of having to make post matrix spikes.
- 6.11.2 Working Standards
These standards are prepared from Standard #1 (6.10.1) 10.0 mg/L NO_3 .as-N and will be digested. Standards are prepared in Milli-Q water and carried through the digestion procedure with the samples. Using simulated standards is not recommended due to nitrogen and phosphorus present in the digestion reagents. **For the digested standards and samples to work with this method, they must be carried through the entire digestion procedure.**
- 6.11.2.1 Prepare standards of the following concentrations from Standard 1: Stock Nitrate Standard, 10.0 mg/L NO_3 as N (6.10.1) mg/L. Prepare standards every two weeks.

—Table 1: Preparation of Working Standards —

Working Standards concentration in mg/L NO ₃ as N	Volume (mL) of Standard 1 (10.0 mg/L NO ₃ as N) in 50 mL
1.00	5.0
0.50	2.50
0.20	1.00
0.10	0.50
0.050	0.25
0.00	0.00

6.12 Digestion Check Standards.

6.12.1 Stock Check Standard #1, 100 mg N/L Nicotinic acid p-toluenesulfonate

6.12.1.1 In a **200 mL** volumetric flask add 0.4217 g Nicotinic acid p-toluenesulfonate in about 100 mL MQ water. Dilute to the mark with MQ water and invert to mix. Store in a plastic bottle. Prepare fresh every month.

6.12.2 Working Check Standard 0.200 mg N/L Nicotinic acid p-toluenesulfonate

6.12.2.1 In a 200 mL volumetric flask dilute 0.40 mL of Stock Check Standard #1 to the mark with MQ water. Invert to mix. Store in a plastic bottle. Prepare fresh bi-monthly.

6.12.3 Mid-point Standard, second source from ERA or AllTech.

6.12.3.1 CPM Anions Mix XX-XXX Midpoint \pm 50% of 0.200 mg/L. Prepare the stock fresh bi-monthly and the working standard weekly.

7.0 Preservation and Holding Times.

7.1 Samples should be collected in trace clean plastic bottles. Volume collected should be sufficient to insure a representative sample, allow for replicate analysis, and minimize waste disposal.

7.2 Samples may be preserved by adjusting the pH \leq 2 with concentrated H₂SO₄ and stored at <6°C. Acid preserved samples have a holding time of 28 days.

7.3 Sample digests should be run within 24 hours of digestion.

8.0 Calibration and Standardization.

8.1 Lachat QuikChem 8500/8000 Data System Parameters. The timing values listed below are approximate and will need to be optimized using graphical events programming.

Low Range

Sample throughput:	38 samples/h, 60.4 s/sample
Pump Speed:	35
Cycle Period:	60.4
Analyte Data:	
Concentration Units:	(mg as N)/L
Peak Base Width:	111 s
Inject to Peak Start:	64.0 s
Chemistry:	Direct

8.2 Calibration curve.

8.2.1 Curve concentrations:

Level	1	2	3	4	5	6
Concentration: mg as N/L	1.0	0.50	0.20	0.10	0.050	0.00

Calibration Rep Handling:	Average
Calibration Fit Type:	2 nd Order Polynomial
Weighting Method:	1/X
Force through zero:	No

8.2.2 $R^2 \geq 0.995$.

8.2.3 If R^2 criteria is not met, review standards for preparation errors, mis-injections, instrument malfunctions, etc. Also review chromatography for acceptability. Prepare new standard or calibration curve if corrective actions are ineffective.

8.2.3.1 An analyst may omit one of the following standards if preparation or injection errors are suspected: the 2nd lowest standard, the penultimate high standard or the highest standard may be omitted. Only one standard may be omitted from a curve. Be aware of the impact on the quantification range.

8.2.3.2 A curve with a minimum of 4 points is required for quantification of analyte.

8.2.3.3 Refer to WPCL-AA-068 "Manual Chromatographic Peak Integration" for manual integration practices.

8.2.3.4 Document actions on the raw data.

- 8.3 Sampler Timing:
- | | |
|----------------------------|--------|
| Min. Probe in Wash Period: | 41.2 s |
| Probe in Sample Period: | 19.3 s |
- 8.4 Valve Timing:
- | | |
|-----------------|--------|
| Time to Valve*: | 22.9 s |
| Load Period: | 12.0 s |
| Inject Period: | 83.0 s |

* Must be determined.

8.5 Total Nitrogen Manifold Diagram. See Figure 1.

8.6 Continuing calibration and reporting limit check.

8.6.1 Calibration blanks are comprised of aliquots of the digested method blank. Digest enough lab blank water to account for instrument verifications.

8.6.2 Initial Calibration Verification (ICV) and Initial Calibration Blank (ICB): Immediately after calibration, analyze a mid-range standard followed by analysis of an ICB. Note that some monitoring programs prefer the analysis of a CRM/SRM in place of an ICV. See Attachments 2 and 3 for acceptance and corrective actions.

8.6.3 Continuing Calibration Verification (CCV) and Continuing Calibration Blank (CCB): after every 10 analyses (i.e. injections) and end of run, analyze a mid-range standard followed by a CCB. See attachments 2 and 3 for acceptance and corrective actions.

8.6.4 Reporting limit check: After calibration and prior to sample analysis, analyze a digested 0.050 mg/L as N standard. See attachments 2 and 3 for acceptance and corrective actions.

9.0 Procedure

9.1 Digestion Procedure.

9.1.1 Both standards and samples should be carried through this procedure. If samples have been preserved with sulfuric acid, standards and blanks should be preserved in the same manner.

9.1.2 The clean glass screw capped sample tubes must be soaked in 1:1 HCL for about 1 hour and thoroughly triple-rinsed with de-ionized water prior to use. After rinsing put them in a wire rack, bake in a 103-105°C oven for 1 hour. After the tubes have cooled they are ready for use. The caps must also be triple-rinsed with DI water, and dried in a 103-105°C oven for about 1 hour before use.

- 9.1.3 Set the block digester to 160°C. Allow the digester to warm up to 150°C.
- 9.1.4 To each of digestion vessels containing 20 mL of sample, add 5 mL of the basic digestion reagent (Reagent 6). Screw the caps on tightly. If the liquid in the tubes boils, either the cap is not on tightly, or there is a chip in the rim of the tube. Do not retighten caps after being placed into the digestion block.
- 9.1.5 Digest samples in the block digester for 30 minutes at 150°C.
- 9.1.6 After the first part of the digestion is completed, remove the tubes VERY CAREFULLY and cool until they can be comfortably handled.
- 9.1.7 When the tubes can be comfortably handled, add 0.5 mL of the acidic digestion reagent (Reagent 7).
- 9.1.8 Tightly recap the tube and vortex to mix. Place the tubes back into the block at 150°C for another 30 minutes.
- 9.1.9 Remove the samples from the block and allow 5 minutes to cool. CAUTION: Internal pressure for hot tubes is about 20 psi. If dropped, they will explode!
- 9.1.10 Vortex the contents and decant into tubes for measurement.
- 9.1.11 Samples can be tested up to 24 hours after digestion if capped tightly and kept refrigerated.

9.2 Start-Up Procedure

- 9.2.1 Degassing: The solutions will degas on their own if they are allowed to sit at room temperature slightly vented overnight.
 - 9.2.1.1 Alternative degassing with Helium: To prevent air spikes use helium to degas all solutions except the standards. Use He at 140kPa (20 lb/in²) through a helium degassing tube (Lachat Part No. 50100). Bubble helium through the solution for one minute.
 - 9.2.1.2 Thoroughly rinse the acid washed probe prior to and after degassing each reagent to avoid contamination of subsequent reagents.
 - 9.2.1.3 Blow out excess water by turning on the helium after taking the probe out of the graduated cylinder and wipe down the probe and probe end with a Chemwipe.
- 9.2.2 Turn instrument on at the power strip located on wall behind FIA.
- 9.2.3 Prepare any reagents and/or standards as stated in section 6.0.
- 9.2.4 Set up manifold as shown in Figure 1.

- 9.2.5 Turn on the computer and printer.
- 9.2.6 On the Windows desktop, click the OMNION 3.0 icon.
- 9.2.7 Once in OMNION 3.0, pull down the FILE menu to OPEN.
- 9.2.8 To start a new run, open a previous run by the date and time the run took place.
- 9.2.9 The data system parameters in 8.1 are determined and save the time of the initial method set up. Changes should not be required except in the case of fine tuning.
- 9.2.10 Once a previous run has been opened, change the old sample schedule to reflect the current sample schedule.
- 9.2.11 Clean the outside of the tubing prior to placing into MQ water. Lock down pump tubing and adjust tension by pushing latch on top of pump tube holder all the way back towards the manifold. Once latch is all the way back, bring it forward one click. Start pump by pressing NORMAL RUN.
- 9.2.12 Check for leaks and smooth flow. Watch lines for pulsating action and listen to the pump for a squeaky noise. Either of these conditions may indicate a plugged line within the system. Refer to the troubleshooting section in the QC8000 FIA Users Manual, pgs. 143-188, if either of these conditions occurs.
- 9.2.13 Remove reagent lines from MQ water and place in appropriate reagents. Place the sample probe rinse line into the carrier container. Allow system to equilibrate to achieve a steady baseline. Open the cadmium column by switching the column valve to the on-line position. Toggle the Preview icon to see baseline visually.
- 9.2.14 Place the standards in the auto sampler rack from high to low concentration. Double check their position compared to the cup number listed on the schedule.
- 9.2.15 Pour samples into the 13 X 100 mm test tubes and load them into sample rack. Double check the schedule sample cup numbers against the actual sample rack.
- 9.2.16 Check to see that the sample probe rinse reservoir is receiving water and is continually upwelling.
- 9.2.17 Examine waste lines to see that three lines are dripping.

9.2.17.1 Three lines drip into the waste container; one line for each of the two channels and one from the valve waste.

9.2.18 To start the run, click START on the OMNION menu.

9.3 Shutdown Procedure.

9.3.1 Switch the cadmium column valve to the off-line position before removing the lines from the reagents.

9.3.2 Remove all reagent lines and place in a beaker of MQ water, rinsing the outside of the lines first.

9.3.3 Replace initial rinse water with fresh MQ water and allow this to be pumped through the manifold for approximately 10 minutes.

9.3.4 Pump the lines dry by removing lines from rinse water.

9.3.5 Empty auto sampler waste receptacle and replace the hazardous waste receptacle with the regular waste receptacle.

9.3.6 Shut down the computer through the Windows menu.

9.3.7 Turn instrument off at power strip.

10.0 Data Analysis and Calculations.

10.1 Once the instrument has run the standards, a calibration curve is prepared by plotting response versus standard concentration. Sample concentration is calculated from the regression equation.

10.1.1 Click on the graph icon along the left side of the channel display box. The calibration curve and the correlation coefficient are displayed.

10.2 Report only those values that fall between the lowest and the highest calibration standards. Samples exceeding the highest standard should be diluted and reanalyzed.

10.3 Once the run has ended, check the integration of the peaks on the channel display.

10.3.1 It is helpful to enlarge the scale of the display in order to view the encompassment of the peaks.

10.3.1.1 Use the buttons on the left side of the channel display to zoom in and out of the x and y axis.

10.3.2 To adjust the window in which the software looks for the peak, right click the mouse on the channel data display.

- 10.3.2.1 Use the peak of the highest standard when adjusting window. The area encompassing the largest peak should also take care of the smallest peaks.
 - 10.3.2.2 Select ADJUST PEAK EXPECTATION WINDOW.
 - 10.3.2.3 Drag the sides of the active box far enough out to encompass entire peak.
 - 10.3.2.4 Right click again and select RERUN PEAK DETECTION. The software will reintegrate the peaks and display the corrected values in the run report.
- 10.3.3 To omit a calibration standard from the run, see 8.2.
- 10.3.3.1 Click the graph icon on the left side of channel display.
 - 10.3.3.2 In the column for UNUSED calibration standards, click the box which corresponds to the standard to be omitted.
 - 10.3.3.3 Go back to channel display and right click mouse. Select RERUN PEAK DETECTION. The data has now been reintegrated, omitting the flawed standard.
 - 10.3.3.4 Document all actions on raw data.
- 10.4 To print results, pull down the tools menu and select CUSTOM REPORT. Click on the yellow icon (cheese) along top of screen.
- 10.5 Select the CALIBRATION box and CHANNEL DATA DISPLAY box. This will print the calibration curve, the channel display and the sample results.
- 10.6 Report sample results for total nitrogen in (mg as N)/L.
- 11.0 Quality Control.**
- 11.1 A batch is defined as up to and including 20 samples of the same matrix processed together. A method blank, laboratory blank, reference standard, laboratory control spike, matrix spike, matrix spike duplicate, and sample duplicate should be analyzed for every 20 samples, sample batch or different sample matrix.
 - 11.2 Method Blank – A digested blank is run at the beginning of the batch to demonstrate freedom from contamination during the digestion procedure. NOTE: Several method blanks must be digested because method blanks are used to dilute samples and are used as instrument calibration blanks.
 - 11.3 SRM/CRM or ICV: A quality control (QC) check standard is used to verify that calibration standards have been made properly. Thus, the reference standard must be made from a different source than the calibration standards. See Attachments 2 and 3 for frequency, acceptance criteria, and corrective actions.

- 11.4 Laboratory control sample (LCS): One per batch. Control limit: 90-110%. Reanalyze LCS. Re-digest and reanalyze all associated samples and QC as necessary.
- 11.5 Matrix spike/matrix spike duplicate (MS/MSD). One pair per batch. Control limit: 80-120%, RPD (relative percent difference): $\pm 25\%$. Reanalyze and compare to LCS to assess matrix interferences.

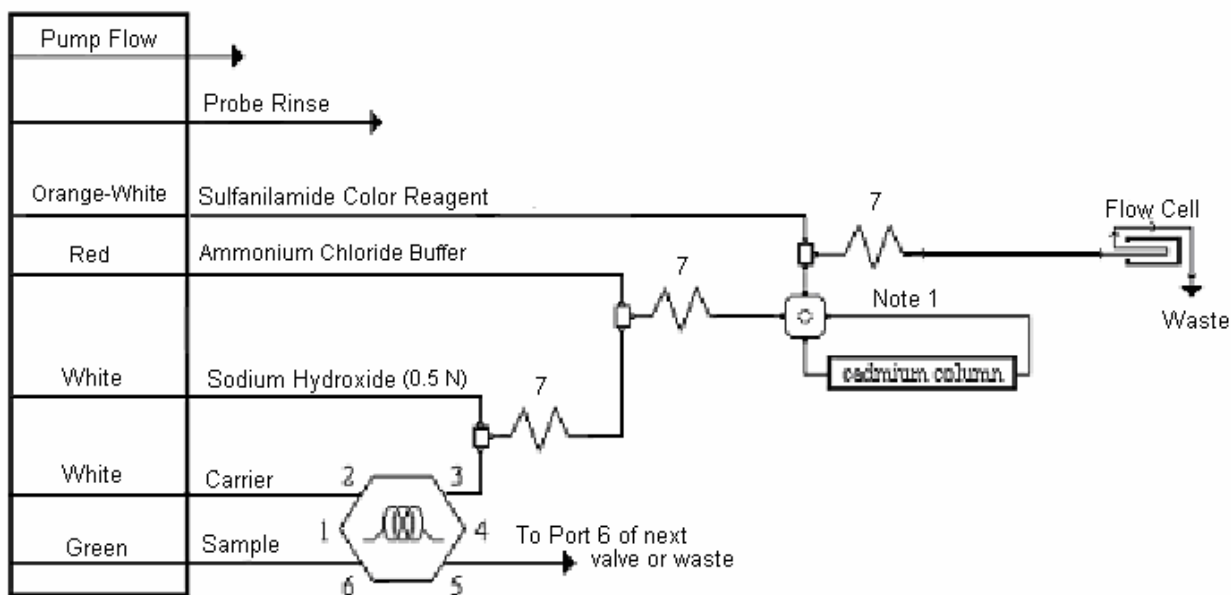
12.0 References.

- 12.1 U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, EPA-600/R-93/100, Rev. 2.0, Revised August 1993, Method 53.2.
- 12.2 Methods for Determination of Inorganic Substances in Water and Fluvial Sediments. Book 5. Chapter A1. U.S. Department of the Interior, U.S. Geological Survey, Method I-2601-78.
- 12.3 Lachat Instruments, QuikChem Method 10-107-01-4-B revised by Scott Tucker and Dennis Jones on 22 June 2007.
- 12.4 Dennis Jones, North Dakota Department of Health, SOP I-1-46, Rev 3-20-03.
- 12.5 WPCL-EH-049, "Standard Operating Procedure for the Handling, Storage, and Disposal of Hazardous and General Laboratory Waste."
- 12.6 WPCL-AA-068, "Manual Peak Integration and Calibration for FIA".

13.0 Attachments.

- 13.1 Figure 1: Total Nitrogen Manifold Diagram.
- 13.2 Attachment 1: Calibration and QC Summary for Compliance and Enforcement Testing.
- 13.3 Attachment 2: Calibration and QC Summary for Monitoring Projects.

Figure 1: Total Nitrogen Manifold Diagram



Carrier: 0.213 M sulfuric acid (Reagent 5)

Manifold Tubing: 0.5 mm (0.020 in) i.d.

QC8500 Sample Loop: 100 cm of 0.5 mm tubing for Low Range
Microloop (16 cm) for High Range (P/N 50091)

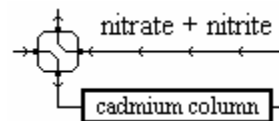
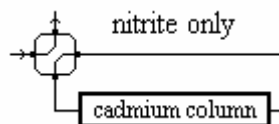
Interference Filter: 520 nm

Apparatus: An injection valve, a 10 mm path length flow cell, and a colorimetric detector module is required.

7: 135 cm of tubing on a 7 cm coil support

Note 1:

This is a two state switching valve used to place the cadmium column in-line with the manifold



Attachment 1: Calibration and QC Summary for Compliance/Enforcement Testing (DEFAULT)

QC Type	Frequency	Criteria	Corrective Actions	Comments
Method Blank	Per batch, up to 20 samples.	\leq MDL	<ul style="list-style-type: none"> Reanalyze to verify. Assess impact on samples. Re-prepare affected samples and QC. 	Prepare extra volume of blank digestate for dilutions and calibration blanks.
Lab control Sample (LCS)	Per batch, up to 20 samples.	90-110%	<ul style="list-style-type: none"> Reanalyze to verify. Re-prepare associated samples and QC. 	
MS/MSD	Per batch, up to 20 samples.	80-120% RPD \pm 25%	<ul style="list-style-type: none"> Reanalyze to verify. Compare to LCS to assess matrix effects. 	
Initial calibration	Changes in system or failure of ICV, CCV, initial calibration.	Minimum 4 points (including blank). $R^2 \geq 0.995$ Residuals \leq 10%	<ul style="list-style-type: none"> Review curve. Reprepare standards and recalibrate. 	Standards must be digested.
ICV	Immediately after multipoint calibration	90-110%	<ul style="list-style-type: none"> Reanalyze. Recalibrate. 	ICV must be digested.
ICB	Immediately after ICV.	\leq MDL	<ul style="list-style-type: none"> Reanalyze. Recalibrate if drift suspected. 	
RL check	After calibration and prior to sample analysis.	\pm 20% of expected value.	<ul style="list-style-type: none"> Reanalyze. Review curve. 	RL check must be digested.
CCV	After every 10 injections and end of run.	90-110%	<ul style="list-style-type: none"> Reanalyze samples back to the last acceptable CCV. 	CCV must be digested.
CCB	Immediately after CCV.	\leq MDL	<ul style="list-style-type: none"> Reanalyze samples back to the last acceptable CCB. 	
SRM/CRM	One per batch up to 20 samples.	80-120%	<ul style="list-style-type: none"> Reanalyze. Reprep associated samples. 	Must be digested.

Attachment 2: Calibration and QC Summary for Non-Compliance/Non-Enforcement Testing

QC Type	Frequency	Criteria	Corrective Actions	Comments
Method Blank	Per batch, up to 20 samples.	\leq Reporting Limit (RL)	<ul style="list-style-type: none"> Reanalyze to verify. Assess impact on samples. Re-prepare affected samples and QC. 	Prepare extra volume of blank digestate for dilution and calibration blanks.
Lab control Sample (LCS)	Per batch, up to 20 samples.	90-110%	<ul style="list-style-type: none"> Reanalyze to verify. Re-prepare associated samples and QC. 	
MS/MSD	Per batch, up to 20 samples.	80-120% RPD \pm 25%	<ul style="list-style-type: none"> Reanalyze to verify. Compare to LCS to assess matrix effects. 	
Initial calibration	Changes in system or failure of ICV, CCV, initial calibration.	Minimum 4 points (including blank). $R^2 \geq 0.995$ Residuals $\leq 10\%$	<ul style="list-style-type: none"> Review curve. Reprepare standards and recalibrate. 	Standards must be digested.
SRM/CRM	Prepare one per batch up to 20 samples. Analyze immediately after multipoint calibration.	80-120%	<ul style="list-style-type: none"> Reanalyze. Recalibrate. 	Must be digested.
ICB	Immediately after ICV.	\leq RL	<ul style="list-style-type: none"> Reanalyze. Recalibrate if drift suspected. 	
CCV	After every 10 injections and end of run.	80-120%	<ul style="list-style-type: none"> Reanalyze samples back to the last acceptable CCV. 	CCV must be digested.
CCB	Immediately after CCV.	\leq RL	<ul style="list-style-type: none"> Reanalyze samples back to the last acceptable CCB. 	

STANDARD OPERATING PROCEDURE
TITLE: Nitrogen as Nitrate-Nitrite in Water by Flow Injection Analysis

REVISION HISTORY		
Revision #	Summary of Changes	Date
4	Reformatted and renumbered SOP. Added criteria and corrective actions for calibration and QC samples. Added handling of brackish samples. Added QC summary attachments for enforcement testing vs. non-enforcement testing.	12/11/09
3	Unknown. Known as Method 18.1.	04/01/09
1-2	Unknown	04/06/05
0	Initial release as Method 18.	Unknown

Author:	<i>Kendell Penney</i>	Date:
Approved:	Laboratory Director <i>David B. Crane</i>	Date:
Approved:	Section Lead <i>Patricia J. Bucknell</i>	Date:
Approved:	Quality Assurance <i>Gail Cho</i>	Date:
Approved:	Health and Safety <i>Thomas Lew</i>	Date:

STANDARD OPERATING PROCEDURE

TITLE: Nitrogen, Nitrate-Nitrite in Water by Flow Injection Analysis

1.0 Scope and Application

- 1.1 This method is applicable to the determination of nitrite singly, or nitrite and nitrate combined in surface and saline waters, as well as domestic and industrial waste waters.
- 1.2 The applicable range for this method is 0.010-0.50 mg/L [NO₃ as N]. The method detection limit (MDL) is 0.005 mg/L NO₃ as N with a reporting limit (RL) of 0.010 mg/L NO₃ as N. Concentrations between 0.005-0.010 mg/L NO₃ as N are considered detectable but not quantifiable.
- 1.3 The applicable range for nitrite by this method is 0.005-0.50 mg/L NO₂ as N. The method detection limit (MDL) is 0.002 mg/L NO₂ as N with a reporting limit (RL) of 0.005 mg/L NO₂ as N. Concentrations between 0.002-0.005 mg/L NO₂ as N are considered detectable but not quantifiable.

2.0 Summary of Method.

- 2.1 Nitrate is quantitatively reduced to nitrite by passage of the sample through a copperized column of cadmium granules. The resulting nitrite (in addition to the nitrite initially present in the sample) is determined by diazotizing with sulfanilamide followed by coupling with N-(1-naphthyl)ethylenediamine dihydrochloride. The resulting water soluble dye has a magenta color which is read colorimetrically at 520 nm. Nitrite alone can be determined by removing the cadmium column. Once nitrite has been quantified, this amount can be subtracted out of the nitrate plus nitrite results to yield the nitrate value alone

3.0 Interferences and Comments.

- 3.1 Build up of suspended matter in the cadmium column or manifold tubing will restrict sample flow. Since nitrate-nitrogen is found in the soluble state, the sample may be pre-filtered through a 0.45 um glass fiber filter or a 0.45 um disposable filter apparatus.
- 3.2 Residual chlorine can interfere by oxidizing the cadmium column.
- 3.3 Low results may be obtained for samples that contain high concentrations of iron, copper or other metals. EDTA is added to the buffer in order to reduce this interference.
- 3.4 Samples that contain large amounts of oil and grease will coat the surface of the cadmium. Pre-extraction of the samples with an organic solvent will eliminate this interference.
- 3.5 This method determines both nitrate and nitrite. If only nitrate is desired, a separate determination must be made for nitrite and subsequent corrections made. The nitrite may be determined by the procedure below without the reduction step.

- 3.6 Interferences from brackish samples, high density samples, or samples with extreme pH may cause a refractive index manifested as baseline fluctuation. Brackish shutter adjustment may be necessary. Refer to section 10.4 and Figure 2 for adjustment guidelines.

4.0 Safety

- 4.1 Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. At a minimum, wear gloves, lab coats, and safety eyewear to minimize contact with chemicals.
- 4.2 The following chemicals have the potential to be highly toxic or hazardous; for detailed explanation consult the MSDS.
- 4.2.1 Phosphoric acid (H_3PO_4)
 - 4.2.2 Hydrochloric acid (HCl)
 - 4.2.3 Ammonium hydroxide (NH_4OH)
 - 4.2.4 Sodium hydroxide (NaOH)
 - 4.2.5 N-(1-naphthyl)ethylenediamine dihydrochloride (NED)
 - 4.2.6 Tetrasodium ethylenediamine tetraacetic acid (Na_4EDTA)
 - 4.2.7 Cadmium granules.
- 4.3 Dispose of wastes and reagents according to WPCL-EH-049 "Disposal of Hazardous Wastes."

5.0 Equipment and Supplies.

- 5.1 LACHAT QuikChem 8000 Series Automated Flow Injection Analyzer (FIA)
- 5.1.1 XYZ ASX-500 Autosampler
 - 5.1.2 Reagent Pump RP-100 Series
 - 5.1.3 Nitrate-Nitrite Manifold
 - 5.1.4 Colorimetric detector with 520 nm filter
 - 5.1.5 Data System – DELL Optiplex GX150 / Omnion 3.0 software
 - 5.1.6 Cadmium – Copper Reduction Column Lachat Part No. 50237
 - 5.1.7 Pump tubing of proper size for nitrate-nitrite method (Table 3).
 - 5.1.7.1 "Orange" tubing, Fisher catalog 14-190-503.
 - 5.1.7.2 "Yellow" tubing, Fisher catalog 14-190-509.
 - 5.1.7.3 "Green" tubing, Fisher catalog 14-190-512.
- 5.2 Pyrex Class A volumetric flasks and graduated cylinders for stock standard and reagent preparation.
- 5.3 Flow Injection Analysis (FIA) standard tubes (50mL): 290.D.x115Lmm disposable polypropylene skirted centrifuge tubes with screw caps (VWR cat#21008-480) for working standards preparation and storage.
- 5.4 FIA sample tubes: 13 X 100 mm disposable borosilicate sample test tubes for XYZ autosampler.

5.5 Analytical balance with the capability of weighing to the nearest 0.0001g.

6.0 Reagents and Standards.

Label all reagents and standards with identity, tracking number, concentration, preparation date, expiration date, preparers' initials.

6.1 Reagent water.

6.1.1 Use ASTM Type II water for all solutions. The Milli-Q (MQ) system produces reagent water meeting specifications. (See Standard Specification for Reagent Water D1193-77 for more information).

6.2 Reagent 1: 15N Sodium Hydroxide.

6.2.1 In a 250 mL volumetric flask slowly add 150 g NaOH to 50 mL Milli-Q (MQ) water.. CAUTION: The solution will get very hot! Swirl until dissolved. Dilute to mark. Label, cool and store in a plastic bottle.

6.3 Reagent 2: Ammonium Chloride Buffer, pH 8.5.

6.3.1 **CAUTION: FUMES!!!** In a hood, using a 4 liter beaker add 1 liter Milli-Q water, 210 mL concentrated hydrochloric acid (HCL), 190 mL ammonium hydroxide (NH₄OH), and 2.0 g disodium EDTA. Dissolve, bring up to the 2 liter mark, and cool to room temperature.. Adjust pH to 8.5 with concentrated HCL or 15 N sodium hydroxide solution. Adjust pH in the hood. Transfer to a 2 liter volumetric flask and dilute to mark. Invert to mix. This solution is good for 6 months.

6.4 Reagent 3: Sulfanilamide Color Reagent

6.4.1 To a 1 liter volumetric flask, add about 600 mL Milli-Q water. then add 100 mL of 85% (concentrated) phosphoric acid (H₃PO₄), 40.0 g sulfanilamide, and 1.0 g N-(1-naphthyl)ethylenediamine dihydrochloride (NED). Stir for 30 min. to dissolve. Dilute to the mark with MQ water, and invert to mix. Store in an amber glass bottle and refrigerate. This solution is stable for one month.

6.5 Carrier

6.5.1 Milli-Q water.

Standard Preparation.

Use MQ water for the preparation of all standards.

6.6.1 Standard 1: Stock Nitrate Standard 10.0 mg/L (NO₃ as N)

6.6.1.1 In a 50 mL FIA standard tube dilute 0.50 mL of 1000 mg/L (NO₃ as N) purchased standard with Milli-Q water. Dilute to the mark with Milli-Q water and invert to mix. This standard is stable for one month.

- 6.6.2 Standard 2: Stock Nitrite Standard 10.0 mg /L (NO₃ as N).
- 6.6.2.1 In a 50 mL FIA standard tube dilute 0.50 mL of 1000 mg N/L Nitrite as N purchased standard with MQ water. Dilute to the mark with MQ water and invert to mix. This solution is stable for 3-5 days.
- 6.6.3 Standard 3: Working Stock Nitrate Standard 1.00 mg/L NO₃ as N
- 6.6.3.1 Add 5.0 mL of Standard 1 (10.0 mg/L NO₃ as N) to a FIA standard tube. Dilute to 50 mL with MQ water and invert to mix. This solution is stable for one week.
- 6.6.4 Standard 4: Working Stock Nitrite Standard 1.00 mg/L NO₂ as N
- 6.6.4.1 Add 5.0 mL of Standard 2 (10.0 mg/L NO₂ as N) to a FIA standard tube. Dilute to 50 mL with MQ water and invert to mix. This solution is stable for 24 hours.
- 6.6.5 Working Standard 1: 0.500 – 0 mg/L NO₃ as N
In FIA standard tubes dilute the indicated amount, according to Table 1 below, of Standard 3 (1.00 mg/L NO₃ as N) to 50 mL with MQ water and invert to mix. These solutions are stable for 24 hours.

—Table 1: Preparation of Working Standards 1—

Working Standards 1 Concentration in mg/L NO ₃ as N	Volume (mL) of Standard 3 (1.00 mg/L NO ₃ as N) in 50 mL
0.500	25.0
0.250	12.5
0.100	5.00
0.050	2.50
0.020	1.00
0.010	0.50

- 6.6.6 Working Standard 2: 0.100 – 0 mg/L NO₂ as N
In FIA standard tubes dilute the indicated amount, according to Table 2 below, of Standard 4 (1.00 mg/L NO₂ as N) to 50 mL with MQ water and invert to mix. These solutions are stable for 24 hours.

—Table 2: Preparation of Working Standards 2—

Working Standards 2 Concentration in mg/L NO ₂ as N	Volume (mL) of Standard 4 (1.00 mg/L NO ₂ as N) in 50 mL
0.100	5.00
0.075	3.75
0.050	2.50
0.025	1.25
0.005	0.25

7.0 Preservation and Holding Times.

- 7.1 Analysis should be made as soon as possible. If analysis can be made within 24 hours, the sample should be preserved by refrigeration at 4°C. When samples must be stored for more than 24 hours, they should be preserved with sulfuric acid. Adjust the sample to pH < 2 with concentrated H₂SO₄, (approximately 1 drop per 30 mL of sample) and refrigerate until analysis. Preserved samples can only be analyzed for nitrate plus nitrite within a maximum of 28 days.

8.0 Calibration and Standardization.

- 8.1 Lachat QuikChem 8500/8000 Data System Parameters.
The timing values listed below are approximate and will need to be optimized using graphical events programming.

Sample throughput:	80 samples/h, 45 s/sample
Pump Speed:	35
Cycle Period:	45
Analyte Data:	
Concentration Units:	(mg as N)/L
Peak Base Width:	18 s
% Width Tolerance:	100
Threshold	17000
Inject to Peak Start	10s
Chemistry	Direct

- 8.2 Calibration curve.

Level	1	2	3	4	5	6
Concentration: mg as N/L	See Table 1 for NO ₃ -NO ₂ curve. See Table 2 for NO ₂ curve.					

Calibration Rep Handling: Average
Calibration Fit Type: 1st Order Polynomial
Weighting Method: None
Force through zero: No

- 8.2.2 $R^2 \geq 0.995$.

- 8.2.3 Residuals $\leq 10\%$.
- 8.2.4 If outside limits, review standards for preparation errors, mis-injections, instrument malfunctions. Prepare new standard or calibration curve if corrective actions are ineffective.
 - 8.2.4.1 Under extenuating situations, an analyst may omit one of the following standards: the 2nd lowest standard, the penultimate high standard or the highest standard may be omitted. Only one standard may be omitted from a curve. Be aware of the impact on the quantification range.
 - 8.2.4.2 A curve with a minimum of 4 points is required for quantification of analyte.
 - 8.2.4.3 Refer to WPCL-AA-068 "Manual Peak Integration and Calibration for FIA" for manual integration practices.
 - 8.2.4.4 Document actions on the raw data.

8.3 Sampler Timing:

Min. Probe in Wash Period:	9 s
Probe in Sample Period:	20 s

8.4 Valve Timing:

Load Time:	0 s
Load Period:	10 s
Inject Period:	35 s

8.5 Total N, NO₃-NO₂ Manifold Diagram. See Figure 1.

8.6 Continuing calibration:

8.6.1 Initial Calibration Verification (ICV) and Initial Calibration Blank (ICB): Immediately after calibration, analyze a mid-range standard followed by analysis of an ICB. Note that some monitoring programs prefer the analysis of a CRM/SRM in place of an ICB. See Attachments 2 and 3 for acceptance and corrective actions.

8.6.2 Continuing Calibration Verification (CCV) and Continuing Calibration Blank (CCB): after every 10 analyses (i.e. injections) and end of run, analyze a mid-range standard followed by a CCB. See attachments 2 and 3 for acceptance and corrective actions.

9.0 Procedure

9.1 Start-Up Procedure

9.1.1 Degassing: The solutions will degas on their own if they are allowed to sit at room temperature slightly vented overnight.

- 9.1.1.1 Alternative Degassing with Helium: To prevent air spikes use helium to degas all solutions except the standards. Use He at 140kPa (20 lb/in²) through a helium degassing tube (Lachat Part No. 50100). Bubble helium through the solution for one minute.
- 9.1.1.1.1 Thoroughly rinse the acid washed probe prior to and after degassing each reagent to avoid contamination of subsequent reagents.
- 9.1.1.1.2 Blow out excess water by turning on the helium after taking the probe out of the graduated cylinder and wipe down the probe and probe end with a Chemwipe.
- 9.1.2 Turn instrument on at the power strip located on wall behind FIA.
- 9.1.3 Prepare any expired reagents and/or standards as stated in section 6.0.
- 9.1.4 Prepare the CB, FB, QC, and LCS as stated in section 11.0. If samples are digested, prepare an MS/MSD pair at this time.
- 9.2 Set up manifold as shown in Figure 1. (There is also a copy of the manifold diagram in the cabinet above the FIA in the Inorganic Laboratory.) of the QuikChem Method 10-107-04-1-B "Determination of Nitrate/Nitrite in Surface Wastewaters by Flow Injection Analysis" written by Diane Pritzlaff of Lachat Instruments.
- 9.2.1 Turn on the computer and printer.
- 9.2.2 On the Windows desktop, click the OMNION 3.0 icon.
- 9.2.3 Once in OMNION 3.0, pull down the FILE menu to OPEN.
- 9.2.4 To start a new run, open a previous run by the date and time the run took place.
- 9.2.5 The data system parameters in 8.1 are determined and set at the time of the initial method set up. Changes should not be required except in the case of fine tuning.
- 9.2.6 Once a previous run has been opened, change the old sample schedule to reflect the current sample schedule.
- 9.2.7 Clean the outside of the tubing prior to placing the lines into MQ water. Lock down pump tubing and adjust tension by pushing latch on top of pump tube holder all the way back towards the manifold. Once latch is all the way back, bring it forward one click. Start pump by pressing NORMAL RUN.
- 9.2.8 Check for leaks and smooth flow. Watch lines for pulsating action and listen to the pump for a squeaky noise. Either of these conditions may indicate a plugged line within the system. Refer to the troubleshooting section in the QC8000 FIA Users Manual, pgs. 143-188, if either of these conditions occurs.

- 9.2.9 Replace reagent lines from MQ water and place in appropriate reagents. Place the sample probe rinse line into the carrier container. Allow system to equilibrate to achieve a steady baseline. Toggle the Preview icon to see baseline visually.
- 9.2.10 Place the standards in the auto sampler rack from high to low concentration. Double check their position compared to the cup number listed on the schedule.
- 9.2.11 Pour samples into the 13 X 100 mm test tubes and load them into sample rack. Double check the schedule sample cup numbers against the actual sample rack.
- 9.2.12 Check to see that the sample probe rinse reservoir is receiving water and is continually upwelling.
- 9.2.13 Examine waste lines to see that three lines are dripping.
 - 9.2.13.1 Three lines drip into the waste container; one line for each of the two channels and one from the valve waste.
- 9.2.14 To start the run, click START on the **Run ON** menu.
- 9.3 Shutdown Procedure.
 - 9.3.1 Remove all reagent lines and place in a beaker of MQ water, rinsing the outside of the lines first.
 - 9.3.2 Replace initial rinse water with fresh MQ water and allow this to be pumped through the manifold for approximately 10 minutes.
 - 9.3.3 Pump the lines dry by removing lines from rinse water.
 - 9.3.4 Wrap reagent lines around a beaker to avoid crimping.
 - 9.3.5 Empty manifold waste receptacle, autosampler waste receptacle, and FIA sample tubes.
 - 9.3.6 Shut down the computer through the Windows menu.
 - 9.3.7 Turn instrument off at power strip.

10.0 Analysis and Calculations.

- 10.1 Once the instrument has read the standards, a calibration curve is prepared by plotting response versus standard concentration. Sample concentration is calculated from the regression equation.
 - 10.1.1 Click on the graph icon along the left side of the channel display box. The calibration curve and the correlation coefficient are displayed

- 10.2 Report only those values that fall between the lowest and the highest calibration standards. Samples exceeding the highest standard should be diluted with MQ water and reanalyzed.
- 10.1 Once the run has ended, check the integration of the peaks on the channel display.
 - 10.1.1 It is helpful to enlarge the scale of the display in order to view the encompassment of the peaks.
 - 10.1.1.1 Use the buttons on the left side of the channel display to zoom in and out of the x and y axis.
 - 10.1.2 If necessary to adjust the window in which the software looks for the peak, right click the mouse on the channel data display.
 - 10.1.2.1 Use the peak of the highest standard when adjusting window. The area encompassing the largest peak should also take care of the smallest peaks.
 - 10.1.2.2 Select ADJUST PEAK EXPECTATION WINDOW.
 - 10.1.2.3 Drag the sides of the active box far enough out to encompass entire peak.
 - 10.1.2.4 Right click again and select RERUN PEAK DETECTION. The software will reintegrate the peaks and display the corrected values in the run report.
 - 10.1.3 To omit a calibration standard from the run, see 8.2.
 - 10.1.3.1 Click the graph icon on the left side of channel display.
 - 10.1.3.2 In the column for UNUSED calibration standards, click the box which corresponds to the standard to be omitted.
 - 10.1.3.3 Go back to channel display and right click mouse. Select RERUN PEAK DETECTION. The data has now been reintegrated, omitting the flawed standard.
 - 10.1.3.4 Document all actions on the raw data.
- 10.2 To print results, pull down the tools menu and select CUSTOM REPORT. Click on the yellow icon (cheese) along top of screen.
- 10.3 Select the CALIBRATION box and CHANNEL DATA DISPLAY box. This will print the calibration curve, the channel display and the sample results.
- 10.4 If samples are brackish, chromatograms may appear as shown in Figure 2.
 - 10.4.1 Adjust the brackish shutter window as indicated in Figure 2.
 - 10.4.1.1 If needed, perform post-spikes on samples to aid in the accurate identification of the window endpoints.
 - 10.4.1.2 Shutter-window adjustments will automatically be applied to standards and quality control samples.

10.4.2 Document actions on raw data.

10.5 Report nitrite sample values in mg/L (NO₂ as N) and nitrate sample values in mg/L (NO₃ as N),

11.0 Quality Control.

11.1 A batch is defined as up to and including 20 samples of the same matrix processed together. A method blank, laboratory blank, reference standard, laboratory control, spike, matrix spike, matrix spike duplicate, and sample duplicate should be analyzed for every 20 samples, sample batch or different sample matrix.

11.2 Method Blank(FB) – A blank is run at the beginning of the batch to demonstrate freedom from contamination during the filtering process.

11.2.1 For every filter unit lot number acquire a method blank monthly.

11.2.2 Within the run make sure the FB lot number is the same as that for the samples.

11.2.3 The FB \leq MDL.

11.2.4 FB NO₂ Preparation: Run 150 mL MQ water through the filter unit. Disassemble the unit and discard the filtrate. Rinse the filter unit with ~25-50 mL MQ water, shake excess water from all parts and allow the disassembled unit to air dry overnight, all parts upside down and vented. Filter 150 mL MQ water through a dry filter unit. Save and label filtrate with date and analysis.

11.2.5 FB NO₃ Preparation: Follow the above FB NO₂ preparation however preserve the NO₃ MB with H₂SO₄ to a pH<2.

11.3 Initial Calibration Blank (ICB) or Continuing Calibration Blank (CCB)

11.3.1 Analyze immediately after the ICV and after every 10 injections, immediately following the CCV.

11.3.2 Prepare in an acid washed FIA standard tube, Use the same MQ water source (i.e. acid washed MQ water bottle) as that used to make the working standards.

11.4 SRM/CRM or ICV: A quality control (QC) check standard is used to verify that calibration standards have been made properly. Thus, the reference standard must be made from a different source than the calibration standards. A mixed anion standard, purchased through ALLTECH Corporation, is currently used for calibration verification. See Attachments 2 and 3 for frequency, acceptance criteria, and corrective actions.

11.5 Laboratory control sample (LCS): One per batch. Control limit: 90-110%. Reanalyze LCS. Re-digest and reanalyze all associated samples and QC as necessary.

- 11.6 Matrix spike/matrix spike duplicate (MS/MSD). One pair per batch. Control limit: 80-120%, RPD (relative percent difference): $\pm 25\%$. Reanalyze and compare to LCS to assess matrix interferences.
- 11.6.1 The working standards stock solution is used as the spiking solution.
- 11.6.2 Spiking concentrations are dependent upon background levels in the original sample.
- 11.6.3 If the background concentration is greater than the mid-point of the standard curve, the concentration of the spike should be approximately one-half of the original concentration.
- 11.6.4 If the concentration in the sample is below the detection limit (MDL), the concentration of the spike should be 5 to 15 times the MDL.
- 11.6.5 If the concentration lies between these extremes, the concentration of the spike should be equal to the original sample concentration.
- 11.6.6 The volume of spike solution which is added should not change the total volume by more than 5%.

12.0 References.

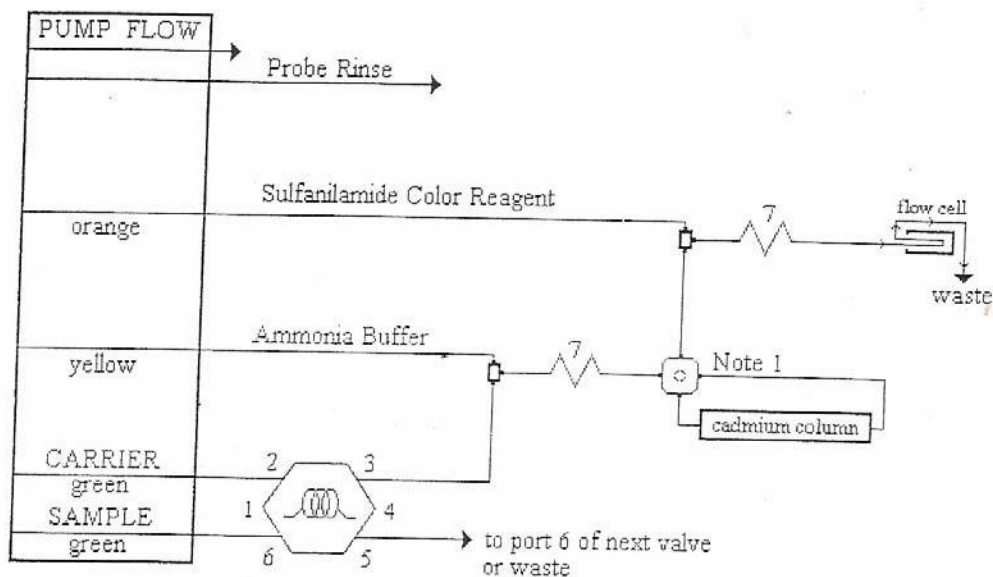
- 12.1 U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020. March 1983, Method 353.2, Nitrogen, Nitrate-Nitrite (Colorimetric, Automated, Cadmium Reduction).
- 12.2 Pritzlaff, D. QuikChem Method 10-107-04-1-B Nitrate/Nitrite in Surface and Wastewaters. Lachat Instruments, Milwaukee, WI, September 1996
- 12.3 WPCL-EH-049, "Standard Operating Procedure for the Handling, Storage, and Disposal of Hazardous and General Laboratory Waste."
- 12.4 WFCL-AA-068, "Manual Peak Integration and Calibration for FIA".

13.0 Attachments.

- 13.1 Figure 1: NO₃-NO₂ Manifold Diagram.
- 13.2 Table 3: Nitrate-Nitrite Pump Tubing Specifications
- 13.3 Attachment 1: Calibration and QC Summary for Compliance and Enforcement Testing.
- 13.4 Attachment 2: Calibration and QC Summary for Monitoring Projects.
- 13.5 Figure 2: Brackish Sample Peak Adjustment Example.

Figure 1: Total Nitrate-Nitrite Manifold Diagram

17.3. NITRATE/NITRITE MANIFOLD DIAGRAM



Carrier: Helium degassed DI water

Manifold Tubing: 0.8 mm (0.032 in) i.d. This is 5.2 $\mu\text{L}/\text{cm}$.

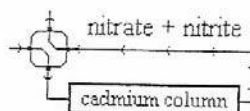
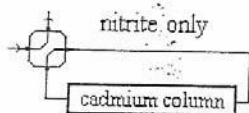
QC8000 Sample Loop: 155.5 cm

Interference Filter: 520 nm

Apparatus: An injection valve, a 1 cm path length flow cell, and a colorimetric detector module is required.

7: 135 cm of tubing on a 7 cm coil support

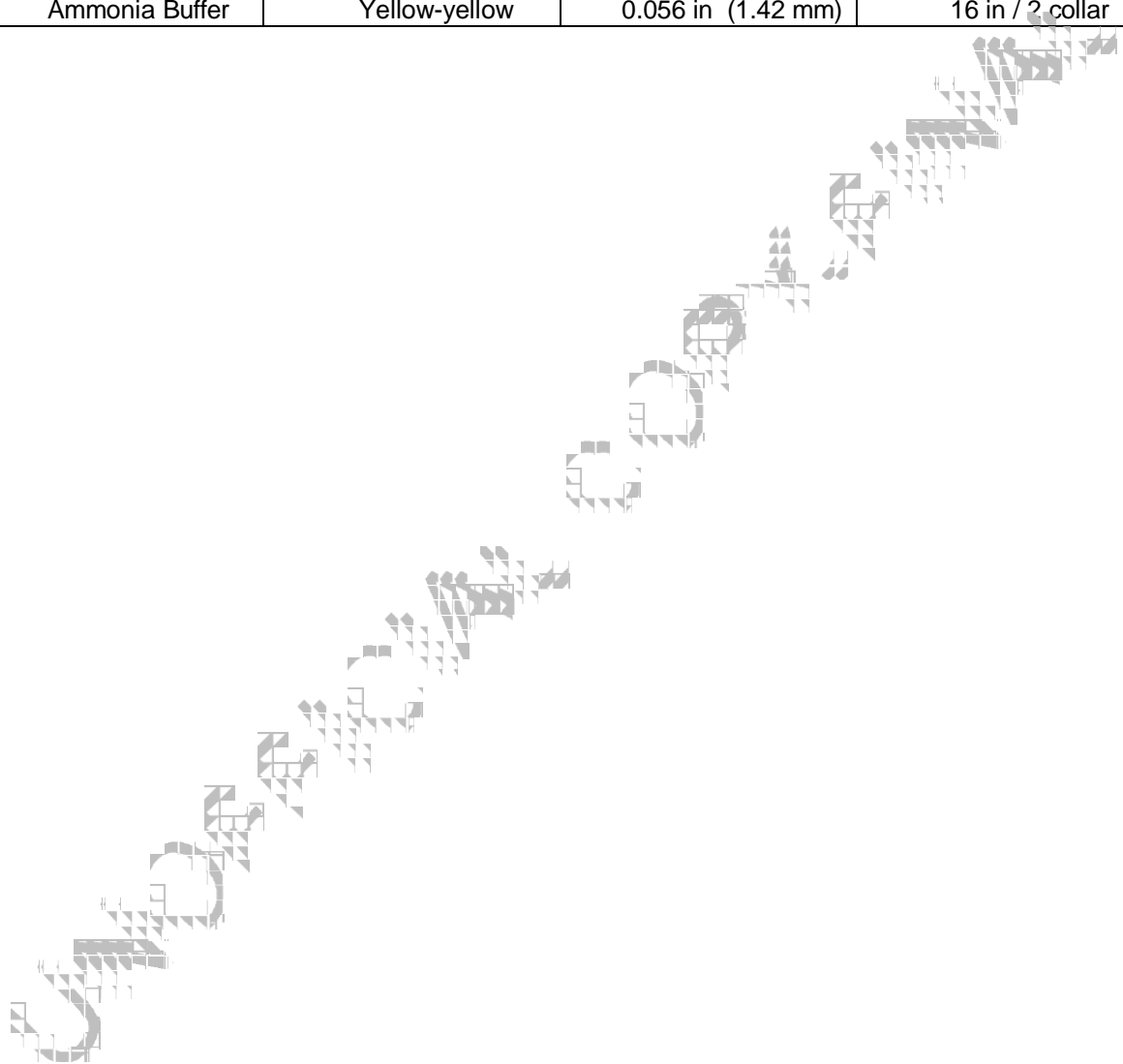
Note 1: This is a 2 state switching valve used to place the cadmium column in-line with the manifold.



Note 2: Backpressure coil, 200 cm x 0.5 mm (0.022 in) id

Table 3: Nitrate-Nitrite Pump Tubing Specifications

Reagent	Tubing color	Inner diameter	Length & collar #
Carrier	Green-green	0.073 in (1.85 mm)	16 in / 2 collar
Sulfanilamide color reagent	Orange-orange	0.025 in (0.64 mm)	16 in / 2 collar
Ammonia Buffer	Yellow-yellow	0.056 in (1.42 mm)	16 in / 2 collar



Attachment 1: Calibration and QC Summary for Compliance/Enforcement Testing

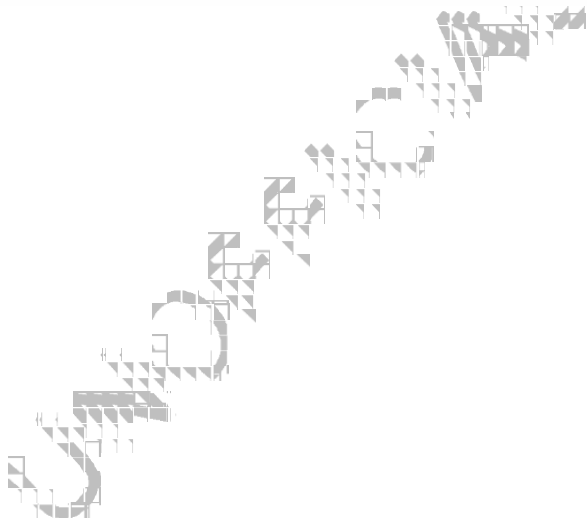
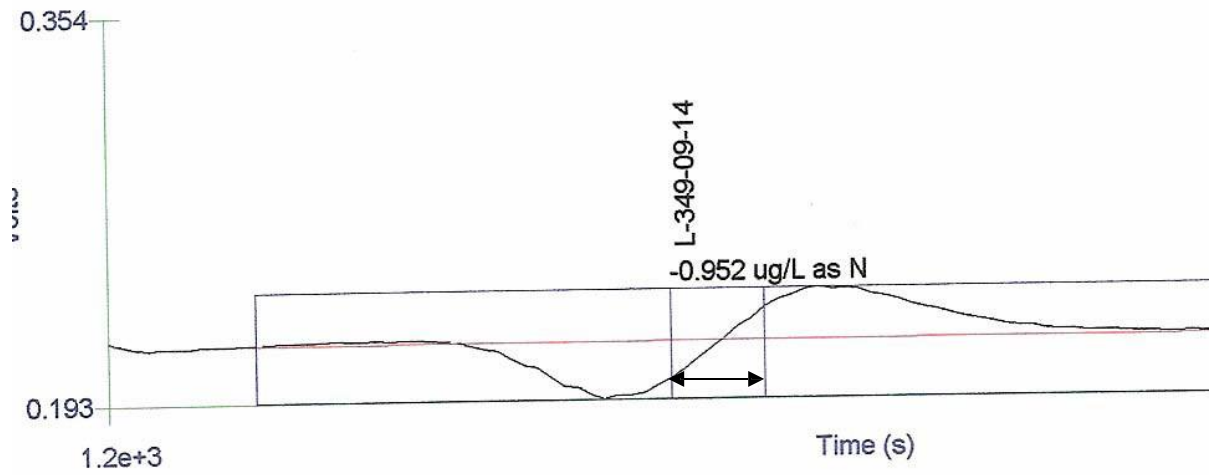
QC Type	Frequency	Criteria	Corrective Actions
Method Blank (FB)	Per batch, up to 20 samples.	\leq MDL	<ul style="list-style-type: none"> • Reanalyze to verify. • Assess impact on samples. Do not blank-subtract. • Re-prepare affected samples and QC.
Lab control Sample (LCS)	Per batch, up to 20 samples.	90-110%	<ul style="list-style-type: none"> • Reanalyze to verify. • Re-prepare associated samples and QC.
MS/MSD	Per batch, up to 20 samples.	80-120% RPD \pm 25%	<ul style="list-style-type: none"> • Reanalyze to verify. • Compare to LCS to assess matrix effects.
Initial calibration	Changes in system or failure of ICV, CCV, initial calibration.	Minimum 4 points (including blank). $R^2 \geq 0.995$ Residuals \leq 10%	<ul style="list-style-type: none"> • Review curve. • Reprepare standards and recalibrate.
ICV	Immediately after multipoint calibration	90-110%	<ul style="list-style-type: none"> • Reanalyze. • Recalibrate.
ICB	Immediately after ICV.	\leq MDL	<ul style="list-style-type: none"> • Reanalyze. • Recalibrate if drift suspected.
CCV	After every 10 injections and end of run.	90-110%	<ul style="list-style-type: none"> • Reanalyze samples back to the last acceptable CCV.
CCB	Immediately after CCV.	\leq MDL	<ul style="list-style-type: none"> • Reanalyze samples back to the last acceptable CCB.
SRM/CRM	One per batch up to 20 samples.	80-120%	<ul style="list-style-type: none"> • Reanalyze. • Reprepare associated samples.

Attachment 2: Calibration and QC Summary for Non-Compliance/Non-Enforcement Testing

QC Type	Frequency	Criteria	Corrective Actions
Method Blank	Per batch, up to 20 samples.	\leq Reporting Limit (RL)	<ul style="list-style-type: none"> • Reanalyze to verify. • Assess impact on samples. Don't blank-subtract. • Re-prepare affected samples and QC.
Lab control Sample (LCS)	Per batch, up to 20 samples.	90-110%	<ul style="list-style-type: none"> • Reanalyze to verify. • Re-prepare associated samples and QC.
MS/MSD	Per batch, up to 20 samples.	80-120% RPD $\pm 25\%$	<ul style="list-style-type: none"> • Reanalyze to verify. • Compare to LCS to assess matrix effects.
Initial calibration	Changes in system or failure of ICV, CCV, initial calibration.	Minimum 4 points (including blank). $R^2 \geq 0.995$ Residuals $\leq 10\%$	<ul style="list-style-type: none"> • Review curve. • Reprepare standards and recalibrate.
SRM/CRM	Prepare one per batch up to 20 samples. Analyze immediately after multipoint calibration	80-120%	<ul style="list-style-type: none"> • Reanalyze. • Recalibrate.
ICB	Immediately after ICV.	\leq RL	<ul style="list-style-type: none"> • Reanalyze. Recalibrate if drift suspected.
CCV	After every 10 injections and end of run.	80-120%	Reanalyze samples back to the last acceptable CCV.
CCB	Immediately after CCV.	\leq RL	<ul style="list-style-type: none"> • Reanalyze samples back to the last acceptable CCB.

Figure 2: Example Chromatogram of a Brackish Blank Sample.

annel 2: Current View



**CDFG FISH AND WILDLIFE WATER POLLUTION CONTROL
LABORATORY STANDARD OPERATING PROCEDURE FOR THE
DETERMINATION OF TOTAL PHOSPHORUS IN WATER USING
PERSULFATE DIGESTION BY FLOW INJECTION ANALYSIS COLORIMETRY
(BLOCK DIGESTOR METHOD)**

10 to 500 µg P/L

1.SCOPE AND APPLICATION

- 1.1. *This method covers the determination of total phosphorus in water using an acidic persulfate digestion.*
- 1.2. *The method is based on reactions that are specific for the orthophosphate ion.*
- 1.3. *The applicable range is 10 to 500 μ g P/L. The method detection limit (MDL) is 5.0 P/L. Approximately 60 samples per hour can be analyzed.*

2.SUMMARY OF METHOD

- 2.1. *This method utilizes an off-line digestion to convert all forms of phosphorus into orthophosphate using an acidic persulfate digestion. The orthophosphate ion (PO_4) produced reacts with ammonium molybdate and antimony potassium tartrate under acidic conditions to form a complex. This complex is reduced with ascorbic acid to form a blue complex which absorbs light at 880 nm. The absorbance is proportional to the concentration of orthophosphate in the sample.*

3.INTERFERENCES

- 3.1. *Silica forms a pale blue complex which also absorbs at 880 nm. This interference is generally insignificant as a silica concentration of approximately 30 ppm would be required to produce a 0.005 ppm positive error in orthophosphate.*
- 3.2. *Glassware contamination is a problem in low level phosphorus determinations. Soak digestion tubes in phosphate-free soap, and then scrub the inside before loading them in the dishwasher. After being washed, digestion tubes should be filled with 1:1 HCl, covered and allowed to sit overnight. When ready to be used, rinse with deionized water and then drain.*
- 3.3. *Samples containing bromide can lead to errors in the phosphate measurement. The bromine formed in the digestion may cause double peaks in low-level samples. Sparging these samples with helium for approximately 10 minutes will remove this interference.*
- 3.4. *Reagent grade chemicals may contain phosphorus. Use the best quality reagents possible to avoid large blank peaks, which can affect detection limits.*

4.SAFETY

- 4.1. *The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials.*
- 4.2. *Each laboratory is responsible for maintaining a current awareness file of the Occupational Health and Safety Act (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data sheets (MSDS) should be made available to all personnel involved in the chemical analysis. The preparation of a formal safety plan is also advisable.*

Safety (continued)

4.3. *The following chemicals have the potential to be highly toxic or hazardous; for detailed explanation consult the MSDS.*

4.3.1. *Sulfuric Acid*

4.3.2 *Molybdate Tetrahydrate*

4.3.3 *Antimony Potassium Tartrate*

4.3.4 *Dodecyl Sulfate, Sodium Salt (SDS)*

4.3.5 *Potassium Persulfate*

4.4. *The following chemicals, reagents and substances contain hazardous chemical concentrations above Title 22 acceptance limits for in-house laboratory waste disposal. For detailed explanation consult the WPCL Inorganic Laboratory SOP for the Handling, Storage, and Disposal of Hazardous Materials and Hazardous and General Laboratory Waste prior to any reagent waste disposal associated with this method.*

4.4.1. *Ammonium Molybdate Tetrahydrate*

4.4.2. *Antimony Potassium Tartrate*

4.4.3. *Stock Ammonium Molybdate Solution*

4.4.4. *Stock Antimony Potassium Tartrate*

4.4.5. *Molybdate Color Reagent*

5.EQUIPMENT AND SUPPLIES

5.1. *Balance -- analytical, capable of accurately weighing to the nearest 0.0001 g.*

5.2. *Glassware -- Class A volumetric flasks and pipettes or plastic containers as required.*

5.3 *Eppendorf pipettes and pipet tips.*

5.4. *Flow injection analysis equipment designed to deliver and react sample and reagents in the required order and ratios.*

5.4.1. *Sampler*

5.4.2. *Multichannel proportioning pump*

5.4.3. *Reaction unit or manifold*

5.4.4. *Colorimetric detector*

5.4.5. *Data system*

- 5.5. *Special Apparatus*
- 5.5.1. *Heating unit*
- 5.5.2. *Block Digestor*
- 5.5.3. *Labware for digestion. For the BD-46, 25 x 150 mm Corning®Screw-capped tubes. Caps should have rubber liners.*
- 5.5.4. *Vortex mixer*
- 5.5.5. *Glass calibration vials must be used. Lachat per no. 21304, XYZ samplers.*

6. REAGENTS AND STANDARDS

6.1. PREPARATION OF REAGENTS

Use ASTM Type II water for all solutions. (See Standard Specification for Reagent Water D1193-77 for more information).

6.1.1 Degassing with helium:

To prevent bubble formation, degas all solutions except the standards with helium. Use He at 140kPa (20 lb/in²) through a helium degassing tube (Lachat Part No. 50100.) Bubble He through the solution for one minute.

6.1.2 Alternative to Degassing:

Solutions will degas on their own if they are allowed to sit at room temperature slightly vented overnight.

Reagent 1. Stock Ammonium Molybdate Solution

In an acid-washed **500 mL** volumetric flask, dissolve **20.0 g ammonium molybdate tetrahydrate** [(NH₄)₆Mo₇O₂₄·4H₂O] in approximately **400 mL water**. Dilute to the mark with **DI water** and stir for minimum of four hours. Store for up to two months in acid-washed plastic and refrigerate.

Reagent 2. Stock Antimony Potassium Tartrate Solution

In an acid-washed 500 mL volumetric flask, dissolve 1.61 g antimony potassium tartrate (potassium antimonyl tartrate trihydrate C₈H₄O₁₂K₂Sb₂·3H₂O) in approximately 400 mL water. Dilute to the mark and mix with an acid-washed magnetic stirrer until dissolved. Store in a dark acid-washed bottle and refrigerate. Store for up to two months.

Reagent 3. Molybdate Color Reagent

To an acid-washed **250 mL** volumetric flask, add about **100 mL water**, and then **add 5.0 mL concentrated sulfuric acid**. Swirl to mix. When the solution is at room temperature, add **53.25 mL Ammonium Molybdate Solution** (Reagent 1) and **18 mL**

Reagents (continued)

Antimony Potassium Tartrate Solution (Reagent 2). Dilute to the mark and invert to mix. Prepare weekly.

Reagent 4. Ascorbic Acid Reducing Solution

In an acid-washed 500 mL volumetric flask, dissolve 30.0 g ascorbic acid in about 400 mL water. Dilute to the mark and mix with an acid-washed magnetic stirrer. Degas this solution with helium. Add 1.0 gm SDS (sodium dodecyl sulfate Aldrich catalog no. 86,201-0). Mix with a magnetic stirrer. Prepare fresh every two days.

Reagent 5. 11N Sulfuric Acid

To an acid washed 1L volumetric flask containing about 500 mL of DI water, add 500 mL of DI water and place in a tub that has cold water in it. Gradually add 305 mL of concentrated sulfuric acid. CAUTION, solution will be hot! Stir to mix, cool to room temperature and dilute to volume. Do not degas this reagent! Prepare monthly.

Reagent 6. Carrier: Sulfuric Acid, 0.231 M

In an acid-washed 500 mL volumetric flask, add 200 mL water and 21 mL Reagent 5 (11N H₂SO₄). Dilute to the mark with DI water and invert to mix. Degas daily. Prepare fresh weekly

Reagent 7. Basic Digestion Reagent (Digestion Reagent 1)

In a 500 mL acid washed volumetric flask dissolve 5.24 g sodium hydroxide (NaOH) and 21 g potassium persulfate (K₂S₂O₈), in approximately 800 mL DI water. Dilute to the mark and invert to mix. Prepare fresh monthly and store in plastic. Do not degas this reagent!

Reagent 8. Acidic Digestion Reagent (Digestion Reagent 2)

In a 250 mL volumetric flask, add 100 mL Reagent 5 (11N H₂SO₄) and 5.75 g potassium persulfate (K₂S₂O₈). Dilute to the mark with Reagent 5 (11N H₂SO₄) and invert to mix. Prepare fresh weekly. Do not degas this reagent!

Reagent 9. Acidic Blank Water

In a 500 mL HDPE bottle, add 300 mL deionized water and 4.0 mL 1:5. Dilute to the mark with DI water and invert to mix. Prepare fresh weekly. Do not degas this reagent!

Reagent 10. 1:5 Sulfuric Acid

In a 250 mL acid washed volumetric flask, add appx. 100 mL deionized water and then 50 mL 36 N sulfuric acid. Dilute to the mark with DI water and invert to mix. Prepare fresh weekly. Do not degas this reagent!

6.2. PREPARATION OF STANDARDS

Standard 1. Stock Standard 250 mg P/L

In an acid-washed 1 L volumetric flask dissolve **1.099 g primary standard grade anhydrous potassium dihydrogen phosphate** (KH₂PO₄) that has been dried for one hour at 105°C in about **800 mL water**. Dilute to the mark with **DI water** and invert to mix. This standard is good for 28 days.

Standard 2. Working Stock Standard Solution 5.00 mg P/L

In an acid-washed 100 mL volumetric flask, dilute 2.0 mL Stock Standard (Standard 1) to the mark with DI water. Invert to mix. Prepare every two weeks.

Standard 3. Working Stock Standard Solution (500 ppb P/L)

In an acid-washed **250 mL** volumetric flask dilute **25.0 mL of T-phos Stock Standard #2 (5.00 mg/L)**, and dilute to the mark with reagent 9, acidic blank water. Invert to mix. Prepare this standard every 2 weeks.

Working Standards

These standards **will be** digested

From **Standard #3 (T-PHOS = 500 ppb standard)**

Prepare standards of the following concentrations. Prepare standards every two weeks.

Note: Standards are prepared in acidified DI water (reagent 9) and carried through the digestion procedure with the samples. Using simulated standards is not recommended, due to nitrogen and phosphorus present in the digestion reagents. **For the digested standards and samples to work with this method, they must be carried through the entire digestion procedure.**

Final Concentration T-PHOS(ppb)	mL of Standard 3 diluted with Reagent #9 (Acidic Blank Water)
T-PHOS	
500	50 (no dilution)
250	25 mls of 500 into 50 mls
100	10 mls of 500 into 50 mls
50.0	5.0 mls of 500 into 50 mls
20.0	2.0 mls of 500 into 50 mls
10.0	1.0 mls of 500 into 50 mls
0.00	0.0

6.3 PREPARATION OF DIGESTION CHECK STANDARDS (CRM)

Stock Check Standard #1, 1000 mg P/L sodium tripolyphosphate (3P)

In a 500 mL acid washed volumetric flask add 2.33 g sodium tripolyphosphate 85%, ($\text{Na}_5\text{P}_3\text{O}_{10}$ FW = 367.86) in about 200 mL DI water. Dilute to the mark with DI water and invert to mix. Prepare fresh monthly.

Working Check Standard 10.0 mg P/L sodium tripolyphosphate (3P)

In an acid-washed 500 mL volumetric flask dilute 5.0 mL of Stock Check Standard #1 to the mark with DI water. Invert to mix. Prepare fresh monthly.

Working Check Standard 100 ppb sodium tripolyphosphate (3P) Note: This standard will be digested

In an acid washed 100 mL volumetric flask dilute 1.00 mL of 10.0 ppm working stock standard solution sodium tripolyphosphate to the mark with reagent 9, acidic blank water. Invert to mix. Prepare fresh monthly. . This standard is digested.

Stock Check Standard #2, Adenosine 5'-triphosphate, disodium salt hydrate (Aldrich A26209), 100 mg P/L

In a 1L volumetric flask add 0.5991g of Adenosine 5'-triphosphate, disodium salt hydrate in about 500 mL DI water. Dilute to the mark with DI water and invert to mix. Prepare fresh monthly.

Working Check Standard, Adenosine 5'-triphosphate, disodium salt hydrate, 500 ppb P/L

Dilute 0.500 mL of Stock Check Standard #2 to 100 mL in reagent #9, acidic blank water. This standard is digested.

7. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 7.1. *Samples should be collected in plastic bottles. All bottles must be thoroughly cleaned and rinsed with reagent water volume collected should be sufficient to insure a representative sample, allow for replicate analysis (if required), and minimize waste disposal.*
- 7.2. *Samples may be preserved by addition of concentrated H_2SO_4 to a pH of ≤ 2 and stored at 4°C . Acid preserved samples have a holding time of 28 days.*
- 7.3. *Sample digests should be run within 2 days of digestion.*

8. QUALITY CONTROL

- 8.1. *A method blank, laboratory blank, two reference standards, laboratory control spike, matrix spike, matrix spike duplicate and sample duplicate should be analyzed for every 20 samples, sample batch or different sample matrix.*

Quality Control (continued)

- 8.1.1. *Method Blank – A digested blank is run at the beginning of the batch to demonstrate freedom from contamination during the digestion procedure.*
- 8.1.2. *Laboratory Blank – Both an initial and final calibration blank must be analyzed during the run in order to demonstrate freedom from contamination.*
- 8.1.3. *Reference Standard – A quality control (QC) check standard is required to verify the calibration standards have been made properly. Thus, the reference standard must be made from a different source than the calibration standards. Both initial and final calibration verification must be run on the sample set. A percent recovery between 80 – 120 % is acceptable.*
- 8.1.4 *Laboratory Control Spike – A laboratory control spike is required as verification of the MS/MSD preparation method. A percent recovery between 80-120% is acceptable.*
- 8.1.5 *Sample Duplicate – Analysis of a sample duplicate indicates precision associated with laboratory procedures but not with sample collection, preservation, or storage procedures. A relative percent difference of less than 20% is acceptable.*
- 8.1.6 *Matrix Spike and Spike Duplicate – A matrix spike and spike duplicate are required to demonstrate method accuracy and precision and to monitor matrix interferences. A percent recovery between 80-120% of the spike value is acceptable.*

9.PROCEDURE

9.1. DIGESTION PROCEDURE

- 9.1.1 *Both standards, CRMs and samples are carried through this procedure.*
- 9.1.2 **To a 20.0 mL sample add 5 mL digestion solution (Reagent 1), cap, and mix. Place tubes in the preheated block digester for 20 minutes at 160°C.**
- 9.1.3 *Remove the samples from the block and allow 15 minutes to cool.*
- 9.1.4 *Add 0.50 mL digestion solution (Reagent 2) to each tube, cap and vortex to mix. Place tubes in the preheated block digester for 30 minutes at 160°C.*
- 9.1.5 *Remove the tubes from the digester and vortex each tube. Let the digestate settle for several hours or overnight. Digests can be analyzed up to two days later.*

9.2. SYSTEM START-UP PROCEDURE

- 9.2.1 *Prepare reagents and standards as described in Section 6.*
- 9.2.2 *Set up manifold as shown in Section 13.3.*
- 9.2.3 *Check all pump tubing for flat lines and replace bad tubing.*
- 9.2.4 *Check for broke O-rings in the flow cell and valves and replace them*
- 9.2.5 *Disconnect the flow cells from tubing and clean the outside and inside the connector holes with DI H₂O and allow to air dry.*
- 9.2.6 *Recommended procedure: At the start of the run, place all of the reagent lines in DI water. Pump DI water through all reagent lines and check for leaks and smooth flow. Then place the color reagent and ascorbic acid transmission lines into the NaOH - EDTA solution. Pump this solution for approximately five minutes to remove any precipitated reaction products. Then place these lines in water and pump for an additional five minutes.*
- 9.2.7 *Switch to reagents and allow the system to equilibrate until a stable baseline is achieved.*
- 9.2.8 *Place standards in the sampler, and fill the sample tray. Input the information required by data system, such as concentration, replicates and QC scheme.*
- 9.2.9 *Open last schedule to ensure most recent conditions.*
- 9.2.11 *Calibrate the instrument by injecting the standards. The data system will then associate the concentrations with responses for each standard.*
- 9.2.12 *Calibrate the instrument by injecting the standards. The data system will then associate the concentrations with responses for each standard.*

9.3. SYSTEM NOTES

- 9.3.1 *Glassware contamination is a problem in low level phosphorus determinations. Glassware should be washed with 1:1 HCl and rinsed with deionized water. Commercial detergents should rarely be needed but, if they are used, use special phosphate-free preparations for lab glassware.*
- 9.3.2. *Allow 15 min for heating unit to warm up to 37°C.*
- 9.2.10 *If sample concentrations are greater than the high standard, the digested sample should be diluted with **Diluent** (Reagent 9). Do not dilute digested samples or standards with DI water.*
- 9.2.10 *To analyze the digestion sample, prepare a standard curve by plotting peak responses of digested standards against concentration values. Compute the concentrations by comparing the sample peak responses with the standard curve.*

10. DATA ANALYSIS AND CALCULATIONS

- 10.1 Once the instrument has read the standards, a calibration curve is prepared by plotting response versus standard concentration. Sample concentration is calculated from the regression equation.*
- 10.1.1 Click on the graph icon along the left side of the channel display box. The calibration curve and the correlation coefficient are displayed.*
- 10.1.2 An acceptable curve has a correlation coefficient of 0.995 or better. The % residual of a standard in relation to the curve should be 10% or less.*
- 10.2 Samples which exceed the highest calibration standard must be diluted to within the calibration range and reanalyzed.*
- 10.2.1 Diluent solution (reagent 9) must be used to make dilutions of samples.*
- 10.3 Once the run has ended, check the integration of the peaks on the channel display.*
- 10.3.1 It is helpful to enlarge the scale of the display in order to view the encompassment of the peaks.*
- 10.3.1.1 Use the buttons on the left side of the channel display to zoom in and out of the x and y axes.*
- 10.3.2 To adjust the window in which the software looks for the peak, right click the mouse on the channel data display.*
- 10.3.2.1 Use the peak of the highest standard when adjusting window. The area encompassing the largest peak should also take care of the smallest peaks.*
- 10.3.2.2 Select ADJUST PEAK EXPECTATION WINDOW.*
- 10.3.2.3 Drag the sides of the active box far enough out to encompass entire peak.*
- 10.3.2.4 Right click again and select RERUN PEAK DETECTION. The software will reintegrate the peaks and display the corrected values in the run report.*
- 10.3.3 It is possible to omit a calibration standard from the run by reintegrating the peaks ignoring calibration standards.*
- 10.3.3.1 Click the graph icon on the left side of channel display.*

Data Analysis and Calculations (continued)

10.3.3.2 In the column for UNUSED calibration standards, click the box which corresponds to the standard to be ignored.

10.3.3.3 Go back to channel display and right click mouse. Select RERUN PEAK DETECTION IGNORING CALIBRATION STANDARDS. The data has now been reintegrated, omitting the flawed standard.

10.4 To print results, pull down the tools menu and select CUSTOM REPORT. Click on the yellow icon (cheese) along top of screen.

10.4.1 Select the CALIBRATION box and CHANNEL DATA DISPLAY box. This will print the calibration curve, the channel display and the sample results.

10.5 Report sample values in mg P/L.

10.6.1 The relationship between the method detection limit (MDL), the reporting limit (RL) and the certainty of the measurement may create results for the total phosphorus that are less than the orthophosphate result. Results near the MDL are less certain ($\pm 100\%$ uncertainty due to instrument background noise or signal-to-noise ratios) than results at the RL ($+30\%$ uncertainty) or results in the range of 10 times the MDL (100% certainty in the area of known quantitation). The MDL is only a statistically determined value and the final sample results may vary due to calibration type (1st or second order, non-linear), matrix effects, dilution errors, sample handling and preparation, sample degradation, holding times and digestion variability.

10.6.2 Confidence limits can be set by statistical determination. For total phosphorus the uncertainty of measurement levels are greater than orthophosphate due to the differences in the two methods for the MDL and RL (5.0/10.0 ug/L vs. 2.0/5.0 ug/L, respectively), calibration curve (1/x weighting, 2nd order calibration curve and no weighting, linear curve, respectively) as well as the differences in the sample preparation (acidic, persulfate digestion vs. lab filtration, respectively).

10.6.3 Due to method limitations as stated above in 10.6.1, the total phosphorus results may be less than the orthophosphate results for certain samples. The results are acceptable within the following guidelines:

Results 0-50 ug/L: Acceptable if the TP result and the OP result are within 2 times \pm reporting limit of 10 ug/L (0-20 ug/L).

Results 50-100 ug/L: Acceptable if the TP result and the OP result are within \pm reporting limit of 10 ug/L (0-10 ug/L).

Results >100 ug/L: Results that are greater than 100 ug/L are within the region of known quantitation of the calibration curve since they are 10 times the method detection limit. TP results should be greater than the orthophosphate result. Rerun the

10.6.3 (continued)

sample to confirm results that are less than the OP and flag the results in the final report if the TP is still less than the OP.

11. SHUTDOWN PROCEDURE

- 11.1 *Remove all reagent lines and place in a beaker of MQ water, rinsing the outside of the lines first.*
- 11.2 *Replace initial rinse water with fresh MQ water and allow this to be pumped through the manifold for approximately 10 minutes.*
- 11.3 *Pump the lines dry by removing lines from rinse water.*
- 11.4 *Empty auto sampler waste receptacle and replace the hazardous waste receptacle with the regular waste receptacle.*
- 11.5 *Shut down the computer through the Windows menu.*
- 11.6 *Turn instrument off at power strip.*

13. TABLE, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

13.1. DATA SYSTEM PARAMETERS FOR QUIKCHEM 8000

The timing values listed below are approximate and will need to be optimized using graphical events programming.

Low Range

Sample throughput: 38 samples/h, 95 s/sample
Pump Speed: 35
Cycle Period: 95

Analyte Data:

Concentration Units: mg P/L
Peak Base Width: 77 s
Inject to Peak Start: 14 s
Chemistry: Direct/Bipolar

Calibration Data:

Level	1	2	3	4	5	6	7
Concentration mg P/L	0.500	0.250	0.100	0.050	0.020	0.001	0.00

Calibration Rep Handling: Average
Calibration Fit Type: 1st Order Polynomial
Weighting Method: 1/x
Force through zero: No

Sampler Timing:

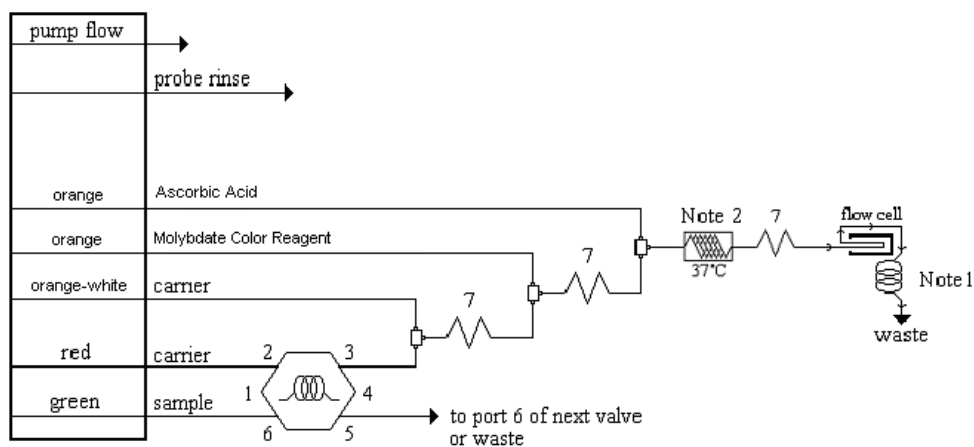
Min. Probe in Wash Period: 10 s
Probe in Sample Period: 30 s

Valve Timing:

Time to Valve^{}:* 23s
Load Period: 24 s
Inject Period: 71 s

** must be determined*

13.3. TOTAL PHOSPHORUS MANIFOLD DIAGRAM




Carrier: 0.213 M sulfuric acid (Reagent 6)

Manifold Tubing: 0.5 mm (0.022 in) i.d.

QC8500 Sample Loop: 150 cm of 0.8 mm (0.032 in) i.d. tubing for Low Range
20 cm of 0.5 mm tubing for High Range

Interference Filter: 880 nm

Apparatus: An injection valve, a 10 mm path length flow cell, and a colorimetric detector module is required.

The  shows 175 cm of tubing wrapped around the heater block at the specified temperature.

7: 135 cm of tubing on a 7 cm coil support

Note 1: 200 cm back pressure loop, 0.5 mm (0.022 in.) i.d.

Note 2: 175 cm of 0.8 mm i.d. tubing on the heater at the specified temperature.

12. REFERENCES

- 12.1. U.S. Environmental Protection Agency, *Methods for Chemical Analysis of Water and Wastes*, EPA-600/R-93/100, Rev. 2.0, Revised August 1993, Method 365.4

- 12.2. *Methods for Determination of Inorganic Substances in Water and Fluvial Sediments. Book 5. Chapter A1. U.S. Department of the Interior, U.S. Geological Survey, Method I-2601-78.*
- 12.3. *Lachat Instruments, QuikChem Method 10-115-01-1-D revised by Ann Zuehlke and Kevin Switala on 01 August 2003.*
- 12.4. *Guideline and Format for EMSL-Cincinnati Methods. EPA-600/8-83-020, August 1983.*

Section Approval: _____

Quality Control Officer: _____

Final Approval: _____

MAMMOTH COMMUNITY WATER DISTRICT

MAMMOTH LAKES, CA

Laboratory Quality Assurance Document

2011

This document has been reviewed and updated with current methods and policy procedures utilized by the Mammoth Community Water District Laboratory staff.

**Mammoth Community Water
District Mammoth Lakes, California
Laboratory Quality Assurance Document
2011**

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ORGANIZATION AND RESPONSIBILITY

Laboratory Director/Principal Analyst

In charge of all analytical and laboratory processes and is responsible for the quality of the data. Responsible for performing all laboratory analysis required for the operation of the wastewater and water treatment facilities and the water distribution system. Prepares all chemical reagents, performs record keeping, performs quality control procedures, oversees laboratory analyst, and instructs treatment plant operators in basic routine laboratory analysis procedures. Principle analyst is in charge of preparing monthly data information and billing for customers, the county health department and water district department heads. Responsible for keeping the laboratory adequately supplied with proper chemicals and equipment. Also acts as laboratory director.

Laboratory Analyst

Assists principal analyst in performing all laboratory analysis required for the operation of the wastewater and water treatment facilities and the water distribution system. Prepares all chemical reagents, performs record keeping, performs quality control procedures.

Treatment Plant Operators

Treatment plant operators perform basic routine laboratory analysis primarily on weekend duty for the purpose of monitoring and controlling the operation of the wastewater treatment facility, water treatment facility, and water distribution system.

QUALITY ASSURANCE OBJECTIVE

The objective of this laboratory is to support the Mammoth Community Water District in its commitment to carefully and effectively manage and maintain our local water resources. The goal of this laboratory is to complete the analyses they are certified to perform with care and accuracy and to assure our customers that the analyses completed are accurate and in accordance with California DPH and Federal EPA standards. We are dedicated to our customers and the environment.

SAMPLING PROCEDURES

Sample Containers

Required sample containers are provided by this laboratory. All samples for biological analysis must be taken in sterile containers. Some samples may be collected in reusable clean liter plastic jars that have been washed, rinsed and dried in the dishwasher or by hand. A final rinsed cycle should be with distilled or deionized water. Any reusable containers must be periodically rinsed with acid, HCl or HNO₃ and then washed by the previously mentioned method.

Composite Sampler

Some wastewater samples are required to be composite samples. We have Teledyne ISCO 6712FR refrigerated samplers permanently installed for collecting these samples. These automatic samplers take samples based upon the flow pacing and then self-purge after taking each sample. The samples are collected in a large plastic container which is refrigerated until it is brought to the lab after the sampling period is completed. The container is washed by hand, rinsed with distilled or deionized water, dried and returned to the sampler.

Sample Delivery to Laboratory

Samples shall be delivered to laboratory as soon as practical. Deliver sample to laboratory technician or other designated personnel with a completed chain of custody.

Sample Labels

Labels are utilized to prevent sample misidentification. The following information is included on each label: initials of collector, date, time and location of collection. Labels are affixed to containers prior to the time of sampling. Time and date of collection and initials of collector are written on the label at the time when sampling occurs. Samples collected at the MCWD wastewater treatment facility are only labeled as to location as samples are analyzed immediately by the sampler. A chain of custody must accompany every sample.

Sample Receiving

Each sample that is being submitted to the laboratory for analysis shall be received by District personnel at the laboratory office. Each sample shall be accompanied by a chain-of-custody record. Required information to be included on the chain of custody is date, time and location of sample; name, billing address and phone number of client; type of sample; and analysis requested. The District personnel, receiving the sample, completes the chain of custody by adding their initials, the date and time of sample receipt and a unique laboratory number. The sample is then stored in the refrigerator at 4 C until analysis is performed. The chain of custody is later filed for future reference.

Assignment of Sample for Analyst

All samples are to be analyzed in a timely manner. Certain analyses have short holding times and require immediate attention. These samples will be expedited by the available laboratory personnel. In the case of a plant operator being assigned to assist in the laboratory, the laboratory director or laboratory technician will assign a sample for analysis as necessary.

Sample Disposal

Any remaining sample after analysis is disposed of by pouring it down the laboratory sink. If holding times are long enough the samples are held for 7 days. We do not knowingly accept hazardous samples; hence hazardous disposal of the samples is not a problem. Positive coliform samples are sterilized by autoclave before disposal.

EQUIPMENT CALIBRATION: PROCEDURES AND FREQUENCY

Turbidimeter

The turbidimeter is calibrated on a daily basis utilizing a set of secondary standards with known turbidity units. Yearly the turbidimeter is calibrated with a formazin turbidity standard. Every three years the turbidimeter is returned to the manufacturer for a check-up and certification.

Spectrophotometer

The spectrophotometer is calibrated before each set of analytes with a range of standards above and below the expected results and a blank. Every three years the spectrophotometer is returned to the manufacturer for a check-up and certification.

pH Meter

The pH meter is checked for damage and calibrated prior to the first use of the day, utilizing a pH 4.0 buffer solution and pH 7.0 buffer solution. The pH probe is cleaned with distilled water and the filling solution is replaced monthly. The electrode is stored in pH electrode storage solution.

Analytical Balance

The Sartorius CPA124S balance is calibrated before the first use of the day, zeroed prior to each use and serviced on an annual basis by a private company.

Triple Beam Balance

The triple beam balance is zeroed and checked with our certified precision weights prior to each use. It is wiped clean and stored after each use.

Thermometers

Each incubator has a thermometer for the temperature range required. Each thermometer is calibrated through the use of a certified thermometer on a biannual basis and any correction factor is posted on the incubator thermometer. Thermometers are checked each day and the temperature of the incubators are recorded in a logbook. Any adjustment necessary is noted to maintain the proper temperature range for each incubator that is in use. Calibration of sterilizing units occurs through the use of sterilization indicators.

Incubators

Temperatures are checked and recorded daily for all incubators in use and the Coliform Incubator temperature is checked and recorded twice daily except for weekends and holidays.

Dissolved Oxygen Probe

The Dissolved Oxygen probe is calibrated prior to each use utilizing the air calibration method described in the probe's instruction manual. The batteries are changed when indicated by the probe.

PROCEDURES FOR OUTSIDE ANALYSIS

The Mammoth Community Water District Laboratory is not certified to perform the following required analyses.

	EPA	Container		Holding
Analysis	Method	Required Volume	Preservative	Time
Ammonia Nitrogen	SM4500NH3H	Plastic, 100	≤6C, H2SO4	28 days
Chloride	EPA 300.0	Plastic, 100	None	28 days
Dioxin	1613	Amber Glass, 1000	≤6C	30 days
MBAS	SM5540C	Plastic, 500	≤6C	48 hours
Metals	6010/6020	Plastic, 500	HNO3	6 months
Nitrate Nitrogen	EPA 300.0	Plastic, 100	≤6C	48 hours
Nitrite Nitrogen	SM4500NO2B	Plastic, 100	≤6C	48 hours
n-nitrosodimethylamine (NDMA)	8270	Amber Glass, 1000	≤6C	40 days
Oil And Grease	1664 A	Amber Glass, 500	≤6C, H2SO4	28 days
Sulfate	EPA 300.0	Plastic, 100	≤6C	28 days
Total Cyanide	SM4500CN	Plastic, 250	≤6C, NaOH	14 days
Total Kjeldahl Nitrogen	351.2	Plastic, 500	≤6C, H2SO4	28 days
Total Phosphorus	SM4500P E	Plastic, 100	≤6C, H2SO4	28 days
Total Trihalomethanes	551.1	Amber Glass Vial 4X40 mls	≤6C, NH4Cl	14 days
Pesticides and PCB's	608	Amber Glass 1000	≤6C	40 days
Volatiles	624	Amber Glass Vial 4X40 mls	≤6C, HCl	14 days
Base, Neutral & Acid Extractables	625	Amber Glass 1000	≤6C	40 days

SM = Standard Methods

Priority Pollutants (EPA Methods 608, 624, 625 and Dioxin)

For the above analyses, we utilize Edward S Babcock & Sons Inc. Environmental Laboratory located at 6100 Quail Valley Court, Riverside, CA, 92507. Our project manager with this laboratory is Hsin-yi Lee; her phone number is (951)653-3351, ext 251. Samples are collected, preserved and shipped via UPS in compliance with this laboratory's requirements. Samples on average arrive within three days of sample collection.

	EPA	Container		Holding
Analysis	Method	Required Volume	Preservative	Time
Gross Alpha	900	Plastic, 1000	HNO3	6 months
Radium 228	904	Plastic, 1000 x 2	HNO3	6 months
Total Radium 226	903	Plastic, 1000	HNO3	6 months
Uranium	908	Plastic, 1000	HNO3	6 months

For radiochemical analyses, we utilize Fruit Growers Laboratory Environmental located at 853 Corporation Street, Santa Paula, CA, 93060. Our contact with the laboratory is Vickie Taylor; her phone number is (805)392-2010. Samples are collected, preserved and shipped via UPS in

compliance with this laboratory's requirements. Samples on average arrive within three days of sample collection.

HOW TO SHIP A SAMPLE FOR ANALYSIS

Step 1:

Collect sample and complete label information using the labels provided by laboratory for the required analysis found in the shipping drawer in the Lab. Include sample site, date, time, sampler's initials, preservative (if any), grab or composite, client (MCWD) and analysis desired.

Step 2:

Bring sample back to MCWD laboratory, add the appropriate preservative as needed, and fill out the required laboratory's chain of custody found in the shipping drawer.

Step 3:

If laboratory personal are not available to assist with shipping sample to contract laboratory, copy completed chain of custody form, attach the copy to the REQ and place on Blair's desk. Place original 3-page chain of custody form in provided plastic bag and package sample for shipping. Containers and packing material are located in the laboratory or under the stairs, please see laboratory personal for location of packing material before you need them.

Step 4:

Take sample to warehouse for UPS shipping (before 11:30). If warehouse personal are not available or if it is after 11:30 and sample must be shipped that same day then take sample to **Access Art & Business Center** located at 437 Old Mammoth Rd in the Vons shopping center, phone number is (760)934-4667. The vendor has the shipping addresses for our contract laboratories and shipping costs are to be charged to account No. 140. Bring receipt back to the front desk personnel at the District office.

PROCEDURES FOR IN-HOUSE ANALYSIS

Alkalinity

Alkalinity of water is its acid neutralizing capacity. Our surface water, Lake Mary, has a very low alkalinity and our ground waters have average alkalinity. Alkalinity is important in the pH control of the wastewater and the corrosivity of the drinking water. We use the method of titration with a standardized sulfuric acid solution to an endpoint pH of 4.6.

Sampling and Storage

Samples are collected in one liter polyethylene bottles. Fill bottles completely and cap tightly avoiding agitation and prolonged exposure to air. Sample should be analyzed within 24 hours or 6 hours if biological activity is present.

Laboratory Equipment and Instruments pH meter

Standardized 0.02N Sulfuric acid solution 400 ml

beakers

50 ml burette

Magnetic Stirrer

Analysis Preparation

Sulfuric acid solution is prepared by adding 0.56ml of concentrated sulfuric acid to 1 liter of distilled water. This solution after thorough mixing is then standardized by titrating 1ml of 0.25N Sodium Hydroxide added to 100ml of distilled water and two drops of phenolphthaleine indicator. The normality of the sulfuric acid titer is then determined by the following formula.

$$0.25 / \text{ml of titer used} = \text{Normality of sulfuric acid titer}$$

Procedure

Place a 250ml sample with a magnetic stirring bar in a 400ml beaker, set on a magnetic stirrer with a pH electrode submerged in the sample as required and using the 50ml burette, filled and zeroed with the sulfuric acid titer, titrate the sample to a pH of 4.6. For quality control performance evaluation tests are performed annually and duplicate samples are run with each analysis. Alkalinity is calculated as follows:

$$\text{Alkalinity, mg CaCO}_3/\text{L} = (\text{ml titer} \times \text{N H}_2\text{SO}_4 \text{ titer} \times 50,000) / \text{ml sample}$$

Chlorine Residual

Select a sample volume that will require no more than 20 mL 0.01N Sodium Thiosulfate and no less than 0.2 mL for the starch-iodide end point. For a chlorine range of 1 to 10 mg/L, use a 500 mL sample; above 10 mg/L, use proportionately less sample. Place 5 mL acetic acid, or enough to reduce the pH to between 3.0 and 4.0, in a flask. Add about 1 g potassium iodide estimated on a spatula. Pour sample in and mix with a stirring rod. Titrate away from direct sunlight. Add sodium thiosulfate from a burette until the yellow color of the liberated iodine almost is discharged. Add 1 mL starch solution and titrate until blue color is discharged.

Calculation:

$$\text{mg CI as CI}_2/\text{L} = (\text{A} \times \text{N} \times 35,450) / \text{ml sample used}$$

A = mL titration for sample

N = normality of sodium thiosulfate

For more detailed information, refer to procedure described on pages 4-38 to 4-39 of Standard Methods, 18th Edition.

Turbidity

Calibrate HACH turbidimeter utilizing standards provided. Run at least one standard in each instrument range to be used. Thoroughly shake sample. Wait until air bubbles disappear and pour sample into turbidimeter tube. Read turbidity directly from instrument scale.

For more detailed information, refer to procedure described on pages 2-9 to 2-10 of Standard Methods, 18th Edition.

pH

Before use, remove electrodes from storage solution, rinse, and blot dry with a soft tissue, place in fresh pH 7.0 buffer solution, and press the 'cal' button when meter reads ready press the yes button. Remove electrodes from first buffer, rinse thoroughly with distilled water, blot dry, and immerse in fresh pH 4.0 buffer. Blot dry, immerse in a fresh portion of the sample, and read pH. The meter self adjusts for temperature hence this measurement is also available.

For more detailed information refer to procedure described on pages 4-68 to 4-69 of Standard Methods, 18th Edition and the manuals for the pH probe and meter.

Dissolved Oxygen

Dissolved Oxygen analysis is a measure of the oxygen present in a water or wastewater sample and is useful in treatment process control. We use the Iodometric or Winkler method which is a titration with standardized sodium thiosulfate using a starch solution as an indicator of the endpoint. We also have a dissolved oxygen probe for solutions where titration is not possible.

Sampling and Storage

A sample is collected carefully avoiding contact with air (fill completely and seal the container immediately), unnecessary agitation (do not shake the sample) and exposure to sunlight. If the sample collected has a high oxygen use rate such as activated sludge, a copper sulfate – sulfamic acid inhibitor solution must be added to stop oxygen use by the biological flock present. These samples must be gently inverted to cease biological activity; the clear supernatant is then placed in a 300ml BOD jar. Dissolved Oxygen analysis must be completed as soon as possible.

Laboratory Equipment and Instruments

Standardized Sodium Thiosulfate solution (0.025N) Starch Indicator solution

MnSO₄ solution

Alkali-iodide-azide reagent

Concentrated Sulfuric acid 1000

ml beakers

25 ml burette 300 ml

BOD jar

Analysis Preparation

The sodium thiosulfate titer is made by mixing 250mls of an approximately 0.2N Na₂S₂O₃ solution with 1750mls of distilled water. The 0.2N Na₂S₂O₃ solution is made by adding 64 grams of sodium thiosulfate anhydrous to 2 liters of distilled water. The Na₂S₂O₃ titer is standardized prior to use against 0.025 N potassium dichromate. 10 mls of .025N potassium dichromate is added to a 250ml flask of 90mls of distilled water. One milliliter of sulfuric acid, one gram of potassium iodide and one milliliter of starch indicator solution are added and the sample is titrated with the sodium thiosulfate titer.

Normality of Na₂S₂O₃ titer = 0.25 / mls of titer used

Procedure

To the sample collected in a 300 mL BOD jar, add 1 mL MnSO₄ solution, followed by 1 mL alkali-iodide-azide reagent, stopper carefully to exclude air bubbles and mix by inverting bottle a few times. When precipitate has settled sufficiently (to approximately half the bottle volume) to leave clear supernatant above the manganese hydroxide flock, add 1.0 mL concentrated sulfuric acid. Restopper and mix by inverting several times until dissolution is complete. Titrate a 100 mL volume of sample with standardized 0.025N sodium thiosulfate solution to a pale straw color. Add a few drops of starch solution and continue titration to first disappearance of blue color. For quality control duplicate samples are run and great care is taken in collecting and expediting the analysis of these samples. The Dissolved Oxygen is calculated by the following equation:

$$\text{mg/L DO} = (A \times N \times 8,000) / \text{ml sample used}$$

A = mL titration for sample
N = normality of sodium thiosulfate

For more detailed information, refer to procedure described on page 4-100 of Standard Methods, 18th Edition.

Chemical Oxygen Demand

CODs are performed on wastewater as needed and monitoring wells quarterly. We use the HACH Reactor Digestion Method both low (0 to 150 mg/L) and high (0 to 1,500 mg/L) ranges. HACH COD Digestion Reagent Vials for the appropriate range are used. The HACH COD Reactor is preheated to 150* C. The reactor is preset to this temperature so it only needs to be turned on to preheat to the correct temperature.

A well-mixed 2 ml sample is carefully placed into the vial of the appropriate range. Duplicate samples are analyzed with each run.

- 1) Raw and primary effluents require high range.
- 2) Final effluent and monitor wells require low range. A blank

and a standard are also analyzed with each run.

- 1) The blank consists of 2 mls of distilled water added to a vial of which ever range is being used.
- 2) The high range standard is 2 mls of a 300 mg/L COD Std solution of Potassium Acid Phthalate placed into a high range vial.
- 3) The low range standard is 2 mls of a 30 mg/L COD Std solution of Potassium Acid Phthalate added to a low range vial.

After the samples are added to the vials carefully check to be sure the caps are firmly on and then wipe the outside of the vial. Cautiously mix the contents of the vials, as they will become hot with mixing due to the presence of sulfuric acid in the digestion vials. Place the vials into the preheated (to 150*C) COD Reactor press start to begin the 2 hour digestion. After the 2 hour digestion the Reactor will shut off and let the vials cool to 120*C or less – approximately 30 minutes – then remove them from the reactor to a rack and let them cool to room temperature – about 1 hour.

To determine the COD use the HACH DR2800 Spectrophotometer set to the appropriate method. The COD in mg/l is displayed on the front of the spectrophotometer after a short wait.

- 1) *Low Range enter method number 430 and press Read/Enter. Wipe each vial with a clean cloth before placing it in the spectrophotometer.*
 - a. *Place the blank in the adapter to zero the machine.*
 - b. *Next read the standard*
 - c. *Then read all the samples*
 - d. *Finally read the standard again to see if there is any drift.*
- 2) *High Range enter method number 435 and press Read/Enter. Wipe each vial with a clean cloth before placing it in the spectrophotometer.*
 - a. *Place the blank in the adapter to zero the machine.*
 - b. *Next read the standard*
 - c. *Then read all the samples*
 - d. *Finally read the standard again to see if there is any drift.*

Biochemical Oxygen Demand and Carbonaceous Biochemical Oxygen Analysis

The Biochemical Oxygen Demand (BOD) test is used to determine the oxygen requirements of wastewaters, effluents and polluted waters. We use this test mainly to measure the waste loading to the treatment plant and evaluate the BOD-removal efficiency of our treatment. The 5-day BOD test measures the oxygen used during this time period for the biochemical degradation of organic material (carbonaceous demand), the oxidation of inorganic materials and the oxidation of reduced forms of nitrogen (nitrogenous demand). The latter can be prevented with the use of nitrification inhibitor.

Sampling and Storage

Samples for BOD analysis must be chlorine free and still may degrade significantly during storage between collection and analysis, resulting in low BOD values. Analysis must begin within 6 hours of collection; when this is not possible, store at or below 4 degrees Centigrade and report length and temperature of storage with the results. In no case start analysis more than 6 hours after grab sample collection. If analysis of grab samples is begun within 2 hours of collection, cold storage is unnecessary. Grab samples may be collected in clean liter plastic bottles that have been washed, rinsed and dried in the dishwasher or by hand. The final rinsed cycle should be with distilled or deionized laboratory grade water. Composite samples are collected under refrigeration with an automatic sampler used solely for a certain sample type (i.e. only influent wastewater or only final effluent). The automatic sample containers are cleaned by the usual laboratory method noted above after each use. BOD analysis must begin immediately after the completion of the composite. Blend samples containing settleable solids with a homogenizer to permit representative sampling.

Laboratory Equipment and Instrumentation

300 ml BOD Incubation jars with plastic cups covering the flared mouth of the jars are used to maintain the water seal and prevent air intake into the jar during incubation.

BOD Incubator is thermostatically controlled at 20C \pm 1C and light is excluded to prevent possible photosynthetic production of Dissolved Oxygen.

Analysis Preparation

BOD Dilution Water is used to fill the 300 ml BOD jars after the sample is added. Dilution water is made by using HACH BOD Nutrient Buffer Pillows (cat. 14861-66) and Arrowhead Distilled water. Prepared Dilution Water is stored in 15 liter quantities in the BOD Incubator. Three 15 liter carboys are used solely for this purpose.

Nitrification Inhibitor is used to determine carbonaceous BOD upon request. HACH Nitrification Inhibitor Formula 2533 (cat. 2533) is used in premeasured dispenser designed for the 300 ml BOD jars.

Analytical Procedure

Make at least two dilutions of collected sample with dilution water to obtain Dissolved Oxygen uptake of at least 2 mg/L after 5 days. Use the BOD Dilution Range table to select sample size. Determine initial DO (refer to DO analysis, page 10) within 30 minutes of adding sample to BOD jar and topping off with dilution water. If using the Winkler Method for Dissolved Oxygen duplicate samples must be made for each dilution. One sample for immediate analysis and one sample for 5 days later. The sample to be stored and analyzed later needs to have a water seal and plastic cup applied before it is placed in the BOD Incubator.

BOD Dilution Range

SAMPLE ml	STARTING D.O.	ENDING D.O.	DEPLETION	BOD mg/L
2 ml	6.00	2.00	4.00	600.00
	6.00	4.00	2.00	300.00
3 ml	6.00	2.00	4.00	400.00
	6.00	4.00	2.00	200.00
5 ml	6.00	2.00	4.00	240.00
	6.00	4.00	2.00	120.00
10 ml	6.00	2.00	4.00	120.00
	6.00	4.00	2.00	60.00
25 ml	6.00	2.00	4.00	48.00
	6.00	4.00	2.00	24.00
50 ml	6.00	2.00	4.00	24.00
	6.00	4.00	2.00	12.00
100 ml	6.00	2.00	4.00	12.00
	6.00	4.00	2.00	6.00
150 ml	6.00	2.00	4.00	8.00
	6.00	4.00	2.00	4.00
200 ml	6.00	2.00	4.00	6.00
	6.00	4.00	2.00	3.00
250 ml	6.00	2.00	4.00	4.80
	6.00	4.00	2.00	2.40

Analytical Quality Control Procedures

- a) Dilution water check: Incubate a BOD bottle full of dilution water for 5 days at 20 degrees C. The dissolved oxygen uptake in 5 days should not be more than 0.2 mg/L and preferably not more than 0.1 mg/L.
- b) Glucose-glutamic acid check: Determine the 5-day BOD of a 2% dilution of the glucose- glutamic acid standard check solution using the HACH BOD Standard Solution (cat. 14865-10) for each set of samples analyzed.
- c) Control Charts
 - 1) Means chart for dilution water blank oxygen depletion.
 - 2) Means chart for glucose-glutamic acid standard.
 - 3) Range chart for duplicate analysis.

Calculation

$$\text{BOD5 mg/L} = \frac{[(\text{DO1} - \text{DO2}) \times 300]}{\text{mls of sample used in dilution}}$$

DO1 = DO of diluted sample immediately after preparation, mg/L.

DO2 = DO of diluted sample after 5 days incubation at 20 degrees C, mg/L.

For more detailed information, refer to procedure described on pages 5-2 to 5-6 of Standard Methods, 18th Edition.

Solids

This term includes analysis of all solids in solutions or soils; total, suspended and dissolved; fixed and volatile.

- 1) *Total Solids are suspended and dissolved solids, in other words all solids in a sample.*
- 2) *Total Suspended Solids are the solids left on the filter paper after a sample is filtered.*
- 3) *Total Dissolved Solids are the solids that pass through the filter paper.*
- 4) *Volatile Solids the solids that burn off when a dish or filter paper are ignited in the muffle oven*
- 5) *Fixed Solids are the solids remaining after ignition of a dish or filter paper.*
- 6) *Settleable Solids are the solids that settle from a well mixed sample in a set time period reported as milliliters per liter.*

Sampling and Storage

At least one liter of sample should be collected in a clean plastic bottle and kept cool until delivered to the laboratory for analysis. Solid samples should be collected in wide mouth jars and tightly capped to prevent any changes in the hydration of the sample. Samples should be brought to room temperature before analysis. Samples should be analyzed preferably within 24 hours and no longer than 7 days.

Laboratory Equipment and Instruments

- *Glass fiber filters – VWR grade no. 691, retention: 1.5 um, flow rate: fast, porosity: fine used for wastewater samples. VWR grade no. 693, retention: 1.1 um, flow rate: fast, porosity: fine used for drinking water or water with very low solid levels.*
- *Filtration apparatus with reservoir and coarse fritted disk as filter support.*
- *Vacuum pump*
- *Drying Oven operating at 103°C to 105°C*
- *Muffle Oven operating at 500 ± 50°C*
- *Analytical balance*
- *Evaporating Dishes*
- *Desiccator*
- *Imhoff Cone*
- *Settometer*

Analysis Preparation

The proper glass fiber filter is rinsed with approximately 250 milliliters of distilled water using the filtration apparatus. The filter is then dried in the drying oven at 103 to 105°C for at least 1 hour. The filter papers can be prepared in advance and stored in a desiccator until needed. They are to be weighed just before use. Evaporating dishes must be washed in the dishwasher with a final rinse of distilled or deionized water and after drying and reaching room temperature may be weighed just before use. If volatile solids are to be measured, ignite a clean evaporating dish at

500 + 50°C for 1 hour in a muffle furnace. Cool in desiccator, weigh, and store in desiccator until ready for use.

Analytical Procedure

Total Solids

Weigh a clean and dried evaporating dish; add 100mls of a well mixed solution with a graduated cylinder being careful to completely rinse with distilled water all residue from the cylinder. Place the dish in the 103°C to 105°C drying oven until the solution has completely evaporated. Then cool the dish in a desiccator and weigh. Repeat the process of drying, cooling desiccating and weighing until a constant weight is obtained or a weight change of less than 4% of the previous weight or 0.5 mg, whichever is less.

$$\text{Total Solids in mg/L} = \frac{(\text{weight of dish and residue} - \text{weight of dish}) \text{ mg} \times 1000}{\text{sample volume in ml}}$$

Total Suspended Solids

A known volume, up to one liter, of the well mixed sample is chosen to yield a dried residue of between 2.5 and 200 milligrams. The sample is measured in a graduated cylinder and poured onto the pre-weighed glass fiber filter which has been placed into the filtration apparatus. The graduated cylinder and filter are rinsed with three 10 milliliter volumes of distilled water. The filter is carefully removed and placed into the 103°C to 105°C drying oven for 1 hour. It is then removed to a desiccator, cooled and weighed until a constant weight is reached. Repeat the process of drying, cooling desiccating and weighing until a constant weight is obtained or a weight change of less than 4% of the previous weight or 0.5 mg, whichever is less.

$$\text{Total Suspended Solids in mg/L} = \frac{(\text{weight of filter and residue} - \text{weight of filter}) \text{ mg} \times 1000}{\text{sample volume in ml}}$$

Total Dissolved Solids

A known volume, up to 100 mls, of the well mixed sample is filtered through a clean 691 filter. The graduated cylinder and filter are rinsed with three 10 milliliter volumes of distilled water. Weigh a clean and dried evaporating dish; add all of the filtrate being careful to completely rinse with distilled water all residue from the filter flask. Place the dish in the 103°C to 105°C drying oven until the solution has completely evaporated. Then cool the dish in a desiccator and weigh. Repeat the process of drying, cooling desiccating and weighing until a constant weight is obtained or a weight change of less than 4% of the previous weight or 0.5 mg, whichever is less.

$$\text{Total Dissolved Solids in mg/L} = \frac{(\text{weight of dish and residue} - \text{weight of dish}) \text{ mg} \times 1000}{\text{sample volume in ml}}$$

Volatile and Fixed Solids

The residue from the three above solids tests are ignited to a constant weight at 500 ± 50°C. The volatile solids are those solids lost on ignition. Fixed solids are the residue that remains after ignition.

$$\text{Volatile solids in mg/L} = \frac{(A - B) \times 1000}{\text{sample volume in ml}}$$

$$\text{Fixed solids in mg/L} = \frac{(B - C) \times 1000}{\text{sample volume in ml}}$$

A = weight of residue and dish or filter before ignition in mg

B = weight of residue and dish or filter after ignition in mg C = weight of dish or filter in mg

Settleable Solids

In determining settleable solids in surface water, wastewater raw influent or final effluent an Imhoff cone is used. An Imhoff cone is filled to the 1-L mark with a well-mixed sample. Settle for 45 minutes, gently agitate sample near the sides of the cone with a rod or by spinning, settle 15 minutes longer and record volume of settleable solids in the cone as milliliters per liter. For determining settleable solids in our activated sludge a settlometer is used. A settlometer is filled with 2 liters of a well-mixed sample of activated sludge. Settle for 30 minutes and record volume of settleable solids in the cylinder as milliliter per liter.

For more detailed information, refer to procedure described on pages 2-53 to 2-57 of Standard Methods, 18th Edition.

Microbiological

This microbiological test is used to identify the presence of coliform bacteria that ferment lactose with gas and acid formation within 48 ± 2 hours when incubated at 35°C . The multiple tube technique using 5, 10 or 15 tubes of double and single strength Lauryl Tryptose Broth or 51 or 97 well Quanti-Trays of Colilert yields results that give an estimate of the mean density of coliforms in the sample. The results are reported as the Most Probable Number (MPN). A Presence-Absence test can also be performed using Colilert in a 100 ml sample. The analysis using Colilert only requires a 24 to 28 hours incubation at 35°C . It is extremely important that the sample being analyzed is well mixed.

Sampling and Storage

Samples of at least 100 mls are to be collected in sterile containers. If chlorine is present in the sample there must be sodium thiosulfate added to the sample. If samples are kept cool at approximately 4°C the following holding times apply:

- 1) 30 hours for drinking water*
- 2) 8 hours for surface or recreational waters*
- 3) 6 hours for wastewater*

Laboratory Equipment and Instruments

Coliform Incubator operating at $35 \pm 0.5^{\circ}\text{C}$ Water Bath

operating at $44.5 \pm 0.2^{\circ}\text{C}$ Autoclave

Sterilizing Oven

Sterile pipettes of various volumes Test tubes

Test tube racks Vials

Sterile inoculating loops

Sterile containers for sample collection (IDEXX 120 ml sterile sample vessels w/sodium thiosulfate and Nasco

Whirl-Pak 4 oz Thio-bags)

IDEXX Colilert and Comparator

IDEXX 51 Well Quanti-Trays and 97 Well Quanti-Trays and Comparators IDEXX Quanti-Tray

Sealer

Analysis Preparation

Lauryl Tryptose single strength Broth is made by adding 35.6 grams of BD Difco Lauryl Tryptose broth to one liter of Arrowhead distilled water. Ten milliliters of this mixture is then pipetted into 18mm x 150mm culture tubes containing inverted 12mm x 35mm shell vials and

capped. These tubes are then sterilized in an autoclave at 121C for 15 minutes after temperature has reached 121C. Total time in the autoclave should not exceed 45 minutes and the final pH should be 6.8 ± 0.2 .

Lauryl Tryptose double strength Broth is made by adding 71.2 grams of BD Difco Lauryl Tryptose broth to one liter of Arrowhead distilled water. Ten milliliters of this mixture is then pipetted into 22mm x 175mm culture tubes containing inverted 12mm x 35mm shell vials and capped. These tubes are then sterilized in an autoclave at 121C for 15 minutes after temperature has reached 121C. Total time in the autoclave should not exceed 45 minutes and the final pH should be 6.8 ± 0.2 .

Brilliant Green Bile is made by adding 40 grams of BD Difco Brilliant Green Bile Both 2% to one liter of Arrowhead distilled water. Ten milliliters of this mixture is pipetted into 18mm x 150mm culture tubes containing inverted 12mm x 35mm shell vials and capped. These tubes are then sterilized in an autoclave at 121C for 15 minutes after temperature has reached 121C. Total time in the autoclave should not exceed 45 minutes and the final pH should be 7.2 ± 0.2 .

EC Medium with MUG is made by adding 37.1 grams of BD Difco EC Medium with MUG to one liter of Arrowhead distilled water. Ten milliliters of this mixture is pipetted into 18mm x 150mm culture tubes containing inverted 12mm x 35mm shell vials and capped. These tubes are then sterilized in an autoclave at 121C for 15 minutes after temperature has reached 121C. Total time in the autoclave should not exceed 45 minutes and the final pH should be 6.9 ± 0.2 .

EC Medium is made by adding 37.0 grams of BD Difco EC Medium to one liter of Arrowhead distilled water. Ten milliliters of this mixture is pipetted into 18mm x 150mm culture tubes containing inverted 12mm x 35mm shell vials and capped. These tubes are then sterilized in an autoclave at 121C for 15 minutes after temperature has reached 121C. Total time in the autoclave should not exceed 45 minutes and the final pH should be 6.9 ± 0.2 .

Analytical Procedure

Multiple-Tube Fermentation Coliform – MPN and P/A Presumptive Phase

Five Tube Test

Twenty milliliters of the sample is added to each of five tubes of Lauryl Tryptose double strength broth. This utilizes 100 mls of sample for the analysis. These 5 tubes are then placed in a 35 ± 0.5 C incubator for 48 ± 2 hours. At 24 hours they are checked for growth, gas and/or acid production. If present this indicates a positive presumptive reaction and these tubes should be transferred to the confirmed phase. If growth, gas and/or acid production are absent at 24 hours, the tubes should be checked again at 48 ± 2 hours. If present at 48 hours, these tubes are also positive presumptive and should be transferred to the confirmed phase. Any samples absent of growth, gas and/or acid production are considered negative presumptive and are complete.

Ten Tube Test

Ten milliliters of the sample is added to each of ten tubes of Lauryl Tryptose double strength broth. This utilizes 100 mls of sample for the analysis. These 10 tubes are then placed in a 35 ± 0.5 C incubator for 48 ± 2 hours. At 24 hours they are checked for growth, gas and/or acid production. If present this indicates a positive presumptive reaction and these tubes should be transferred to the confirmed phase. If growth, gas and/or acid production are absent at 24 hours, the tubes should be checked again at 48 ± 2 hours. If present at 48 hours, these tubes are also

positive presumptive and should be transferred to the confirmed phase. Any samples absent of growth, gas and /or acid production are considered negative presumptive and are complete.

Fifteen Tube Test

Ten milliliters of the sample is added to five tubes of Lauryl Tryptose double strength broth, one milliliter of sample is added to five tubes of Lauryl Tryptose single strength broth and one tenth of a milliliter is added to five tubes of Lauryl Tryptose single strength broth. These 15 tubes are then placed in a $35 \pm 0.5^{\circ}\text{C}$ incubator for 48 ± 2 hours. At 24 hours they are checked for growth, gas and/or acid production. If present this indicates a positive presumptive reaction and these tubes should be transferred to the confirmed phase. If growth, gas and/or acid production are absent at 24 hours, the tubes should be checked again at 48 ± 2 hours. If present at 48 hours, these tubes are also positive presumptive and should be transferred to the confirmed phase. Any samples absent of growth, gas and /or acid production are considered negative presumptive and are complete.

Confirmed Phase

Brilliant Green Bile is used to confirm a positive presumptive as total coliform. Positive presumptive tubes are transferred with a sterile platinum loop from each positive Lauryl Tryptose tubes to a sterile BGB tube. These tubes are then placed in a $35 \pm 0.5^{\circ}\text{C}$ incubator for 48 ± 2 hours. At 24 hours they are checked for growth, gas and/or acid production. If present this indicates a positive confirmation reaction. If growth, gas and/or acid production are absent at 24 hours, the tubes should be checked again at 48 ± 2 hours. If present at 48 hours, these tubes are also positive confirmed phase. Any samples absent of growth, gas and /or acid production are considered negative for total coliform and are complete.

EC Medium is used to confirm a positive presumptive as fecal coliform. Positive presumptive tubes are transferred with a sterile platinum loop from each positive Lauryl Tryptose tubes to a sterile EC Medium tube. These tubes are then placed in a $44.5 \pm 0.2^{\circ}\text{C}$ water bath for 24 ± 2 hours. Gas production within 24 ± 2 hours is considered a positive fecal coliform reaction and no gas production within this time is a negative reaction.

EC Medium with MUG is used to confirm a positive presumptive as E.coli coliform. Positive presumptive tubes are transferred with a sterile platinum loop from each positive Lauryl Tryptose tubes to a sterile EC Medium with MUG tube. These tubes are then placed in a $44.5 \pm 0.2^{\circ}\text{C}$ water bath for 24 ± 2 hours. A bluish fluorescence under long-wave UV light and gas production within 24 ± 2 hours is considered a positive E. coli coliform reaction and no gas production within this time is a negative reaction.

Colilert – MPN and P/A

A 100 ml sample is collected or placed in a 120 ml sterile container and the content of one 'Snap Pack' of Colilert media is added. The sample is incubated at 35°C , after 24 hours but not longer than 28 hours the sample is compared to the provided comparator. The results are interpreted as follows:

Less yellow than Comparator	Negative for total coliforms and E.coli Yellow
\geq to Comparator Positive for total coliforms	
Yellow and fluorescence \geq to Comparator	Positive for total coliforms and E.coli

A 100 ml sample is collected or placed in a 120 ml sterile container and the content of one 'Snap Pack' of Colilert media is added, When the media has dissolved the sample is added to the appropriate Quanti-tray and sealed using the Quanti- tray Sealer. The Quanti-tray is then placed in a $35 \pm 0.5^{\circ}\text{C}$ incubator for 24 ± 4 hours. At 24 hours but not longer than 28 hours they are

checked for the above color production. The results can be interpreted using the MPN chart for either 51 or 97 wells provided by IDEXX.

Analytical Quality Control Procedures

Completed Test

The completed test is done on 10% of the positive confirmed coliform tubes or at least once quarterly. This test is used to provide quality control data and double confirm the presence of coliform bacteria. This test is done by using a sterile loop to transfer sample from a tube of BGB showing gas production to a LES Endo agar plate. The resulting agar plates are inverted and incubated at $35 \pm 0.5^{\circ}\text{C}$ for 24 ± 2 hours. Three types of isolated colonies will develop: typical, atypical and negative. Typical colonies are pink to red with a green metallic surface sheen. Atypical colonies are red, pink, white or colorless without surface sheen. All other colonies present are negative. Choose a well isolated typical colony, transfer it with a sterile loop to a tube of single strength Lauryl Tryptose broth and incubate at $35 \pm 0.5^{\circ}\text{C}$. Check the tube for gas production at 24 and 48 hours. The presence of gas production in the secondary single strength Lauryl Tryptose tube is a positive result for the completed test. The Colilert test does not require a completed test.

Interpretation

The number of tubes that confirm as positive in Brilliant Green Bile, EC Medium or EC Medium with MUG are counted and the following tables are used to calculate the MPN (Most Probable Number) of total coliform, fecal coliform or *E. coli* coliform present.

Most Probable Number Tables

Five tubes per dilution (10 ml in LTDS, 1.0 ml and 0.1 ml in LTSS)

Combination of Positive	MPN Index/100ml
4 3 0	27
4 3 1	33
4 4 0	34
5 0 0	23
5 0 1	30
5 0 2	40
5 1 0	30
5 1 1	50
5 1 2	60
5 2 0	50
5 2 1	70
5 2 2	90
5 3 0	80
5 3 1	110
5 3 2	140
5 3 3	170
5 4 0	130
5 4 1	170
5 4 2	220
5 4 3	280
5 4 4	350
5 5 0	240
5 5 1	300
5 5 2	500
5 5 3	900
5 5 4	1600

Microbiological Examination

1. Laboratory Equipment and Instrumentation

- a) Thermometer/temperature-recording instruments: Check accuracy of thermometers or recording instruments semiannually against a certified National Institute of Standards and Technology thermometer. For general purposes use thermometers graduated in increments of 0.5 degrees or less. For a 44.5 degree C water bath, use a submersible thermometer graduated to 0.2 degrees C or less. Record temperature checks data in a quality control log. Mark calibration corrections on each thermometer used with an incubator and refrigerator.*
- b) Balance: Wipe balance before and after each use with a soft brush made of such material as camel's hair. Check weights monthly against certified weights. For weighing 2 g or less, use an analytical balance with a sensitivity less than 1 mg at a 10-g load. For larger quantities, use a pan balance with a sensitivity of 0.1 g at a 150-g load.*
- c) pH meter: Standardize pH meter with at least two standard buffers (pH 4.0, 7.0, or 10.0) and compensate for temperature before each series of tests. Date buffer solutions when opened.*
- d) Water deionization unit: The conductivity is monitored continually when the unit is in use and the filters are changed according to manufacturer's instructions and usage.*
- e) Media dispensing apparatus: Check accuracy of volumes dispensed with a graduated cylinder at start of each volume change. At the end of work day, break apparatus down into parts, wash, rinse with reagent water, and dry.*
- f) Hot-air oven: Test performance quarterly with commercially available spore strips or spore suspensions. Monitor temperature with a thermometer accurate in the 160 to 180 degree C range and record results. Use heat-indicating tape to identify supplies and materials that have been exposed to sterilization temperatures.*
- g) Autoclave: Record items sterilized, temperature, pressure, and time for each run. Check operating temperature weekly with a minimum/maximum thermometer. Test performance with spore strips or suspensions monthly. Use heat-indicating tape to identify supplies and materials that have been sterilized.*
- h) Refrigerator: Check and record temperature daily and clean monthly. Identify and date materials stored.*
- i) Water bath: Monitor and record temperature daily. Use only stainless steel, plastic-coated, or other corrosion-proof racks.*
- j) Incubator: Check and record temperature twice daily (morning and afternoon) on the shelf areas in use. If a glass thermometer is used, submerge bulb and stem in water or glycerine to the stem mark.*

2. Laboratory Supplies

- a) Glassware: Before each use, examine glassware and discard items with chipped edges or etched inner surfaces. Test for inhibitory residues on glassware and plasticware annually and before using a new supply of detergent.*
- b) Reagent-grade water quality: Test for bacteriological quality of water at least annually.*

- c) *Reagents: Use only chemicals of ACS or equivalent grade. Date chemicals and reagents when received and when first opened for use. Label prepared reagents with name and concentration, date prepared, and initials of preparer.*
- d) *Culture media: Prepare media in containers that are at least twice the volume of the medium being prepared. Identify and date prepared media. Prepare all media in distilled water of proven quality. Water volumes and media are measured with graduates or pipettes conforming to NIST and APHA standards. Check pH of a portion of each medium after sterilization and cooling. Results are recorded in the media prep logbook. If the pH difference is larger than 0.5 units, discard the batch and resolve the problem. Examine prepared media for unusual color, darkening, or precipitation and record observations. If any of the above occur, discard the medium. Expose lauryl tryptose, BGB broth, etc. to sterilization temperature of 121 degrees C for a period of 15 minutes. Remove sterilized media from autoclave as soon as chamber pressure reaches zero. Check effectiveness of sterilization with each run by using Bacillus stearothermophilus spore suspensions or strips inside glassware. Lauryl tryptose and BGB media prepared in loose-cap tubes can be stored up to one week at 4 degrees C. Media prepared in screw-cap tubes can be stored up to 3 months at 4 degrees C. If fermentation tube media are refrigerated, incubate overnight before use and check for false positive gas bubbles. Maintain in a bound book a complete record of each batch of medium prepared with name of preparer and date, name and lot number of medium, amount of medium weighed, volume of medium prepared, sterilization time and temperature, pH measurements and adjustments.*

3. Analytical Quality Control Procedures

- a) *General quality control procedures: For multiple-tube procedures, check sterility of media, dilution water, and glassware. To test sterility of media, subject a representative portion of each batch to incubation at an appropriate temperature for 24 to 48 hours and observe for growth. Check each batch of dilution water for sterility by adding 20 ml water to 100 ml of a non-selective broth. Incubate at 35±0.5 degrees C for 24 hours and observe for growth. If any contamination is indicated, reflect analytical data from samples tested with these materials and request immediate resampling. Perform duplicate analyses on 5% of samples and on at least one sample per test run.*
- b) *For each lot of medium, check with known positive and negative control cultures for the organism(s) under test. An appropriate set of control microbial cultures should be available and maintained in the laboratory. The following table provides examples of test cultures.*

	LTDS	LTSS	BGB	EC Medium	Colilert
			Total	Fecal	Total/Fecal
Escherichia coli	POS	POS	POS	POS	POS/POS
Enterobacter aerogenes	POS	POS	POS	NEG	POS/NEG
Pseudomonas aeruginosa	NEG	NEG	NEG	NEG	NEG/NEG
Klebsiella pneumoniae	POS	POS	POS	NEG	POS/NEG

4. Control Charts

- a) *Means chart for media sterility check.*
- b) *Means chart for dilution water sterility check.*
- c) *Means chart for known positive/negative control standards.*
- d) *Range chart for duplicate analysis (water and wastewater).*

5. Records and Data Reporting

Keep records of microbiological analyses for at least 5 years.

Biochemical Oxygen Demand

1. Sampling and Storage

- a) *If analysis is begun within 2 hours of collection, cold storage is unnecessary. If analysis is not started within 2 hours of sample collection, keep sample at or below 4 degrees C from the time of collection. Begin analysis within 6 hours of collection; when this is not possible, store at or below 4 degrees C and report length and temperature of storage with the results. In no case start analysis more than 24 hours after sample collection.*

2. Laboratory Equipment and Instrumentation

- a) *Thermometer/temperature-recording instruments: Check accuracy of thermometers or recording instruments semiannually against a certified National Institute of Standards and Technology thermometer. Use a general purpose thermometer graduated in increments of 0.5 degrees or less. Record temperature check data in a quality control log. Mark calibration corrections on each thermometer used with an incubator, refrigerator, or freezer.*
- b) *Refrigerator: Check and record temperature daily and clean monthly. Identify and date materials stored.*
- c) *Incubator: Check and record temperature once daily on the shelf areas in use. If a glass thermometer is used, submerge bulb and stem in water or glycerine to the stem mark. Exclude all light to prevent possibility of photosynthetic production of dissolved oxygen.*

3. Laboratory Supplies

- a) *Incubation bottles: Clean bottles with a detergent, rinse thoroughly, and drain before use. As a precaution against drawing air into the dilution bottle during incubation, use a water-seal.*

4. Analytical Quality Control Procedures

- a) *Dilution water check: Incubate a BOD bottle full of dilution water for 5 days at 20 degrees C. The dissolved oxygen uptake in 5 days should not be more than 0.2 mg/L and preferably not more than 0.1 mg/L.*
- b) *Glucose-glutamic acid check: Determine the 5-day BOD of a 2% dilution of the glucose- glutamic acid standard check solution for each sample analysis.*

5. Control Charts

- a) *Means chart for dilution water blank oxygen depletion.*
- b) *Means chart for glucose-glutamic acid standard.*
- c) *Range chart for duplicate analysis.*

Chemical Oxygen Demand

1. Sampling and Storage

- a) *Preferably collect samples in glass bottles. If delay before analysis is unavoidable, preserve sample by acidification to pH < 2 using concentrated sulfuric acid.*

2. Analytical Quality Control Procedures

- a) *Preparation of calibration curve: Prepare at least five standards from potassium hydrogen phthalate solution with COD equivalents from 20 to 900 micrograms oxygen per liter. Prepare calibration curve for each new lot of tubes or ampules or when standards differ by $> 5\%$ from calibration curve.*

b) Standard and blank: Prepare one blank and one standard with each set of samples run.

c) Perform duplicate analysis on a once per week basis.

3. Control Charts

a) Means chart for control standard.

b) Range chart for duplicate analysis.

Chlorine Residual

1. Sampling and Storage

a) Start chlorine determinations immediately after sampling, avoiding excessive light and agitation. Do not store samples to be analyzed for chlorine.

2. Analytical Quality Control Procedures

a) Volume of sample: Select a sample volume that will require no more than 20 ml 0.01N sodium thiosulfate and no less than 0.2 ml for the starch-iodide end point. For a chlorine range of 1 to 10 mg/L, use a 500 ml sample.

b) Blank titration: Correct result of sample titration by determining blank contributed by oxidizing or reducing reagent impurities. Before calculating the chlorine concentration, subtract or add the blank titration from the sample titration.

c) Perform duplicate analysis on a once per week basis.

3. Control Charts

a) Range chart for duplicate analysis.

Dissolved Oxygen

1. Sampling and Storage

a) Collect samples very carefully. Do not let sample remain in contact with air or be agitated. Determine DO immediately on all samples. Samples with no iodine demand may be stored for a few hours without change after adding manganese sulfate solution, alkali-iodide solution, and sulfuric acid, followed by shaking in the usual way. Protect stored samples from strong sunlight and titrate as soon as possible.

1. Analytical Quality Control Procedures

a) Perform duplicate analysis on a once per week basis.

2. Control Charts

a) Range chart for duplicate analysis.

pH

1. Sampling and Storage

a) Samples should be analyzed immediately.

2. Analytical Quality Control Procedures

a) Instrument calibration: Standardize pH meter by placing electrode in initial buffer solution of pH 4 and set. Select a second buffer within 2 pH units of the sample pH and bring sample and buffer to same temperature. Select a third buffer below pH 10, approximately 3 pH units different from the second. Reading should be within 0.1 unit for pH of the third buffer. Standardization should be performed before each measurement of sample groups.

b) Perform duplicate analysis on a once per week basis.

3. Control Charts

- a) Means chart for buffer standards.
- b) Range chart for duplicate analysis.

Solids (filterable, nonfilterable, volatile)

1. Sampling and Storage

- a) Use resistant glass or plastic bottles. Begin analysis as soon as possible. Refrigerate sample at 4 degrees C up to analysis to minimize microbiological decomposition of solids.

2. Analytical Quality Control Procedures

- a) Perform duplicate analysis of samples on a once per week basis.

3. Control Charts

- a) Range chart for duplicate analysis.

4. Acceptance Limits and Control Chart Analysis

- a) Warning and Control Limits
 - i. Each chart will contain upper and lower warning limits which commonly will equal a $\pm 10\%$ limit.
 - ii. Each chart will contain upper and lower control limits which commonly will equal a $\pm 20\%$ limit.

PREVENTIVE MAINTENANCE

pH

Electrode

On a weekly basis perform the following: 1) inspect the electrode for scratches, cracks, salt crystal build-up, or membrane/junction deposits; 2) Rinse off any salt build-up with distilled water, and remove any membrane/junction deposits as directed in cleaning procedures; 3) Drain the reference chamber, flush it with fresh filling solution and refill the chamber.

Dissolved Oxygen Probe

Change the batteries when the pH meter can no longer read 13.40 or greater. The membrane module with normal laboratory use is three months. Probe should be stored in humid environment to prevent drying out of the gel electrolyte. Calibrate before each use as instructed in the user guide.

Analytical Balance

Annual maintenance and calibration is performed by a contracted certified balance service company, Watson Brothers, Inc. from Burbank, CA.

Autoclave

After each use, drain all water from unit and keep door open.

Vacuum Pump

Check oil level before operating. Change oil when inspection shows contamination with water or debris.

ASSESSMENT OF PRECISION AND ACCURACY

Performance Evaluation Samples

At least annually performance evaluation samples are run on each analyte that the laboratory is certified to analyze. This year we utilized ERA, Environmental Resource Associates. We completed water pollution proficiency testing for total and fecal coliform (MPN), pH, BOD, cBOD, COD, TSS, TS TDS, alkalinity, conductivity, total chlorine residual and turbidity. We also performed the water supply proficiency testing for alkalinity, conductivity, TDS, pH, turbidity, free and total chlorine residual and total, fecal and E.coli coliform (P/A) using the multiple tube technique and the Colilert method. We tested a water supply source sample for E.coli using the multiple tube method, confirming with EC Medium + MUG.

CORRECTIVE ACTION

Control Charts

If control limits or acceptable limits are not met by an analysis then the analysis shall be repeated immediately. If the repeat analysis does not meet the limits then the analysis shall be discontinued and the problem corrected.

If two out of three successive points exceed a warning limit, analyze another sample. If the next point is less than the warning limit, continue analyses; if next point exceeds the warning limit, discontinue analyses and correct the problem.

If six successive samples are above the central line, analyze another sample. If the next point is below the central line, continue analyses; if the next point is on the same side, discontinue analyses and correct the problem.

Corrective Action

- a) Control limit: If one measurement exceeds a control limit, repeat the analysis immediately. If the repeat is within the control limit, continue analyses. If the repeat exceeds the control limit, discontinue analyses and correct the problem.*
- b) Warning limit: If two out of three successive points exceed a warning limit, analyze another sample. If the next point is less than the warning limit, continue analyses. If the next point exceeds the warning limit, discontinue analyses and correct the problem.*
- c) Central line: If six successive samples are above the central line, analyze another sample. If the next point is below the central line, continue analyses. If the next point is on the same side, discontinue analyses and correct the problem.*
- d) Method precision: In the means and control charts, if measurements never or rarely exceed the warning limit, recalculate the warning limit and control limit using the 20 most recent data points.*

Second Party Review

- a) Control charts shall be reviewed on a weekly basis by the laboratory director to verify that analysis is being performed in an accurate and precise manner.*

Total Dissolved Solids

Sampling and Storage

At least one liter of sample should be collected in a clean plastic bottle and kept cool until delivered to the laboratory for analysis. Samples should be analyzed preferably within 24 hours and no longer than 7 days.

Principle

A well mixed sample is filtered through a standard glass fiber filter and the filtrate is dried in a weighed dish to a constant weight at 180°C. The filtrate from the total suspended solids test may be used for this analysis. The final weight minus the original weight, multiplied by 1,000,000, divided by the sample volume yields the total dissolved solids in milligrams per liter or parts per million.

Apparatus

- 1) Glass fiber filters – VWR grade no. 691, retention: 1.5 um, flow rate: fast, porosity: fine.
- 2) Filtration apparatus with reservoir and coarse fritted disk as filter support.
- 3) Suction flask
- 4) Vacuum pump
- 5) Drying oven operating at 180°C
- 6) Porcelain crucibles
- 7) Graduated cylinder
- 8) Analytical balance
- 9) Desiccator

Procedure

Preparation

A clean and dried glass fiber filter is used to filter a well mixed volume of the sample. A porcelain crucible is washed, dried in the 180°C drying oven for 1 hour, cooled for 15 to 20 minutes in a desiccator and weighed immediately prior to use.

Sample Analysis

A 100ml volume of the well mixed sample measured in a graduated cylinder and poured onto the glass fiber filter which has been placed into the filtration apparatus that has a clean empty filtration flask. The graduated cylinder and filter are rinsed with three 10 milliliter volumes of distilled water. The filtrate with washings is transferred to the weighed, clean and dried porcelain crucible which is first dried in the drying oven (103*-105*) and finally in a 180°C oven for one hour. It is then removed to a desiccator, cooled and weighed until a constant weight is reached. Repeat the process of drying, cooling desiccating and weighing until a constant weight is obtained or a weight change of less than 4% of the previous weight or 0.5 mg, whichever is less.

Calculations

$$\text{Total Dissolved Solids in mg/L} = \frac{(\text{weight of dish and residue} - \text{weight of dish})\text{mg} \times 1,000}{\text{sample volume in ml}}$$

Total Suspended Solids

Sampling and Storage

At least one liter of sample should be collected in a clean plastic bottle and kept cool until delivered to the laboratory for analysis. Samples should be analyzed preferably within 24 hours and no longer than 7 days.

Principle

A known volume of a well mixed sample is filtered through a pre-weighed, washed and dried glass-fiber filter. The filter is dried to a constant weight in a 102* to 105* C drying oven. The final weight minus the original weight, multiplied by 1,000,000, divided by the sample volume yields the total suspended solids in milligrams per liter or parts per million.

Apparatus

- 1) Glass fiber filters – VWR grade no. 691, retention: 1.5 um, flow rate: fast, porosity: fine.
- 2) Filtration apparatus with reservoir and coarse fritted disk as filter support.
- 3) Suction flask
- 4) Vacuum pump
- 5) Drying oven operating at 102°C to 105°C
- 6) Graduated cylinder
- 7) Analytical balance
- 8) Desiccator

Procedure

Preparation

A glass fiber filter is washed with approximately 250 milliliters of distilled water using the filtration apparatus. The filter is then dried in the drying oven for 1 hour, cooled for 15 to 20 minutes in a desiccator and weighed.

Sample Analysis

A known volume, up to one liter, of the well mixed sample is chosen to yield a dried residue of between 2.5 and 200 milligrams. The sample is measured in a graduated cylinder and poured onto the pre-weighed glass fiber filter which has been placed into the filtration apparatus. The graduated cylinder and filter are rinsed with three 10 milliliter volumes of distilled water. The filter is carefully removed and placed into the 102°C to 105°C drying oven for 1 hour. It is then removed to a desiccator, cooled and weighed until a constant weight is reached.

Calculations

$$\text{Total suspended solids mg/L} = \frac{(A - B) \times 1,000,000}{\text{Sample volume}}$$

A = weight of filter and dried residue in g
B = weight of washed and dried filter in g
Sample volume in mls

Modification of EPA Method 1631e

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Sample bottle cleaning methods were modified from that described in EPA 1631e in order to shorten cleaning time and reduce hazards to staff. It was felt that heating concentrated acid for a period of 48 hours would result in unsafe working conditions and would unnecessarily increase the wear and tear on equipment.

It was determined through R&D that bottles cleaned under the following conditions resulted in mercury levels below the detection limit set forth in EPA 1631e (modifications are listed according to section number):

6.1.2.1 New environmentally clean bottles are cleaned by heating to 65-70°C in 7.5N HNO₃ for 8 hours or soaking for 3 days unheated in the same acid. The bottles are rinsed 5 times with reagent water and filled with reagent water containing 0.5% HCl. The bottles are capped tightly and placed on a mercury free clean bench until the outside surfaces are dry. The bottles are double-bagged in new polyethylene zip-type bags until needed, and stored in the original cardboard container.

6.1.2.2 Used sample bottles are not re-cleaned and/or re-used.

6.1.3.1 Filter—0.45-µm, 15-mm diameter capsule filter (Gelman Supor 12175, or equivalent) or disposable filter unit (Nalge Nunc Inc. Part # 157-0045) prepared using Method # MPSL- 101.

8.4 Samples may also be filtered in the lab with a cleaned disposable filter units

8.5 Preservation- samples are preserved by adding 0.5% v/v BrCl to the sample bottle. More BrCl may be added if a permanent yellow color is not obtained. Preserved samples are stable for up to 90 days of the date of collection. (This is a clarification, rather than a modification.)

9.1.7, 9.3 QA samples are performed at a rate of 5%. Varying matrices are not analyzed simultaneously.

11.1.1 30 mL aliquots of sample are used for analysis.

11.2.1.1 100µL of NH₂OH is added to destroy BrCl

Modification of EPA Method 1638

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It was determined through R&D samples analyzed under the following conditions resulted in trace metal levels below the detection limits set forth in EPA 1638 (modifications are listed according to section number):

Note: High salinity (greater than 3 pp thousand) and boron (B) samples are analyzed with a High Resolution ICP-MS, also using this method.

1.2 This method is applicable to the following elements:

Analyte	Symbol	CAS Registry Number
Barium	Ba	7440-39-3
Boron	B	7440-42-8
Aluminum	Al	7429-90-5
Antimony	Sb	7440-36-0
Arsenic	As	7440-38-2
Cadmium	Cd	7440-43-9
Chromium	Cr	7440-47-3
Cobalt	Co	7440-48-4
Copper	Cu	7440-50-8
Lead	Pb	7439-92-1
Manganese	Mn	7439-96-5
Nickel	Ni	7440-02-0
Selenium	Se	7782-49-2
Silver	Ag	7440-22-4
Thallium	Tl	7440-28-0
Zinc	Zn	7440-66-6

A separate analytical run is required to analyze the following elements:

Analyte	Symbol	CAS Registry Number
Calcium	Ca	7440-70-2
Magnesium	Mg	7439-95-4
Phosphorus	P	7723-14-0
Potassium	K	7440-09-7

6.10.3 Pre-cleaned 60mL polypropylene syringes are used with a Gelman Supor 0.45µm syringe filter, instead of a tubing set and capsule filter.

7.3 – 7.4 Multi-element stock standards are purchased from Perkin Elmer.

8.0 Samples are collected, etc. according to Modified EPA 1669.

9.1.4 The analyst will spike at least 5% of the samples.

9.1.5 Ongoing precision and recovery (OPR) are not maintained with the ICP-MS. However, continuing calibration verification (CCV) and SRM percent recovery are monitored.

9.2.4 All standards are obtained from an outside source; therefore a QCS is performed with each analysis.

9.7 OPR is not performed. ICV and CCV are analyzed continually, and sample spikes are performed with each batch.

11.3.1 Cleaning procedures for sample collection and analytical containers can be found in Method # MPSTL-101.

13.2 Values are reported to a maximum of 2 decimal places. Samples slightly greater than the ML may be reported to only 1 or 2 significant figures.

13.8 Sample results are blank corrected.

Appendix B3

Field Sampling SOP References, Data Sheets/Forms

REFERENCED FIELD SAMPLING STANDARD OPERATING PROCEDURES (SOPs)

SOPs Referenced:

Collection of Water Samples for Analysis of Conventional Constituents & Field Measurements:

California Department of Fish and Game - Marine Pollution Studies Laboratory (DFG-MPSL) Standard Operating Procedure (SOP), "Conducting Field Measurements and Field Collections of Water and Bed Sediment Samples in the Surface Water Ambient Monitoring Program", (MPSL- DFG_FieldSOP_v1.0, SOP Procedure Number: 1.0, Date: October 15, 2007)

(http://swamp.mpsl.mlml.calstate.edu/wp-content/uploads/2009/04/swamp_sop_field_measures_water_sediment_collection_v1_0.pdf)

Collection of Water Samples for Analysis of Trace Metals Constituents:

U.S. Environmental Protection Agency Method 1669, "Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels (July 1996)

http://water.epa.gov/scitech/methods/cwa/metals/upload/2007_07_10_methods_method_inorganics_1669.pdf

Modification of EPA Method 1669, Marine Pollution Studies Lab (see Appendix D) Brooks Rand Labs, "EPA

Method 1669 Field Sampling Quick Reference" (see Appendix D)

Example Field Data Sheets/Forms:

Field Measurement Data Sheet Sample Data
Sheet

Modification of EPA Method 1669

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It was determined through field blanks that samples collected under the following conditions resulted in metals levels below the detection limit set forth in EPA 1630, EPA 1631E, and EPA 1638. Modifications to EPA 1669 are listed according to section number:

- 6.1 Cleaning methods can be found in Method # MPSL-101.
- 6.3 Container sizes are chosen based on the quantity of sample needed for analysis. Trace metal samples are collected in pre-cleaned 60 mL HDPE bottles. Mercury and methylmercury samples are collected in prepared 250 mL glass bottles. Cleaning for methylmercury may is not required if environmentally cleaned bottles are tested prior to collection.
- 6.6 A glove box is not necessary as long as the collection container and sample can be handled cleanly. Demonstration of this is through field blanks.
- 6.7 Wind suits are not required.
- 6.14.3 Disposable Filter Units, 250 mL can be used for lab filtering. These are prepared according to Method # MPSL-101.
- 6.15.2-6.15.3 Tubing specifications and cleaning procedures can be found in Method # MPSL-101.
- 9.3 Bottle blanks and equipment blanks are often taken in the lab to ensure that all equipment is properly cleaned prior to collection.
- 9.4-9.5 Field blanks and duplicates are generated at a rate of 5% based on the total number of samples per sampling event. An event is defined as collection at successive sites, potentially over the course of several days, until the sampling team(s) returns to the lab.

10.1 Field equipment such as tubing or bottles is not recleaned.

EPA Method 1669

Field Sampling Quick Reference

EPA Method 1669 was designed to support water quality monitoring programs authorized under the Clean Water Act. This sampling method was developed by EPA specifically to address needs for measuring toxic metals at very low levels. In developing this method, EPA found that one of the greatest difficulties in measuring pollutants at these levels was preventing sample contamination during collection and transport. Therefore, this method is designed to provide the level of protection necessary to prevent contamination in nearly all situations.

The method requires that a two-member team participate in the collection of samples. “Clean Hands” is responsible for all procedures involving direct contact with the sample and sample container. “Dirty Hands” is responsible for preparing the sample containers for collection, operations of any machinery, and all other activities that could lead to contamination of the sample.

Prior to Beginning to Collect Samples

- *select and prepare an appropriate location for a sampling station*
- *establish, and eliminate if possible, any sources of contamination*
- *organize all equipment and ensure adequate supplies*
- *remove jewelry, watches, metallic items, etc.*
- *thoroughly cleanse and dry hands*

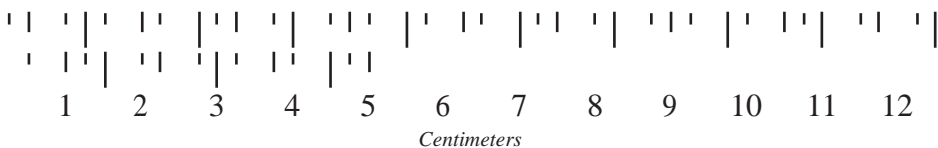
- *determine roles and responsibilities (“Clean Hands”/“Dirty Hands”)*
- *wear clean gloves at all times and change them frequently*
- *allow site to stabilize prior to collecting samples*

“Dirty Hands”

- *does not disturb sample source*
- *does not touch primary container bags*
- *does not touch sample containers*
- *does not touch “clean equipment”*
- *handles all “non-clean” materials*
- *arranges sampling materials*
- *opens/closes shipping containers*
- *opens/closes secondary container bags*
- *operates pump/metalic equipment*
- *fills out necessary documentation*

“Clean Hands”

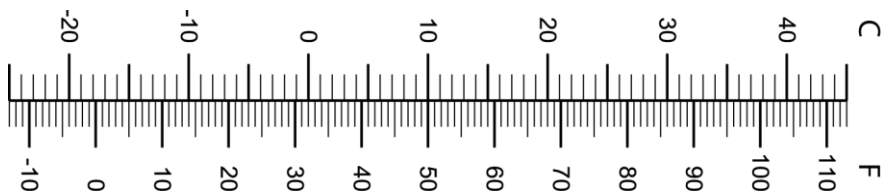
- *does not touch “non-clean” materials*
- *does not touch secondary container bags*
- *does not touch shipping containers*
- *handles all “clean” materials*
- *assembles clean sampling equipment*
- *assembles sample tubing & filters*
- *directly contacts sample source*
- *opens/closes primary container bags*
- *directly holds sample container*
- *submerges container/collects sample*



Container & Preservation Requirements for Aqueous Samples

Analyses	Container	Field Preserve	Temperature	Holding Time
Mercury EPA 1631	250-500 mL FLPE	no	ambient	P - 28 days A - 90 days
Methylmercury EPA 1630	250 mL FLPE	preferably	0-4 °C	P - 48 hrs. A - 180 days
Arsenic EPA 1632	125-250 mL HDPE	yes	0-4 °C	P - immediately A - 28 days
Trace Metals EPA 1638	125-250 mL HDPE	no	ambient	P - 14 days A - 180 days
Trace Metals EPA 1640 (seawater)	1-2 L HDPE	no	ambient	P - 14 days A - 180 days

Samples that will be filtered in the laboratory *must not* be preserved, *must* be kept cold, and *must* be rush shipped.



Packing & Shipping Instructions

- all sample containers must be sealed in double-bags
- all sample containers must be clearly labeled with a unique ID
- all sample containers must be documented on the COC

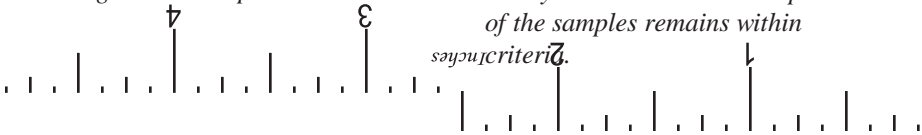
- all COCs must be filled out completely and signed
- all shipping containers must be sealed with custody seals

Ambient Samples

Samples that can be maintained at an ambient temperature may be shipped in a cardboard box at standard shipping rates. However, the samples must arrive at the laboratory before the preservation holding time has expired.

Temperature-Sensitive Samples

Samples that must maintain a specific temperature (e.g. 0-4 °C) must be shipped in a cooler packed with ice to arrive at the laboratory the following day. Distribute the ice evenly to ensure that the temperature of the samples remains within



Field Measurement Data Sheets

Date (mm/dd/yy)	Sample Site ID	Sample Site Name					Personnel			
Site Observations:										
YSI 6820V2 Measurements:										
Time (PST 24-hr) (hh:mm)	Temp. (°C)	Specific Cond. (mS/cm)	Cond. (mS/cm)	TDS (g/L)	Salinity (ppt)	pH (pH units)	Turbidity (NTU)	Dissolved Oxygen (%)	Dissolved Oxygen (mg/L)	

Date (mm/dd/yy)	Sample Site ID	Sample Site Name					Personnel			
Site Observations:										
YSI 6820V2 Measurements:										
Time (PST 24-hr) (hh:mm)	Temp. (°C)	Specific Cond. (mS/cm)	Cond. (mS/cm)	TDS (g/L)	Salinity (ppt)	pH (pH units)	Turbidity (NTU)	Dissolved Oxygen (%)	Dissolved Oxygen (mg/L)	

Date (mm/dd/yy)	Sample Site ID	Sample Site Name					Personnel			
Site Observations:										
YSI 6820V2 Measurements:										
Time (PST 24-hr) (hh:mm)	Temp. (°C)	Specific Cond. (mS/cm)	Cond. (mS/cm)	TDS (g/L)	Salinity (ppt)	pH (pH units)	Turbidity (NTU)	Dissolved Oxygen (%)	Dissolved Oxygen (mg/L)	

Water Quality Sampling Data Sheets

Date <i>(mm/dd/yyyy)</i>	Sample Site ID	Sample Site Name	Personnel				
Site Observations/Description/ Equipment Used, etc.:							
Water Quality Samples Collected							
Sample Depth _____ <i>(0.1 m)</i>	Total Mercury	Total Manganese	Total Nitrogen	Nitrate + Nitrite	Total Phosphorus	TDS	TSS
Time <i>(PST, 24-hr)</i> <i>(hh:mm)</i>							
Sample ID <i>(Site ID-Date)</i> <i>(yymmdd)</i>							
# sample bottles							
Duplicate ID <i>(Site ID-2-Date)</i> <i>(yymmdd)</i>							
# duplicate bottles							

Date <i>(mm/dd/yyyy)</i>	Sample Site ID	Sample Site Name	Personnel				
Site Observations/Description/ Equipment Used, etc:							
Water Quality Samples Collected							
Sample Depth _____ <i>(0.1 m)</i>	Total Mercury	Total Manganese	Total Nitrogen	Nitrate + Nitrite	Total Phosphorus	TDS	TSS
Time <i>(PST, 24-hr)</i> <i>(hh:mm)</i>							
Sample ID <i>(Site ID-Date)</i> <i>(yymmdd)</i>							
# sample bottles							
Duplicate ID <i>(Site ID-2-Date)</i> <i>(yymmdd)</i>							

Appendix E

YSI 6820V2 Sonde Field Instrument – Sensor Maintenance & Calibration SOPs

- (1) Owens Valley Indian Water Commission, YSI 6820V2 Sonde, Field Instrument – Sensor Maintenance & Calibration Standard Operating Procedures**
- (2) Owens Valley Indian Water Commission, YSI 6820V2 Calibration Record

Owens Valley Indian Water Commission

YSI 6820V2 Sonde

FIELD INSTRUMENT - SENSOR MAINTENANCE & CALIBRATION Standard Operating Procedures

**Version: 8/28/2008 (wipers eliminated)
Revised: 5/7/2012**

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FIELD INSTRUMENT - SENSOR MAINTENANCE & CALIBRATION

STANDARD OPERATING PROCEDURES

YSI 6820V2 Sonde

Sensors: Conductivity/Temperature (YSI 6560); pH (YSI 6561); Optical Dissolved Oxygen (YSI 6150 ROX); Optical Turbidity (YSI 6136)

Note: Always write on calibration solution bottles the date the bottle was opened. Always check on supplies that need to be ordered, and notify the Environmental Specialist (ES) if supplies are running low.

YSI technicians affirmed that turbidity and optical dissolved oxygen (ODO) probes wipers are not necessary for spot checking water quality during a phone conversation on 8/18/08 with ES. Optical probe wipers are only necessary for extended deployments of the sonde, and so all wipers were removed from the YSI 6820V2 sonde as of 8/28/08.

YSI 600QS Sonde - Sensor Maintenance & Calibration

I. Calibration Preparation (Refer to "Getting Ready to Calibrate" pages 2-34 to 2-35 in YSI Manual)

- 1) *Fill bucket full of tap water; fill clear container full of DI water*
- 2) *Remove YSI 6820V2 sonde from the storage solution and rinse it in the bucket of tap water, being very careful of the probes because the probe guard is not attached during calibrations.*
- 3) *Verify the accuracy of the temperature probe using the registered -20 to 110°C non-mercury glass thermometer stored in the cupboard. Be careful, it is fragile!*
 - *Attach the probe guard to the sonde.*
 - *Tape the calibration thermometer to the sonde so that the tip is even with the temperature probe tip (on the conductivity probe), and so you can read the thermometer.*
 - *Place the sonde in the bucket of tap water so that the temperature probe and thermometer tip are at the same depth.*
 - *Record both the sonde temperature and the thermometer temperature on the calibration sheet. (Note the digital YSI has a greater degree of precision than the thermometer, so for this general comparison +/- 0.5° is adequate).*
 - *Detach the thermometer and put it away and remove the sonde probe guard.*
- 4) *If necessary (if the conductivity/temperature probe vent holes are clogged) use the short handled black tube brush to brush out the YSI 6820V2 conductivity entrances; rinse well.*
- 5) *Rinse the YSI in bucket of tap water, then in cup of DI, shake off excess water and dry the outside of the sonde probes with a clean paper towel, being very careful of the probes because the probe guard is not attached and they are very vulnerable to damage. Hang on cupboard door knob with out touching the sonde tip to anything.*
- 6) *For calibrations, use the (laboratory) extended calibration cup, stored in the cupboard above the sink which was cleaned and rinsed in DI water and stored upside down on a clean paper towel to dry in the cupboard. The correct laboratory cup has volume lines drawn on it.*
- 7) *Read "Calibration Tips" page 2-34 in the YSI Manual and the calibration tips for each of the individual probes. Copies of these are appended to this document.*

II. Turbidity Calibration (Refer to TURBIDITY 2-POINT, pages 2-45 to 2-46 in the YSI Manual)

NOTE: Before calibrating the YSI 6136 turbidity sensor, pay attention to the following cautions:

- ♦ To properly calibrate the YSI turbidity sensors you **MUST** use standards that have been prepared properly (see page 2-45 in the YSI manual). Hach StableCal standards in various NTUs or dilutions of these can be used. You should enter the value of the Hach standards or dilutions during the calibration procedure.
 - ♦ When calibrating the 6136 turbidity sensor you **MUST** follow the instructions found in Section 2.6.1 in the YSI Manual (page 2-37 & 2-38) and use the calibration cup provided.
 - ♦ **One standard must be 0 NTU, and this standard must be calibrated first.**
 - ♦ **Calibrating turbidity is best done in a lab environment; calibrations in the field can result in errors.**
- 1) Rinse the YSI in bucket of tap water, then in cup of DI, shake off excess water and dry the outside of the sonde with a clean paper towel. Hang on cupboard door knob without touching the sonde sensors to anything. Be very careful because the probe guard is not attached the sensors are vulnerable and fragile.
 - 2) Clean the face of the turbidity sensor by rinsing it with DI water from the plastic squirt bottle labeled DI stored above the lab sink. Do not touch the optical lenses with anything other than a lint free cloth. The sonde and sensors **MUST** be clean before doing a zero calibration! The long calibration cup must be used for turbidity calibrations, and **MUST** be clean and free of dust, dirt, and sediments! Use only lint free clothes to clean and dry the sensors and the cal cup. Wipe off any fingerprints or smudges from the optics.
 - 3) Confirm that the output of the probe increases when you place your finger in front of (not touching!) the optics. If the probe does not show an increase in output with your finger in front of the optics, then you must stop the calibration and determine the cause of the problem.
 - 4) Install a clean wiper on the Sonde, enable the Sonde to wipe (Sonde Menu – Advanced – Sensor – Wipes=1), and check to see that the dissolved oxygen probe wiper rotates twice in the counter clockwise direction (looking down) and once in the clockwise direction, the turbidity wiper rotates twice in a clockwise direction (looking down) and once in the counter clockwise direction, and that both wipers park on the side of the probe opposite the sensor windows.
 - 5) Rinse the calibration cup with tap water and then with Distilled water and shake it as dry as possible.
 - 6) Place **225 ml** of distilled, deionized water (DI water) into the calibration cup provided with the sonde (marked on cup). Pour the DI water into the cal cup down the side of the cup so you do not aerate the sample. Check to make sure there are no air bubbles.
 - 7) Immerse the sonde in the water, setting the sonde on top of the calibration cup...do not engage the threads,
 - 8) Activate the wiper – highlight “Clean Optics” on upper right 650 MDS screen, and enter to activate.
 - 9) Select **Optic T – Turbidity-6136** from the Calibrate menu, and then **2- 2-point**
 - 10) Input the value 0 NTU at the prompt and press **Enter**. The screen will display real-time readings that will allow determination of when the readings have stabilized.
 - 11) Verify that there are no air bubbles on the probe face. Wait at least 60 second after a wipe cycle before accepting a calibration point.
 - 12) After stabilization is complete (readings have not changed significantly in 30 seconds) **record the Pre-Calibration values of Turbidity; Temp (°C); & Time**. Then press **Enter** to “confirm” the 1st calibration.
 - 13) The upper right screen will indicate that the calibration has been accepted and the 0 NTU calibrated value of the turbidity sensor and all other sensors will appear on the screen. **Record the Post-Calibration values of Turbidity; Temp (°C); & Time**. Press **Enter** to continue.
 - 14) Dry the sonde carefully and hang it without touching the sensors to anything. Pour the DI out of the long (turbidity) calibration cup and shake the cup as dry as possible.

- 15) Use the 2nd turbidity solution that was used for the last turbidity calibration (to save on calibration solution costs), which has been stored in the plastic bottle, labeled “Used Turbidity Standard” (with a piece of tape on it showing the NTU of the used standard) in the calibration cupboard. Pour the used turbidity solution into the calibration cup, being sure to invert the used turbidity standard several times in its storage bottle to gently mix it well without creating bubbles before pouring it into the cup.
- 16) Place the calibration cup on the sonde to rinse the sensors with the used standard. Then remove the sonde carefully, shake off excess standard and hang the sonde being careful not to touch any of the sensors to anything.
- 17) Discard the used turbidity solution down the sink with plenty of running water and shake the calibration cup as dry as possible.
- 18) Place **225 ml** of the second turbidity standard (either Hach brand StableCal 100 NTU Standard or a diluted lower NTU standard) into the calibration cup provided with the sonde (marked on the cup), being sure to invert the turbidity standard several times gently to mix it well without creating bubbles before pouring it into the cup.
- 19) Immerse the sonde in the turbidity standard, setting the sonde on top of the calibration cup...do not engage the threads.
- 20) Activate the wiper – highlight “Clean Optics” on upper right 650 MDS screen, and enter to activate.
- 21) Input the correct turbidity value in NTU (100 or diluted NTU) and press **Enter**. The screen will display real-time readings that will allow determination of when the readings have stabilized.
- 22) Verify that there are no air bubbles on the probe face. Wait at least 60 second after a wipe cycle before accepting a calibration point.
- 23) After stabilization is complete (readings have not changed significantly in 30 seconds) **record the Pre-Calibration values of Turbidity; Temp (°C); & Time**. Then press **Enter** to “confirm” the 2nd calibration.
- 24) The upper right screen will indicate that the calibration has been accepted and the 2nd NTU calibrated value of the turbidity sensor and all other sensors will appear on the screen. **Record the 2nd Post-Calibration values of Turbidity; Temp (°C); & Time**. Press **Enter** to return to the Calibrate menu.
- 25) Thoroughly rinse and dry the sonde carefully and hang it without touching the sensors to anything.
- 26) Pour the used turbidity standard out of the calibration cup into the plastic bottle marked “Used Turbidity Standard” and place a piece of tape on it showing the NTU of the used standard. Save this for the turbidity check after sampling. Thoroughly rinse the calibration cup in tap water, then in DI water and shake the cup as dry as possible.
- 27) Remove the wiper from the Sonde. To save battery life disable the Sonde wiping mechanism (Sonde Menu – Advanced – Sensor – Wipes=0)

Warning: Do not use the Sonde if the wipers do not wipe properly in Step 4, above, notify ES.

III. Conductivity Calibration (Refer to CONDUCTIVITY, pages 2-40 to 2-41 in the YSI Manual)

- 1) Rinse the YSI in bucket of tap water, then in cup of DI, shake off excess water and dry the outside of the sonde probes with a clean paper towel, being very careful of the probes because the probe guard is not attached and they are very vulnerable to damage. Hang on cupboard door knob with out touching the sonde tip to anything.
- 2)

Record Pre-Calibration Conductivity Calibration Constant:

 Sonde menu→Advanced→Cal constants→Record Cond.:
- 3) Using the old conductivity calibration standard, which has been stored in the plastic bottle, labeled “Conductivity Calibration” in the calibration cupboard, rinse the calibration cup and the sonde in the old standard three times. Then shake the excess old standard off the sonde and re-hang it on the knob being careful not to touch the tip on anything.
- 4) Discard the old conductivity standard down the drain with lots of running water. Shake excess old standard out of the calibration cup vigorously. **DO NOT RE-RINSE THE CUP!**
- 5) Using new conductivity calibration standard from the cupboard, shake the standard, and then pour new clean standard into the sonde calibration cup to the **150 ml** fill line marked on the cup.
You need to accomplish this calibration with the sonde in an upside down position (cable end down) to save calibration solution (see Table 1B in the YSI 6-Seires manual, page 2-26).
- 6) Carefully immerse the probe end of the sonde into the solution. Make sure that the rubber gasket is in place on the sonde, and then screw the calibration cup onto the 6820V2 sonde snugly. **Invert the calibration cup so that the cable is down.** Gently rotate and/or move the sonde to remove any bubbles from the conductivity cell. The probe must be completely immersed past its vent hole (check this through the cup side to make sure). Place the sonde in the inverted position into the 3-prong clamp holder on the support stand and tighten the clamp screws to hold it securely. **NOTE:** If you must do the conductivity calibration in the upright position (cable up) then you must fill the calibration cup to the 320 ml line (see Table 1B in the YSI manual, page 2-26).
- 7) Allow at least one minute for temperature equilibration before proceeding.
- 8) From the **Calibrate** menu, select **Conductivity**, select **1-** to access the specific conductance calibration procedure. Enter the calibration value of the standard you are using (**10.0 mS/cm at 25° C**) and press **Enter**. The current values of all sensors will appear on the screen and will change with time as they stabilize.
- 9) Watch the conductivity and temperature related values, and when they have shown no significant change for approximately 30 seconds, **record the Pre-Calibration values of SpCond (1st mS/cm); Cond (2nd mS/cm); Temp (°C); & Time** (*Note: the Conductivity Calibration Constant should already have been recorded...see # 2, above).
- 10) If the conductivity values have remained stable (have not changed significantly in approximately 30 seconds), press **Enter**. If the sonde reports “Out of Range”, investigate the cause! Never override a calibration error message without knowing the cause. Typical causes for the error message are, incorrect entries (entering 1000 µS/cm instead of 1.0 mS/cm), low solution level, air bubbles in the probes cell, calibrating in conductivity instead of in sp/cond, and bad standard.
- 11) The upper right screen will indicate that the calibration has been accepted and the calibrated value of the conductivity sensor and all other sensors will appear on the screen. **Record the Post-Calibration values of SpCond (1st mS/cm); Cond (2nd mS/cm); Temp (°C); & Time.**
- 12) Press **Enter** to return to the calibration menu. Press **Esc.** several times to page back to the Sonde menu.

- 13) Record the Post-Calibration Conductivity Calibration Constant: **Sonde menu**→**Advanced**→**Cal constants**→**Record Cond**: Check to make sure that this number is within the acceptable range of 4.55 to 5.45. If this number is out of the acceptable range, notify Environmental Specialist.
- 14) Remove the sonde from the calibration solution, rinse the sonde in bucket of tap water, then in cup of DI, shake off excess water and dry the outside of the sonde with a clean paper towel. Hang on cupboard door knob with out touching the sonde sensors to anything.
- 15) Pour the used calibration standard into the plastic bottle, labeled "Conductivity Calibration". Place Parafilm over the used solution in the calibration bottle to save it for the post-sampling calibration check and for rinsing the sonde prior to the next conductivity calibration.

IV. **pH Calibration** (Refer to pH 2-POINT, page 2-43 in the YSI Manual)

- Rinse the YSI in bucket of tap water, then in cup of DI, shake off excess water and dry the outside of the sonde with a clean paper towel. Hang on cupboard door knob without touching the sonde sensors to anything. Be very careful because the probe guard is not attached the sensors are vulnerable and fragile.
- Rinse the extended calibration cup in tap water 3-times, then with DI. Shake dry.

A. Buffer X (7.00, 10.00, or 4.00) pH Calibration

NOTE: Always do the **Buffer 7.00 pH calibration first**, then the **Buffer 10.00 pH calibration second**, and, if necessary (during groundwater sampling events only), do the **Buffer 4.00 pH calibration last**.

- 1) Using the old Buffer X (7, 10, or 4) pH calibration standard, which has been stored in the plastic bottle, labeled "pH Calibration, pH = X" in the calibration cupboard, rinse the calibration cup and the sonde in the old standard. Then shake the excess old standard off the sonde and re-hang it on the knob being careful not to touch the sensors on anything.
- 2) Discard the old pH standard down the drain with lots of running water. Shake excess old standard out of the calibration cup vigorously. **DO NOT RINSE THE CUP!**
- 3) Using new Buffer Solution pH X (7, 10, or 4) calibration standard from the cupboard, shake the solution, and then pour the new solution into the short (standard) calibration cup to the **175 ml** fill line marked on the cup (see Table 1B in the YSI 6-Series manual, page 2-26). **You need to accomplish this calibration with the sonde in an inverted position (cable end down)** to save calibration solution
- 4) Carefully immerse the probe end of the sonde into the solution. Make sure that the rubber gasket is in place on the sonde, and then screw the calibration cup onto the 6820V2 sonde snugly. **Invert the calibration cup so that the cable is down.** rotate and/or move the sonde to remove any bubbles from the pH cell. The pH sensor must be completely immersed in calibration solution (check this through the cup side to make sure). Place the sonde in the inverted position into the 3-prong clamp holder on the support stand and tighten the clamp screws to hold it securely.
NOTE: If you must do the pH calibration in the upright position (cable up) then you must fill the calibration cup to the 240 ml line (see Table 1B in the YSI 6-Series manual, page 2-26).
- 5) Allow at least one minute for temperature equilibration before proceeding.
- 6) From the **Calibrate** menu, select **ISE1 pH**, select **either 2- 2-Point** (for normal monthly surface water measurements) or **3- 3-Point** (for sampling groundwater) to access the specific pH calibration procedure. Enter the calibration value of the pH standard you are using After entering the correct pH value of the buffer, press **Enter**. The current values of all sensors will appear on the screen and will change with time as they stabilize in the solution.
NOTE: The actual pH value of all buffers is somewhat variable with temperature and the correct value from the bottle label for your calibration temperature should be entered for maximum accuracy. **For example the pH of pH 7 Buffer is 7.00 at 25°C, but 7.02 at 20°C.**
- 7) Watch the pH and temperature related values, and when they stabilize (have shown no significant change for approximately 30 seconds), **record the Pre-Calibration values of pH; pH mV; Temp (°C); & Time.**
- 8) If the pH values have stabilized such that they do not change significantly in approximately 30 seconds, press **Enter**.

- 9) The upper right screen will indicate that the calibration has been accepted and the Buffer X (7, 10, or 4) calibrated value of the pH sensor and all other sensors will appear on the screen. **Record the Post-Calibration values of pH; pH mV; Temp (°C); & Time.** Press **Enter** to continue.
- 10) *Remove the sonde from the calibration solution, rinse the sonde in the bucket of tap water, then in the cup of DI, shake off excess water and dry the outside of the sonde with a clean paper towel. Hang on cupboard door knob with out touching the sonde sensors to anything.*
- 11) *Pour the used calibration standard into the plastic bottle, labeled “pH X (7.00, 10.00, or 4.00) Calibration”. Place Parafilm over the used Buffer X solution in the bottle to save it for the post-sampling pH calibration check and for rinsing the sonde prior to the next pH calibration event. Rinse the calibration cup in tap water 2-3 times, then in DI. Shake to dry as much as possible.*
- 12) *Check to see that the Buffer X pH mV is within the acceptable range. The acceptable pH mV range for Buffer 7.00 is 0 ± 50 . If the calibrated pH mV value falls outside of this range, notify ES.*
- 13) *Repeat steps 1-12 with X = Buffer 10.00 pH calibration solution. Check to see that the buffer 10 pH mV is within the acceptable range of -180 ± 50 mV (-130 mV to -230 mV). If the buffer 10 pH mV value falls outside of this range, notify ES.*
- 14) **Determine the slope of the sensor for the buffer 7 & buffer 10 mV values and check to see if it is within the acceptable range of 165 to 180.** Determine the slope of the sensor by determining the difference between the two calibration points that were used. For example, if you recorded a -3 mV for buffer 7 and a -177 mV for buffer 10, then the slope would be $174 [-3 - (-177) = -3 + 177 = 174]$. Enter the slope on the calibration sheet. The acceptable range for the slope is 165 to 180. Once the slope drops to below 160, the sensor should be taken out of service.
- 15) *If a 3-Point pH calibration is necessary (when calibrating for groundwater quality measurements during a groundwater quality sampling event), repeat steps 1-12 with X = Buffer 4.00 pH calibration solution. Check to see that the buffer 4 pH mV is within the acceptable range of $+180 \pm 50$ mV ($+130$ mV to $+230$ mV). If the buffer 4 pH mV value falls outside of this range, notify ES..*
- 16) **Determine the slope of the sensor for the buffer 7 & buffer 4 mV values and check to see if it is within the acceptable range of 165 to 180.** Determine the slope of the sensor by determining the difference between the two calibration points that were used. For example, if you recorded a -3 mV for buffer 7 and a +177 mV for buffer 4, then the slope would be $180 [177 - (-3) = 177 + 3 = 180]$. Enter the slope on the calibration sheet. The acceptable range for the slope is 165 to 180. Once the slope drops to below 160, the sensor should be taken out of service.
- 17) **Warning:** Do not use a probe that has given any “Calibration Error” or “Out of Range” warning. If the calibrated Buffer 10 or 4 pH mV value falls outside of this range, notify ES.

V. Dissolved Oxygen (DO) Calibration (Refer to ROX OPTICAL DISSOLVED OXYGEN, pages 2-42 in YSI Manual)

- *NOTE: Field DO calibrations should be avoided!*
- The DO wiper must be removed when the sensor is exposed to the Hach Company turbidity standards or it will be fouled.
- The ROX sensor **MUST remain hydrated at all times!**

1 Point “Saturated Air” Calibration Method:

- 1) Dry the temperature sensor and the ROX membrane to remove any water droplets.
- 2) Put about 1/8 inch of water in the bottom of the short (standard) YSI 6820V2 calibration cup (or 1 inch of water in the long “turbidity” cal cup).
- 3) Place the sonde sensors into the calibration cup, making sure that the DO and temperature probes are not immersed in the water and they remain dry.
- 4) Screw the calibration cup on very loosely, making sure to engage only one half thread so that the DO probe is vented to the atmosphere. Do not fully thread the calibration cup onto the end cap!
- 5) Turn on the YSI 6820V2 and record Pre-Calibration ODO gain: **Sonde menu→Advanced→Cal constants→ Record ODO gain:**
- 6) Allow the sonde to sit in the calibration cup for 15-minutes to ensure that the air in the calibration cup is 100% water saturated and that the temperature stable.
- 7) While waiting for equilibration, obtain and record the “True Barometric Pressure” reading:
 - a) Read and record the handheld barometer pressure reading, which is reported in inches of Hg by the barometer (handheld barometer is kept in the YSI 6820V2 case). (Compare this reading to the most recent National Weather Service barometric pressure for Bishop (from the web).)
 - b) Go to the Excel spreadsheet “OVIWC Calibrations Form” and enter in the handheld barometric pressure reading in the row for the appropriate town (Bishop for a lab calibration), and the spreadsheet calculations will yield the true barometric reading for the DO calibration...or do the following calculations with a calculator:
 - i) **Multiply the handheld barometric pressure reading (in Hg) by 25.4;** this converts the barometric pressure to mm Hg.
 - ii) **Subtract 105 (for Bishop) from the product obtained in i), above, and record as the True Barometric Pressure.** Please note: The number that you subtract in this step depends on the elevation of the point where you are performing the calibration. The number given above is for the office lab in Bishop. The formula for converting weather service or handheld BP to True BP is: (Weather Service or Handheld BP * 25.4) – [2.5*(Local Altitude in feet above sea level/100)]
 - c) **Record the True Barometric Pressure reading** (converted from the handheld barometric pressure reading) **and the time** the reading was taken.
- 8) From the **Calibrate** menu select Optic C – Dissolved Ox: then ODOsat %; and then 1 Point to begin the DO calibration procedure. (Note: Calibration of the dissolved oxygen sensor in the DO% procedure also results in calibration of the DO mg/L mode and vice versa).
- 9) At the prompt enter the current “True Barometric Pressure” in mm Hg with elevation corrections removed as measured and converted in step #7), above.
- 10) Press **Enter** and the current values of all enabled sensors will appear on the screen and change with time as they stabilize. Observe the readings under ODO %. When they show no significant change for approximately 30 seconds record the **Pre-Calibration values of ODO %, ODO mg/L, Temp (°C); & Time** (NOTE: ODO Gain should already have been recorded in step # 4), above.

- 11) When the ODO % values have not changed significantly within 30 seconds press **Enter** to “confirm” the calibration.
- 12) The upper right screen will indicate that the calibration has been accepted and the calibrated value of the ODO sensor and all other sensors will appear on the screen. **Record the Post-Calibration values of ODO %; ODO mg/L; Temp (°C); & Time.**
- 13) Press **Enter** to return to the **Calibrate** menu.
- 14) Record Post-Calibration ODO Gain: Sonde menu→**Advanced**→**Cal constants**→**Record ODO Gain.**
(Note: ODO gain is ideally 1.0, but is acceptable if between 0.75 – 1.25) (??or 0.7 to 1.4). If it is out of this range, notify ES.
- 15) Thoroughly rinse and dry the sonde carefully and hang it without touching the sensors to anything.

VI. Prepare the YSI 6820V2 for transport to the field

- 1) Moisten the sponge in the calibration cup with tap water or pour tap water into the bottom of the cup.
- 2) Place the calibration cup over the end of the sonde and tighten it, being sure that there is moisture in the cup. **The ODO sensor and the pH sensor must never be dried out.**
- 3) Be sure the probe guard is taken into the field with the sonde to be installed on the sonde before it is placed in the water.

Post-Sampling Calibration Check & Sonde Storage Procedures

YSI 6820V2 Sonde

I. Calibration Check Preparation

- 1) Fill bucket full of water
- 2) Fill cup full of DI water

II. Conductivity Check (a reading only...not done in calibration mode. To save calibration solution costs, this check is to be done using the calibration solution that was used to calibrate the instrument, knowing that the used solutions will likely cause some variation in readings.)

- 1) Turn on YSI 6820V2. Select **Sonde run**.
- 2) Remove the sonde from the storage/calibration cup. Rinse the YSI in bucket of tap water, then in cup of DI; shake off excess water being very careful not to touch the exposed sensors to anything. They are very vulnerable to injury because they have no guard protecting them. Dry the outside of the sonde with a clean paper towel. Hang on cupboard door knob with out touching the sonde sensors to anything.
- 3) Rinse the calibration cup in tap water then in DI, and then shake it as dry as possible.
- 4) Use the conductivity calibration standard that was used for the last conductivity calibration (to save on calibration solution costs), which has been stored in the plastic bottle, labeled “Conductivity Calibration” in the calibration cupboard. Pour the used solution into the calibration cup.
- 5) Carefully immerse the probe end of the sonde into the solution and engage the cups threads, screwing it onto the sonde snugly. Invert the sonde upside down (so that the cable is up) and hold it securely until the calibration check is completed. Gently rotate and/or move the sonde to remove any bubbles from the conductivity cell. The probe must be completely immersed past its vent hole in this

inverted position.

- 6) Allow at least one minute for temperature equilibration before proceeding.
- 7) Watch the conductivity and temperature related values, and when they have stabilized (have shown no significant change for approximately 30 seconds), **record the Post-Sampling Check values of SpCond (1st mS/cm); Cond (2nd mS/cm); Temp (°C); & Time** (*Note: Since it is unchanged since the calibration recorded on this sheet, Cond Gain should already have been recorded).
- 8) Are the two conductivity values within $\pm 10\%$ of the Post-Calibration values previously recorded? If not, notify the Environmental Specialist.
- 9) Remove the sonde from the calibration solution, rinse the sonde in bucket of tap water, then in cup of DI, shake off excess water and dry the outside of the sonde with a clean paper towel. Hang on cupboard door knob without touching the sonde sensors to anything.
- 10) Pour the used calibration solution back into the plastic bottle; place Parafilm over it to save it for rinsing the sonde prior to the next conductivity calibration.
- 11) Rinse the calibration cup in tap water then in DI, and then shake it as dry as possible.

III. pH Check (a reading only...not done in calibration mode)

- 1) Make sure the YSI 6820V2 is on and **Sonde run** has been selected.
- 2) Make sure the sonde and calibration cup have been rinsed, as described in II-2) and II-10, above.
- 3) Use the Buffer 7 pH solution that was used for the last pH calibration (to save on calibration solution costs), which has been stored in the plastic bottle, labeled "pH Calibration, pH = 7.00" in the calibration cupboard. Pour the used pH buffer into the calibration cup.
- 4) Carefully immerse the probe end of the sonde into the solution and engage the cups threads, screwing it onto the sonde snugly. Invert the sonde upside down (so that the cable is up) and hold it securely until the calibration check is completed. Gently rotate and/or move the sonde to remove any bubbles from the pH sensor. The pH probe must be completely immersed in calibration solution in this inverted position.
- 5) Allow at least one minute for temperature equilibration before proceeding.
- 6) Watch the pH and temperature related values, and when they begin to stabilize (have shown no significant change for approximately 30 seconds), **record the Post-Sampling Check values of pH; pH mV; Temp (°C); & Time**.
- 7) Is the pH value within $\pm 10\%$ of the Post-Calibration values previously recorded? If not, notify the Environmental Specialist.
- 8) Remove the sonde from the calibration solution, rinse the sonde in bucket of tap water, then in cup of DI, shake off excess water and dry the outside of the sonde with a clean paper towel. Hang on cupboard door knob without touching the sonde sensors to anything.
- 9) Pour the used calibration solution back into the plastic bottle; place Parafilm over it to save it for rinsing the sonde prior to the next Buffer 7 pH calibration.
- 10) Rinse the calibration cup in tap water then in DI, and then shake it as dry as possible.
- 11) Repeat the steps above using the used Buffer 10 solution (and, if you did a 3-point calibration last, using the used Buffer 4 solution).

IV. Turbidity Check (a reading only...not done in calibration mode)

- 1) Make sure the YSI 6820V2 is on and **Sonde run** has been selected.
- 2) Make sure the sonde and calibration cup have been rinsed, as described in II-2) and II-10, above. Be very careful because without the probe guard attached the sensors are vulnerable and fragile.
- 3) Place 225 ml of distilled, deionized water (DI water) into the calibration cup pouring down the side of the cup to decrease formation of bubbles.

- 4) *Immerse the sonde in the water setting the sonde on the calibration cup with no threads engaged.*
- 5) *Check to make sure there are no bubbles on or near the optical turbidity probe window.*
- 6) *Watch the turbidity values, and when they begin to stabilize (have shown no significant change for approximately 30 seconds), **record the Post-Sampling Check values of Turbidity; Temp (°C); & Time.***
- 7) *Remove the calibration cup from the sonde. Dry the sonde carefully and hang it without touching the sensors to anything.*
- 8) *Pour the DI out of the calibration cup and shake the cup as dry as possible.*
- 9) *Use the 2nd turbidity solution that was used for the last turbidity calibration (to save on calibration solution costs), which has been stored in the plastic bottle, labeled "Used Turbidity Standard" (with a piece of tape on it showing the NTU of the used standard) in the calibration cupboard. Pour the used turbidity solution into the calibration cup, being sure to invert the used turbidity standard several times in its storage bottle to gently mix it well without creating bubbles before pouring it into the cup.*
- 10) *Immerse the sonde in the standard setting the sonde on the cup with no threads engaged. Check for bubbles on the optical turbidity probe window.*
- 11) *Watch the turbidity values, and when they begin to stabilize (have shown no significant change for approximately 30 seconds), **record the 2nd Post-Sampling Check values of Turbidity; Temp (°C); & Time.***
- 12) *Remove the calibration cup from the sonde. Thoroughly and carefully rinse the sonde in tap water and then DI water and dry the sonde and hang it without touching the sensors to anything.*
- 13) *Pour the used turbidity standard out of the calibration cup into the plastic bottle marked "Used Turbidity Standard" and place a piece of tape on it showing the NTU of the used standard. Cap the sample bottle and save the used standard to rinse the sensor before the next 2nd turbidity calibration.*
- 14) *Thoroughly rinse the calibration cup in tap water, then in DI water and shake the cup as dry as possible.*

V. Short Term YSI 6820V2 Storage Procedures (less than 3 weeks)

- 1) *Dampen the sponge in the small ODO sensor cap and carefully place it over the end of the Optical Dissolved Oxygen sensor (YSI 6250 ROX in the sonde's optical C port).*
- 2) *Place 1/8 inch of tap water into the bottom of the calibration cup and screw the cup firmly onto the sonde. **It is very important that the Dissolved Oxygen probe and the pH probe never be dried.***
- 3) *Store the sonde in its hard sided case.*

VI. YSI 6820V2 Long-Term (>3 weeks) Storage Procedures

- 1) *Remove the pH probe from the Sonde using the tools provided by the manufacturer.*
- 2) **Use the plug provided by the manufacturer to securely cover the pH port in the Sonde** before storing the instrument.
- 3) *Place pH Buffer 4 solution in the small pH probe storage bottle provided by the manufacture. Place the probe storage bottle cap and o-ring on the pH probe, and immerse the probe in the Buffer 4 solution in the storage bottle before tightening the cap onto the storage bottle securely. Store the pH probe with the bottle upright in a ziplock baggie in the cupboard above the lab sink. Extra Buffer 4 solution is stored in the right-hand calibration cupboard. Storage solution can be formulated using KCl crystals, which are stored in the right-hand calibration cupboard. The formula for making the 2 mole KCl solution is: 74.6 g of KCl crystals dissolved in 500 ml reagent grade distilled, deionized water.*
- 4) *Check to make sure the pH port is closed securely before placing the Sonde in the field calibration cup with ½ inch tap water in the bottom of the cup. Screw the cup with water onto the Sonde firmly.*
- 5) *Store the sonde in the office laboratory sink, held upright and stabilized by the clamp screws.*

Owens Valley Indian Water Commission (revised 1/28/10)

YSI 6820V2 Calibration Record

Calibration Date: _____ Technician: _____
 Battery Voltage Gauge (approximate reading): _____

Temperature Verification: (comparison of temperatures - YSI probe to calibration thermometer SN:)

Date/Time	YSI Temperature Reading:	Thermometer SN K20571 Temperature Reading:

Optical Turbidity Calibration: (Try to complete calibration using Hach standard in 2 minutes or less to avoid settling)

	NTUs	Date	(PST) Time	(NTU) Turbidity	(deg C) Temp	
Pre-Calibration	0 (Distilled Water)					NOTE: One standard must be 0 NTU and this standard must be calibrated first. - Clean the turbidity sensor by rinsing the optical sensor with DI & drying it <u>with the lint free cloth.</u> - Confirm that the output of the probe increases when you place your finger <u>in front of</u> the optics. - Calibration of optical sensors <u>must be done in</u> an upright position!!
Post-Calibration						
Post- Sampling Check						
Pre-Calibration	(fill in NTU value of 2nd standard)					- Calibration of optical sensors <u>must be done in</u> an upright position!!
Post-Calibration						
Post- Sampling Check						

Post-sampling check to be done using used 2nd standard, just to see if reading is good

Conductivity Calibration: (Conductivity Standard = 10 mS/cm)

	Date	(PST) Time	(mS/cm) SpCond	(mS/cm) Cond	(deg C) Temp	* Conductivity Calibration Constant
Pre-Calibration						
Post-Calibration						
Post- Sampling Check						Using used standard, just to see if reading is good

(* Conductivity Calibration Constant - Ideal Reading = 5.0 +/- 0.45; Acceptable Range = 4.55 - 5.45)

pH Calibration: (Two or Three-Point Calibrations with Buffer 7, Buffer 10, and Buffer 4 Solutions)

	Buffer	Date	(PST) Time	pH	(mV) pH mV	(deg C) Temp	pH mV Acceptable Range
Pre-Calibration	7.00						Buffer 7 = 0 +/- 50 mV
Post-Calibration							
Post- Sampling Check						(re-used std.)	
Pre-Calibration	10.00						Buffer 10 -180 +/- 50 mV Slope = difference between 7's & 10's mV OK Range = 165 - 180 mV _____
Post-Calibration							
Post- Sampling Check						(re-used std.)	
Pre-Calibration	4.00						Buffer 4 +180 +/- 50 mV Slope = difference between 7's & 4's mV OK Range = 165 - 180 mV _____
Post-Calibration							
Post- Sampling Check						(re-used std.)	

Dissolved Oxygen (ODO) Probe Calibration: Calibrate to partial pressure of air - once per day in office calibration is sufficient

Date/Time: _____ Conversion formulas from Handheld BP to True BP:

NWS or Handheld Barimetric Pressure (BP) (in. Hg): _____ (Handheld BP) x 25.4 - [2.5 x (Local Altitude/100)] = True BP

True Barimetric Pressure (to be entered in calibration) (mm Hg): _____ Hand held barometric pressure (in. Hg) X 25.4 = (mm Hg)

	Date	(PST) Time	(%) ODO	(mg/L) ODO	(deg C) Temp	*ODO Gain
Pre-Calibration						(*ODO Gain - Ideal reading = 1.0; Acceptable Range = 0.75 to 1.25) * If out of range, recalibrate; if you can't get it in range, probe needs maintenance.
Post-Calibration						

Appendix F

List of Abbreviations and Acronyms

ASTM	American Society for Testing and Materials
BLM	Bureau of Land Management
CalTrout	California Trout
CEDEN	California Environmental Data Exchange Network
Commission	Owens Valley Indian Water Commission
CDFG	California Department of Fish and Game
CDFG-WPCL	California Department of Fish and Game – Water Pollution Control Laboratory
DM	Data Manager (California Trout)
EPA	Environmental Protection Agency
FM	Field Manager (California Trout)
FT	Field Technician (California Trout)
GIS	Geographic Information Systems
GM	Grant Manager (State Water Resources Control Board)
Hg	Mercury
I-M IRWMP	Inyo-Mono Integrated Regional Water Management Program
LADWP	Los Angeles Department of Water and Power
IRWMP	Integrated Regional Water Management Program
LRWQCB	Lahontan Regional Water Quality Control Board
MC	Mammoth Creek
MCWD	Mammoth Community Water District
MDL	Method Detection Limit
MLB-NPS	Mammoth Lakes Basin Nonpoint Source
MLML-MPSL	Moss Landing Marine Laboratory – Marine Pollution Studies Laboratory
MODIS	Meteorological Assimilation Data Ingest System
Mn	Manganese
NOAA	National Oceanic and Atmospheric Administration
NPS	Nonpoint Source
PD	Project Director (California Trout)
OVIWC	Owens Valley Indian Water Commission
QA	Quality Assurance
QA/QC	Quality Assurance/Quality Control
QC	Quality Control
QC (method)	QuikChem Method by Lachat Instruments
QAM	Quality Assurance Manager (State Water Resources Control Board)
QAPP	Quality Assurance Project Plan
SCB	sample collection bottle
SM	Standard Methods for the Examination of Water and Wastewater, 20th edition
SOP	Standard Operating Procedure
State Board	State Water Resources Control Board
SWAMP	Surface Water Ambient Monitoring Program
SWRCB	State Water Resources Control Board

TAC	Technical Advisory Committee
TMDL	Total Maximum Daily Loads
TDS	Total Dissolved Solids
TSS	Total Suspended Solids
TOML	Town of Mammoth Lakes
U.S. EPA	United States Environmental Protection Agency
USFS	United States Forest Service
WQS	Water Quality Specialist (California Trout)