AMBIENT LAKES MONITORING PROGRAM (ALMP)

QUALITY ASSURANCE PROJECT PLAN

PROGRAM ADMINISTERED AND PLAN PREPARED BY:

NORTH CAROLINA DEPARTMENT OF

ENVIRONMENT & NATURAL RESOURCES

DIVISION OF WATER RESOURCES

ENVIRONMENTAL SCIENCES SECTION

INTENSIVE SURVEY BRANCH

Version 2.0

EPA Approved
March 28, 2014

Abbreviations

AGPT	Algal Growth Potential Tests
ALMP	Ambient Lakes Monitoring Program
APHA	American Public Health Association
AU	Assessment Unit
BAR	Basin Assessment Report
BMP	Best Management Practices
CHL	Chlorophyll *a*
CWA	Clean Water Act
DENR	Department of the Environment and Natural Resources
DO	Dissolved Oxygen
DWR	Division of Water Resources
EPA	Environmental Protection Agency
ESS	Environmental Sciences Section
GIS	Geographic Information System
ISB	Intensive Survey Branch
MDL	Method Detection Limit
NCTSI	North Carolina Trophic State Index
NH_3	Ammonia
NO_x	Nitrite + Nitrate
NPDES	National Pollutant Discharge Elimination System
NTU	Nephelometric Turbidity Units
PQL	Practical Quantitation Limit
QA	Quality Assurance
QAM	Quality Assurance Manual
QC	Quality Control
SAR	Sample Anomaly Report
SCUR	Sample Condition Upon Receipt
SD	Secchi Depth
SOP	Standard Operating Procedures
TKN	Total Kjeldahl Nitrogen
TMDL	Total Maximum Daily Load
TON	Total Organic Nitrogen
TP	Total Phosphorus
TSS	Total Suspended Solids
USGS	United States Geological Survey

REVISION LOG

ALMP QAPP

Date Edited	Editor	Version Edited	Section Edited	Changes/updates
12/19/2013	Jason Green	Ver 1.1	Entire Document	Updated Division of Water Quality (DWQ) to Division of Water Resources (DWR). Updated Intensive Survey Unit (ISU) to Intensive Survey Branch (ISB) Updated website hyperlinks Updated all headers, footers, and hyperlinks. Updated document date and version to January 2014 and Version 2.0
12/19/2013	Jason Green	Ver 1.1	A1. Signature and Approval Sheet	Updated personnel and organizational names.
12/19/2013	Jason Green	Ver 1.1	A2. Distribution List	Updated personnel and organizational names.
12/19/2013	Jason Green	Ver 1.1	A3. Project Organization	Updated staff duties & personnel names
12/19/2013	Jason Green	Ver 1.1	A4. Project Organization	Updated org chart, project contacts and staff information
12/19/2013	Jason Green	Ver 1.1	B3. Sample Handling & Custody	Updated Figure B3.1
12/19/2013	Jason Green	Ver 1.1	Attachment 1	Updated Intensive Survey Branch Standard Operating Procedures to Ver 2.1
12/19/2013	Joanna Gmyr	Ver 1.1	Attachment 5	Removed attachment 5 (Calibration Sheet) & changed in-text references to (ISB SOP, Figure 10)
12/19/2013	Joanna Gmyr	Ver 1.1	Attachment 6	Changed to Attachment 5 & updated in-text references
11/14/2011	Debra Owen	Ver. 1.0	Cover page & Footers	Updated document date & version number to November 2011 & Version 1.1
11/14/2011	Debra Owen	Ver. 1.0	Document hyperlinks	Updated links to web sites.
11/14/2011	Debra Owen	Ver. 1.0	Revision Log	Added a Revision Log.
11/14/2011	Debra Owen	Ver. 1.0	A3 Distribution List	Updated contact information.
11/14/2011	Debra Owen	Ver. 1.0	A4 Project Organization	Updated project contacts and field staff
11/14/2011	Debra Owen	Ver. 1.0	URL links	All Internet hyperlinks were checked and functioned correctly.
11/14/2011	Debra Owen	Ver. 1.0	Table A6.2 Basin Assessment Periods	Updated the river basin assessment schedule
11/14/2011	Debra Owen	Ver. 1.0	Table B2.1 Source of Equipment and Disposables	Updated conductivity standards
11/14/2011	Debra Owen	Ver. 1.0	B3. Sample Handling and Custody	Updated date format
11/14/2011	Debra Owen	Ver. 1.0	Table B6.1 Field Equipment Maintenance	Added inspection of probes for multiparameter meters
11/14/2011	Debra Owen	Ver. 1.0	References and Resources	Updated references.

REVISION LOG

ALMP QAPP

Date Edited	Editor	Version Edited	Section Edited	Changes/updates
11/14/2011	Debra Owen	Ver. 1.0	B7 Instrument Calibration & Frequency	Changed conductivity standard values to 500 and 1000 umhos/cm
11/14/2011	Debra Owen	Ver. 1.0	B9 Acquired Data (Non-Direct Measurements)	48-hour precipitation – added ACOE as source of rain data
11/14/2011	Debra Owen	Ver. 1.0	Figure B10.1 ALMP Data Flow	Changed ALMP Chemistry and Physical Database to ALMP Lakes Database
11/14/2011	Debra Owen	Ver. 1.0	Attachment 3	Updated NC Surface Water Quality Standards from 2003 to 2007 version
11/15/2011	Debra Owen	Ver. 1.0	Attachment 4	Update the NC DWR ALMP Station Information
11/16/2011	Debra Owen	Ver. 1.0	Table of Contents	Updated page numbers
11/16/2011	Debra Owen	Ver. 1.0	References to Laboratory QAP Document	Updated references to various sections of this document in the ALMP QAP
11/16/2011	Debra Owen	Ver. 1.0	References to ISB SOP – Nov.2011	Updated references to various sections of this document in the ALMP QAP
11/18/2011	Debra Owen	Ver. 1.0	B.6 Instrument/ Equipment Testing, Inspection and Maintenance	Updated the Field Equipment section to reflect items in the ISB SOP.
11/18/2011	Debra Owen	Ver.1.0	B10. Data Management	Updated figure numbers in first paragraphs.
11/23/2-11	Debra Owen	Ver.1.0	B.3. Sample Handling and Custody	Revised guidance for proper completion of sampling tags to indicate that chlorophyll a and fecal coliform bacteria sampling tags need sampling time per DWR laboratory guidance.
12/2/2011	Jason Green	Ver. 2.0	Entire Document	Update to new version number to encompass numerous revisions.

SECTION A: PROJECT MANAGEMENT

A1 Signature and Approval Sheet

APPROVED BY

D Reid 1/24/2014

Dianne Reid, Section Chief, ESS — Date

1/10/14

Jason Green, Intensive Survey Branch Supervisor — Date

1/10/14

Joanna Gmyr, ESS QA Coordinator — Date

1/10/14

Debra Owen, ALMP Coordinator — Date

Marion D. Hopkins 3/28/2014

EPA Region 4 Designated approving Official — Date

A2. Table of Contents

TABLES

FIGURES

A3. Distribution List

Primary Distribution:

Marion Hopkins, NC Monitoring, Grant Technical Officer, EPA, Region 4

Tom Reeder, Director, NC Division of Water Resources

Tom Fransen, Water Planning Section Chief

Dianne Reid, Environmental Sciences Section Chief

Jason Green, Intensive Survey Branch Supervisor

Joanna Gmyr, Environmental Sciences Section, Quality Assurance Coordinator

Debra Owen, Lakes Monitoring Program Coordinator and Data Management, Intensive Survey Branch

Kent Wiggins, Division of Water Resources Laboratory Section Chief

Dana Satterwhite, Laboratory Section QA/QC Officer

Kathy Stecker, Modeling & Assessment Branch Supervisor

Ian McMillian, Basin Planning Branch Supervisor

Landon Davidson, Water Quality Supervisor, Asheville Regional Office

Vacant, Water Quality Supervisor, Fayetteville Regional Office

Michael Parker, Water Quality Supervisor, Mooresville Regional Office

Danny Smith, Water Quality Supervisor, Raleigh Regional Office

David May, Water Quality Supervisor, Washington Regional Office

Jim Gregson, Water Quality Supervisor, Wilmington Regional Office

Corey Basinger, Water Quality Supervisor, Winston-Salem Regional Office

Courtesy Distribution:

Rich Gannon, Nonpoint Source Planning Branch Supervisor

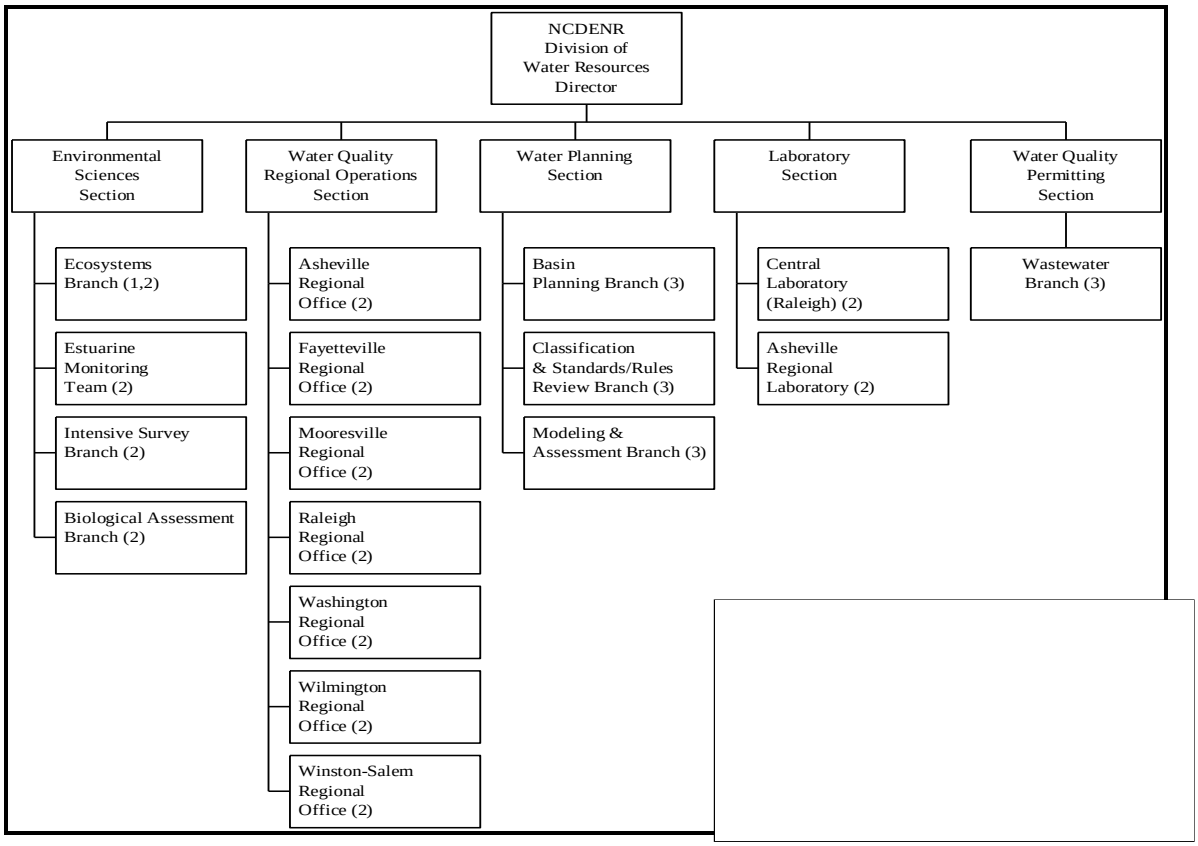
Matt Matthews, Water Quality Permitting Section Chief

Jeff Poupart, Wastewater Branch Supervisor

A4. Project Organization

The Ambient Lakes Monitoring Program (ALMP) is implemented within the North Carolina Department of Environment and Natural Resources (NCDENR) Division of Water Resources (DWR). An overview of DWR branches involved in the ALMP is provided in Figure A4.1. Detailed information on project contacts is provided below.

Figure A4.1: North Carolina Division of Water Resources Organizational Chart



Project Contacts

Project Manager

Jason Green

Supervisor, Intensive Survey Branch, ESS

- Supervises Ambient Lakes Monitoring Coordinator/Data Manager, QA Coordinator, and field staff.
- Responsible for ensuring that the ALMP is conducted in accordance with all relevant QAPPs and SOPs.
- Reviews and approves all reports, work plans, corrective actions, QAPP, and any other major work products and their revisions.
- Approves changes to program; ensures changes comply with DWR regulations and policies as well as data users' needs.
- Program development.
- Reports to Environmental Sciences Section Chief.

Project Coordinator and Data Manager

Debra Owen

ALMP Program Coordinator, Intensive Survey Branch, ESS

- Acts as liaison between program management, field staff, analytical laboratory, and data users.
- Coordinates logistics of program, such as maintaining sampling schedule, producing and distributing sample submission forms to field staff, maintaining station information database, providing certain supplies.
- Responds to issues raised by any program participant or outside party, identifies root causes and recommends response actions to the Project Manager.
- Communicates needed or suggested changes to ALMP to Project Manager for approval.
- Performs all aspects of data management, including tracking, compilation, review, data entry, identifying and correcting errors, and upload of data to the Lakes Database. Fulfills requests for raw data.
- Assists in training field staff.

ISB Equipment Manager

Joseph Smith

Environmental Senior Technician, Intensive Survey Branch, ESS

- Responsible for the general maintenance and repair of sampling equipment and meters used by the ALMP.

Computer Support

Donald Kean

Computer Consultant, Ecosystems Branch, ESS

- Provides technical support to Intensive Survey Branch staff for in-house databases.

Project QA Coordinator

Joanna W. Gmyr

ESS QA Coordinator, Ecosystems Branch, ESS

- Documents QA practices of ALMP.
- Maintains ALMP QAPP.
- Develops and recommends QA/QC improvements.

Data Generation (Measurements and Analyses)

Field staff: Perform all field activities including field measurements, observations, and sampling.

ISB Staff

- Debra Owen, (ALMP Coordinator)
- Jason Doby
- Harold Quidley
- Joseph Smith
- Mark Hale
- Jeff Deberardinis
- Staff of Winston-Salem Regional Office
- Staff of other Regional Offices

Laboratories: Perform all chemical, physical, and bacterial laboratory analyses.

DWR Laboratory Section

Kent Wiggins, Section Chief

Data End Users

Primary: Used to support DWR water pollution management programs

Ecosystems Branch staff

Biological Assessment Branch staff

Classifications & Standards/Rules Review Branch staff

Basin Planning Branch staff

Modeling and Assessment Branch staff

Intensive Survey Branch staff

Regional Office staff

U.S. EPA

A5. Problem Definition/Background

As part of funding agreements with the Environmental Protection Agency (EPA), North Carolina agrees to monitor the waters of the State and report findings to the EPA to support the goals of the Clean Water Act (CWA). The Federal Water Pollution Control Act Amendments of 1972, commonly referred to as the Clean Water Act (CWA), and subsequent amendments define the following as their objective:

> *"to restore and maintain the chemical, physical, and biological integrity of the Nation's waters, and, where attainable, to achieve a level of water quality that provides for the protection and propagation of fish, shellfish, and wildlife, and for recreation in and on the water".*

The Federal Clean Lakes Program, which was established as Section 314 of the Clean Water Act (CWA), enabled North Carolina in 1981 to receive federal funding to classify the trophic (or nutrient enrichment) status of the State's publicly owned freshwater lakes and to prioritize lakes for restoration. A sampling program was established in 1981 to survey the trophic condition of 65 lakes. Thirty-one of these lakes were sampled again in 1982. From this work, the North Carolina Trophic State Index (NCTSI) that the State has used for all subsequent trophic classifications was developed. The State has continued to monitor the significant lakes. The current Ambient Lake Monitoring Program (ALMP) consists of approximately 160 lakes statewide.

In addition to the development of North Carolina's lake monitoring program, major provisions of the CWA also led to the development of state-based water pollution management controls, which are based primarily on development and enforcement of numerical and narrative water quality standards. The current numerical standards and action levels are described in the NC Administrative Code (Chapter 2, Subchapter 2B), which is commonly referred to as the "Red Book". The full text of the Code is available online at http://portal.ncdenr.org/c/document_library/get_file?folderId=285750&name=DLFE-8513.pdf.

Since it is a project of indefinite duration, the ALMP is a valuable tool for identifying long-term spatial or temporal patterns in lakes across the state. Data produced by the ALMP support the activities of several different sections within the Division of Water Resources:

- Environmental Sciences Section
- Identification of long-term temporal or spatial patterns
- Determine current trophic status and identify potential changes in trophic state
- Identification of water quality standard violations present in lakes
- Provide background information for Intensive Survey Branch special studies, Biological Assessment Branch (BAB) monitoring and Ecosystems Branch special studies
- Analysis of lake data in support of §314 reporting requirements (305(b) report)
- Planning Section
- Biennial 303(d) and 305(b) reporting to EPA, including identification of lakes with impairment or degradation;
- Basinwide Water Quality Plan development;
- Identification of outstanding or unique lakes;
- Total Maximum Daily Load (TMDL) development;
- Background information for reclassification studies;
- Triennial review of water quality standards.

Lakes in North Carolina have been given a classification based on their intended use, which determines the level of protection required. Major classifications appropriate to lakes and their corresponding uses are shown in Table A5.1. Class C waters are protected to support the propagation and maintenance of aquatic life and incidental recreational uses. Class B waters are protected for full body contact

(organized swimming) and all Class C uses. The WS classifications II through IV are intended to protect source water and all Class C uses. The WS-I classification is to protect water supplies that are not filtered prior to use. There are no WS-I lakes in the ALMP.

Table A5.1: North Carolina Lake Classifications and Uses

Lake Classification	Protected Uses by Classification			
	Aquatic Life	Secondary Recreation	Primary Recreation	Water Supply
C	X	X		
B	X	X	X	
WS (I-V)	X	X		X

In addition to these major classifications, North Carolina also has supplemental classifications to protect for additional uses, such as trout survival and propagation, outstanding resource waters, future water supplies, and nutrient sensitive waters. Descriptions of the State's lakes classifications can be found on the Classification and Standards/Rules Review Branch's website: http://portal.ncdenr.org/web/wq/ps/csu/classifications. Classifications for individual lakes can also be obtained at this website.

Different uses are protected by varying combinations of legislatively mandated requirements for activities within a watershed such as:

- Number and type of allowable discharges;
- Stream buffers;
- Erosion and sediment controls;
- Agricultural best management practices (BMPs);
- Forestry BMPs;
- Transportation BMPs;
- Number and type of landfills;
- Number and types of dams/water resources projects.

In addition to these managerial controls, there are also corresponding numerical water quality standards and action levels which specify the chemical, physical, and microbial pathogen characteristics required to ensure that the water is of sufficient quality for the intended use.

Information on DWR's assessment protocol and current Integrated Report is available in on the Modeling and Assessment Branch's website at: http://portal.ncdenr.org/web/wq/ps/mtu/assessment.

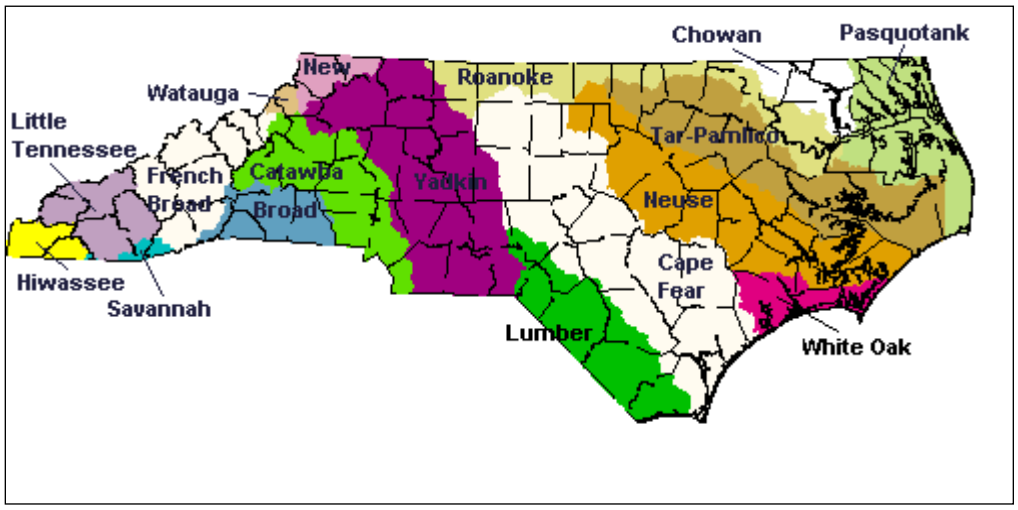
A6. Project/Task Description and Schedule

Overview

The ALMP consists of a relatively static network of stations located on lakes throughout the State, which provide site-specific, long-term water quality information. This network is based on sampling lakes that are publicly accessible, water supply lakes, or lakes that have been previously monitored by the Division of Water Resources (DWR). There are currently 422 lake monitoring stations established throughout the State's seventeen major river basins (Figure A6.1). These stations are typically monitored by boat.

Other Intensive Survey Branches studies differ from the ALMP in that they tend to be special studies targeted to many different types of waters. For the other studies, individual study plans are developed that include QAPP type information specific to the study.

Figure A6.1: North Carolina Major River Basins



Water Quality Indicators

The ALMP focuses primarily on chemical, physical, and biological characteristics of lakes. The indicators are primarily selected from those chemicals that have current NC water quality standards and that can be cost-effectively analyzed. Additional indicators are also included that may not have specific standards associated with them but are useful for interpretation of other measurements. Others are, of themselves, useful for identifying long-term trends.

A basic core suite of parameters is measured at all stations:

Temperature	Total Suspended Residue	pH
Specific Conductance	Secchi depth	Phytoplankton
Turbidity	Nutrients(nitrogen & phosphorus)	Chlorophyll *a*
Total Residue	Dissolved Oxygen	

Additional indicators may be included depending on lake-specific concerns such as classification and historical or suspected issues. Additional field observations, such as weather conditions, water color or appearance, presence of aquatic macrophyte, and/or visible indicators of algal blooms (i.e. floating mats, scums, or flecks) are also recorded at all site visits.

Measurement Methods Overview

Field measurements

Measurements made in the field include water temperature, specific conductance, stream flow severity, salinity, Secchi depth, DO, pH, air temperature, and wind velocity and direction. Field measurements are discrete and are made *in situ* by field staff at the time of the station visit. All field activities are performed in accordance with the ISB SOP (Attachment 1).

Analytical samples

Samples are submitted to the DWR Laboratory for analysis for turbidity, TSS, metals, nutrients, TS, chlorides, fecal coliform, and chlorophyll *a*. All sampling, preservation and handling, and analytical methods are performed in accordance with the ISB SOP (Attachment 1). Analytical methods are performed in accordance with the Laboratory Section's Quality Assurance Manual (QAM) (Attachment 2). The Laboratory's analytical methodologies are not managed as part of the ALMP and are beyond the scope of this QAPP.

In rare cases, it may be necessary for samples to be analyzed by other laboratories. These alternative labs must meet the minimum criteria inherent in the NC Laboratory Certification regulations and are required to provide reporting levels, analytical methods, accuracy, and precision equivalent to or better than those of the DWR laboratories. If a private laboratory is used, it must be certified by the NC Laboratory Certification Program to perform the analysis requested.

Sampling Schedule

The ALMP is geared towards the collection of long-term data and is therefore a continuous project. Stations are visited at least three times during May through September of a single year for each five-year river basin cycle for collection of field measurements and analytical samples. Lake sampling is conducted in each river basin the summer before the Basin Assessment Report (BAR) is due to Planning Section Staff.

The ALMP Coordinator prepares a draft-sampling schedule by March for the upcoming summer sampling trips. Each sampling trip consists of a lake or a group of lakes based on travel distance between lakes and number of sampling sites at each lake. All reasonable efforts are made to sample each lake per the sampling schedule; however, changes may be necessary due to weather conditions and/or staffing issues.

Data Management

The ALMP Coordinator is responsible for the compilation, review, verification and validation, and warehousing of all data produced by the ALMP. At the end of each sampling trip, water samples are submitted to the Central Laboratory for analyses. Hardcopies of the field data sheets are given to the ALMP Coordinator who manually enters the field data into the Lakes Database. Approximately 30 days after sample collection, the ALMP Coordinator receives hardcopy reports of the laboratory's finalized analytical results. The ALMP Coordinator reviews and manually enters the analytical results into the Lakes Database (see section A9 for specifics on data management). The finalized data are then used to determine the trophic status of each lake.

Reporting

The primary method of reporting for the ALMP is the Basin Assessment Reports (BAR). Results of lake monitoring efforts and lake classifications are reported for each of the seventeen major river basins in NC on a rotating five-year schedule based on the DWR Basinwide Planning Schedule (Table A6.2).

After reviewing lab analyses, previous reports and plans, and discussions with appropriate staff, results are presented by subbasin as narrative summaries, tables and graphical representations. Descriptions of known issues or sources of bias (e.g., analytical, field, climatic, significant events such as droughts or hurricanes, etc.) should be sufficient to give the reader adequate context for appropriate interpretation of the results. Each lake's data is summarized in a table by basin to help facilitate assessment.

The main audience for the information reported in the BAR is staff from the DWR Planning Section. Information from the BAR is used in developing the Integrated Report. The information also goes into the Planning Sections Basinwide Water Quality Plans (http://portal.ncdenr.org/web/wq/ps/bpu/basin).

Table A6.2: Basin Assessment Periods.

River Basin	Assessment Period	BAR Finalized
Broad Chowan Neuse Pasquotank	2006 – 2010	2011
Lumber Yadkin	2007 – 2011	2012
Catawba French Broad Tar-Pamlico	2008 – 2012	2013
Cape Fear New	2009 – 2013	2014
Hiwassee Little Tennessee Roanoke Savannah Watauga White Oak	2010 – 2014	2015

A7. Quality Objectives and Criteria

Precision, Accuracy, and Sensitivity

All field measurements, sample collection, preservation and handling are performed in accordance with the ISB SOP (Attachment 1). Analytical methods are performed according to the Laboratory Section's Quality Assurance Manual (Attachment 2). Quality assurance targets for accuracy and precision are listed in Table 5.1 of the Laboratory Section's Quality Assurance Manual (Attachment 2). Results from the ALMP are compared to NC Surface Water Standards (Attachment 3); reporting limits for these indicators should be at or below these critical values when possible.

Bias

The ALMP is based in judgmental sampling design; as a result, bias will exist due to station locations. However, this is acceptable given that stations are generally established at lake center or main-stem locations to capture whole-lake water quality and at the mouths of significant tributaries and/or tributaries suspected of contributing to water quality concerns in the lake.

Other sources of bias include:

- Samples are typically collected during the summer months, which often represent worst-case conditions in a lake.
- Sample size is limited by sampling summer months once every five years.
- Samples are collected during the day only. Stations may also be sampled at different times of the day from month to month, which may affect indicators such as DO, pH, and nutrients. Typically sampling is conducted after 10:00 A.M. to capture algal blooms that may be present and more active during the heat of the day.
- Extreme or unusual conditions, such as storm events, may not be sufficiently sampled due to field staff safety concerns during these events and scheduling.
- As noted above, most large reservoir sampling sites are located within the main-stem and near the mouths of large tributaries. Water quality conditions in coves, along the shoreline, and in small tributaries are not generally captured in the sampling effort.

Using consistent sampling methods, SOPs, and analytical methods minimizes bias from other sources.

Representativeness

Lake monitoring data generally show high variation due to natural conditions such as precipitation, diurnal patterns, and biological activity. Reservoirs also exhibit variation due to management of the water level for recreation, hydropower generation, and flood control.

In order to sample relatively stable conditions, the specified sampling point must have sufficient volume throughout the year. As a result, the collected samples represent an "average" condition of the waterbody at that point in time. In the event of volume loss due to drought or significant drawdown of a reservoir for dam repair, at least one year for stabilization of the system is required following the return to normal pool levels before sampling is resumed. However, under some circumstances these water bodies may be sampled within a year of volume loss to address specific needs.

Comparability

Fixed station locations and standardized operating procedures for sampling and analytical methods ensure that comparable samples are taken during each site visit.

Deviations from the SOP, due to unusual sampling conditions, are documented in the appropriate report or memorandum. Calibration procedures ensure accuracy and comparability of water quality measurements.

Measurements of water temperature, dissolved oxygen, specific conductance, and pH are made in the field with calibrated meters. The measurements are taken from just below the water surface (depth = 0.15 m) and at every meter to a depth of 10 meters. Readings are then taken every 5 meters until reaching a depth of 40 meters. After forty meters, readings are taken every 10 meters until reaching the bottom.

Completeness

It is expected that some site visits or samples will be missed due to problems such as inclement weather, equipment problems, vacant positions, and staff issues. Maximum possible effort is made to sample each monitored lake within a river basin three times within each five-year cycle period.

A8. Special Training/Certifications

Field Staff

Field personnel are trained in the methods described in the Intensive Survey Branch SOP (Attachment 1), this QAPP, and the Laboratory Section's Quality Assurance Manual (Attachment 2). Intensive Survey Branch staff generally performs initial training for new employees in meter calibration, required documentation, sampling methods, sample handling, safety, GPS operation, and other field activities. Employees are required to attend the Central Laboratory's sample submission training class when hired and take a refresher course every three years.

Staff performing boat work will be thoroughly trained in the safe and proper handling of boats and trailers. Hazardous material training is not required for Lakes Monitoring Program activities.

All staff involved in lake sampling participates in an annual field audit to evaluate knowledge of sampling procedures, safety, boat handling, and record keeping. Results of the field audits are tabulated and this information is used to evaluate the need for additional training and/or changes in protocols to improve overall quality assurance.

Laboratory (Analytical) Staff

Required training of DWR Laboratory Section staff is detailed in Section 4.0: *Organization, Facilities, and Equipment* of the Laboratory Section Quality Assurance Manual (Attachment 2). If a private laboratory is used for any analyses, it must certified by the appropriate NC Laboratory Certification program, and staff training must be performed in accordance with the requirements inherent in their certification. If another state agency's laboratory is used, its training requirements should be at least equivalent to those of a certified private laboratory.

A9. Documentation and Records

Quality Assurance Information, SOPs, and Other Program Documentation

Once all approval signatures have been obtained, the QA Coordinator will distribute copies of the approved QAPP via email to persons on the distribution list in Section A3 of this document. Copies must be disseminated within 30 days of final approval. The original hard copy with approval signatures will be kept on file in the QA Coordinator's office at ESS.

The QA Coordinator is to be notified of changes made to SOPs, analytical methods, and/or any other documentation referenced by this QAPP. This should be done before the summer field sampling season begins. The QA Coordinator will be responsible for distributing the information, as described above.

Since the ALMP is an ongoing project, this QAPP will be reviewed annually. If appropriate, changes or updates will be made at that time. However, critical revisions can be made at any time.

The QA Coordinator is responsible for completing revisions, obtaining signatures of approval, and disseminating the revised document to those on the distribution list within 30 days of final approval. The version or revision number and date shall be easily identifiable by the document control information on each page. A complete list of all revisions/updates will be provided with each annual update.

Field staff (listed in Section A4) that assist in the sample collection aspects of the ALMP are responsible for reading and reviewing the ALMP QAPP on an annual basis. Documentation that each staff member has reviewed this QAPP will be maintained by the QA Coordinator.

Project Records

Original hardcopies of all ALMP meter calibration sheets are retained a minimum of five years in the ESS Calibration Laboratory.

Original hardcopies of the following records are retained a minimum of five years in the "Lakes Files", which are located in the ESS building in Raleigh, NC:

- Field Data Sheets
- Stratified Field Data Sheets
- Field Observation Form
- Analytical Reports/Documents
- Sample Submission Sheets
- Analytical Laboratory Report

The following electronic records are retained indefinitely and are kept on the ALMP Coordinator's Lakes Database located on the ESS server:

- Physical Field Data
- Analytical Laboratory Results

Tape backups are run weekly on the ESS servers. The Lakes Database is updated weekly, at a minimum, during the summer sampling period. Details of electronic data management and warehousing methods are further described in section *B10: Data Management* of this document.

Analytical Laboratory Activities

Detailed descriptions for handling of original sample submission sheets, sample tags, and laboratory documentation and the required retention times and storage methods are listed in the Section 12 of the Laboratory Section Quality Assurance Manual (Attachment 2).

Data Reporting: Basin Assessment Reports

Data are analyzed and summarized for monitored lakes in each of the seventeen major basins on a rotating five-year schedule. The ALMP Coordinator provides all available historic and current raw data, data, station visit comments/observations, and station information (including lake classifications) as electronic files. These data are used to produce the ALMP portion of the Basin Assessment Report (BAR), which summarizes all monitoring activities during the appropriate assessment period. The final BAR is made publicly available via the ESS website at http://portal.ncdenr.org/web/wq/ess/reports.

The ALMP Coordinator also provides raw data upon request to staff from other state and federal agencies, private consultants, academia, municipalities, private citizens, and others. Raw data are generally provided in an electronic format (delimited text file or Microsoft Excel spreadsheet) and contain the same information listed above for internal analysis, unless otherwise instructed by the requestor.

SECTION B: DATA GENERATION AND ACQUISITION

B1. Sampling Process Design

The Federal Clean Lakes Program, which was established as Section 314 of the Clean Water Act (CWA) enabled North Carolina in 1981 to receive federal funding to classify the trophic (or nutrient enrichment) status of the State's publicly owned freshwater lakes and to prioritize lakes for restoration. A sampling program was established in 1981 to survey the trophic condition of 65 lakes. Thirty-one of these lakes were sampled again in 1982. From this work, the North Carolina Trophic State Index (NCTSI) that the State has used for all subsequent trophic classifications was developed.

The NCTSI is based on total phosphorus (TP in mg/L), total organic nitrogen (TON in mg/L), Secchi depth (SD in inches), and chlorophyll *a* (CHL in µg/L). Lake-wide means for TP, TON, SD, and CHL are used to calculate a NCTSI score for each lake, using the following equations:

$$NCTSI = TON_{Score} + TP_{Score} + SD_{Score} + CHL_{Score}$$

Where:

$$TON_{Score} = ((\text{Log } (TON) + 0.45)/0.24)*0.90$$

$$TP_{Score} = ((\text{Log } (TP) + 1.55)/0.35)*0.92$$

$$SD_{Score} = ((\text{Log } (SD) - 1.73)/0.35)*-0.82$$

$$CHL_{Score} = ((\text{Log } (CHL) - 1.00)/0.48)*0.83$$

In general, NCTSI scores relate to trophic classifications (Table B1.1). When scores border between classes, best professional judgment is used to assign an appropriate classification. NCTSI scores may be skewed by highly colored water typical of dystrophic lakes. Therefore, a trophic state is not assigned to dystrophic lakes.

Table B1.1: Trophic Classification Criteria for Lakes

NCTSI Score	Trophic Classification
< -2.0	Oligotrophic
-2.0 to 0.0	Mesotrophic
0.0 to 5.0	Eutrophic
> 5.0	Hypereutrophic

Data analysis is focused on those data generated during the five-year assessment period. Analysis begins with review of the data set for each indicator, generally lake-wide by sampling station. Graphical exploration of the data may be used if the lake has a number of sampling sites, generally greater than three, which makes such an analysis reasonable. Spatial patterns may be more evident at this point. Temporal patterns, with the exception of large step-type changes, are generally not easily discernable over such a short time period. Patterns or anomalies noted during this process are more closely examined and appropriate sources, such as Regional Office staff, NPDES permits, lake managers and municipalities, are consulted to determine a possible cause.

Station Locations

Stations are established at GIS referenced fixed locations (i.e., specific lat/long). A full station list is available in Attachment 4. Stations are strategically located to obtain data which are representative of the main body of the lake and capture the following:

- Overall water quality in the lake or reservoir;
- Effects of point source discharges;
- Effects of non-point sources of pollution (e.g., urban areas, animal operations, agriculture);
- Effects of land use changes;
- Levels of constituents of concern for load calculations, TMDLs, and determination of water quality standard attainment;
- Lakes of significant ecological, recreational, political, or municipal use;
- Lakes which show an impairment due to unknown causes (e.g., biological data shows possible impairment); and
- Lakes that have had public complaints or concerns related to water quality.

Many of the current stations have been active for over thirty years. This focus on long-term data is integral in identifying temporal patterns within a lake and to gaining an understanding of the variability within each system. Consequently, requests for station establishment and/or discontinuation are assessed based on the value gained from a long-term perspective. Special studies may also be conducted; however, these activities are beyond the scope of this QAPP. Study plans that contain QAPP information are developed for all special studies.

Adjustments to station locations and sampling regimens may be made with sufficient reason, such as:

- Safety concerns of field staff;
- Changes regarding location accessibility (i.e., sediment deposition preventing boat access);
- Original intent of sampling is no longer valid (i.e., a discontinued discharge);
- Emergence of new water quality concerns;
- Resource constraints, particularly field and laboratory staff vacancies; and/or
- Redundancy with a cooperating agency (e.g. sampling performed by lake managers, etc.).

If any of these concerns arise, the ALMP Coordinator, Intensive Survey Branch Supervisor, and any other involved parties (e.g., Planning Section staff), will collectively decide if it is appropriate to make modifications.

Actual sampling points are generally located within the center or main-stem of the lake, or as determined by field staff as representative of the lake or specific areas of concern within the lake.

Indicators Measured

The selection of indicators is primarily focused on those with NC water quality standards that can be cost-effectively analyzed and trophic state indicators appropriate to lakes. Additional indicators, which may not have specific standards associated with them but are useful for interpretation of water quality in lakes, are also included.

Field staff may use their discretion to sample for any additional indicators believed to be of concern due to unusual circumstances encountered during a lake visit. Permanent changes to parametric coverage at a station may be made in response to requests from DWR staff. These changes undergo a review process similar to that for station location changes.

Currently, all measurements and samples are taken on whole water samples. Analyses for dissolved fractions may be performed as part of special studies. See Table B1.2 for a complete listing of the indicators sampled. Attachment 4 lists the current ALMP stations and provides information on where additional indicators are collected. Core and lake specific indicators are collected during every sampling trip. When additional lake sampling is required, a study plan is developed to address sampling locations, parameters, frequency, and duration.

Table B1.2: Water Quality Indicators Collected in the ALMP

Indicator Type	Core Indicators	Lake-Specific Indicators
Physical	Temperature (°C) Specific conductance (μmhos/cm @25°C) Turbidity (NTU) Total residue (TS) Total suspended residue (TSS) Secchi depth (transparency)	
Chemical	Nutrients*: NH_3, NO_x, TKN, TON, TIN, TN, Total P Dissolved oxygen (DO) pH	Metals (water supplies only): Cd, Cr (total), Cu, Fe, Mn, Ni, Pb, Zn) Hardness (w*ater supplies only)* - Calculated using Calcium & Magnesium Chloride
Biological	Phytoplankton Chlorophyll *a* Aquatic macrophyte presences	Fecal coliform (Class B waters only) Aquatic macrophyte coverage Algal bloom (water color, mats, flecks, scums) Algal Growth Potential Tests (AGPT)
Other	Air temperature (°C) Cloud cover (%) Wind velocity (mi/hr) Wind direction (degrees from North) 48 hour precipitation (inches) Shoreline development and density Land uses observed in the watershed	

* Nitrogen species calculated using these equations: TON = TKN - NH_3 TN = TKN + NOx TIN = NH3 + NOx

Sampling and Measurements

Field measurements and samples are collected in accordance with Sections III, IV, and XI of the ISB SOP (Attachment 1). Required sample volumes, containers, preservation, and other handling information are detailed in Section 6 of the QA Manual for the Laboratory Section (Attachment 2).

After collection and chemical preservation, samples are immediately stored on ice in coolers. The sample coolers are either hand-delivered by field staff or sent via NC Department of Administration's courier to the appropriate DWR Laboratory for analyses.

There is no re-sampling in the event that samples are lost after leaving a lake or if samples are rejected by laboratory staff due to improper handing (temperature out of range, inadequate preservation, etc).

Every reasonable attempt is made by field staff to complete all site visits each month (May through September); however, some missed visits are to be expected due to situations such as bad weather, station inaccessibility, extreme drop in lake water volume, meter problems, staff shortages/vacancies, etc. In these cases, the reasons are documented in a memorandum, which is copied to the ALMP Coordinator, Intensive Survey Branch Supervisor, and the applicable lake file. If a station location is inaccessible during a sampling trip, field staff should consult with the ALMP Coordinator for consideration of changing the station location or permanent discontinuation of the station. It is important that stations not be moved without sufficient reason, as an uninterrupted long-term record is critical to this program.

B2. Sampling Methods

Samples and measurements are to be taken in accordance with ISB SOP sections III, IV, and XI (Attachment 1). Any irregularities or problems encountered by field staff are communicated, either verbally or via email, to the ALMP Coordinator who will assess the situation, consult with other project personnel, and recommend a course of action for resolution. Irregularities are documented on the field sheets by the field staff and by the ALMP Coordinator either in the database or by memo to the lake files. An overview of the different sampling methods employed is described below.

Field Measurements

Field parameters (temperature, dissolved oxygen, pH, and conductivity) are measured just below the water surface (depth = 0.15 m) and at every meter to a depth of 10 meters. Readings are then taken every 5 meters until reaching a depth of 40 meters. After forty meters, readings are taken every 10 meters until reaching the bottom.

A Secchi disk is lowered into the water to the depth at which the disk is no longer visible, then raised to where it just becomes visible. The average of these two depths is recorded as the Secchi depth. Additional information on this procedure is documented in Section III of the ISB SOP (Attachment 1).

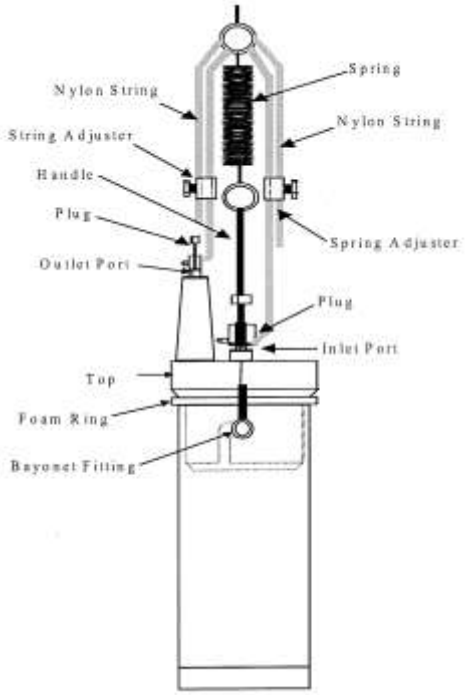
All readings and measurements are recorded in the field on a Stratified Field Data Sheet (Attachment 1, ISB SOP, Section II, Figure 5).

Samples

Refer to Section IV of the ISB SOP for general information on sampling methods (Attachment 1). Three basic methods are employed in the ALMP:

- Surface Grab: Samples are taken just below the surface (depth = 0.15m). Opened sample bottles are filled by plunging them into the water by hand. This method is used for fecal coliform bacteria, metals, and chloride.
- Photic Zone: A composite sample over the entire depth of the photic zone, defined as twice the Secchi depth, is taken using a Labline® Poly-Pro water sampler (see Figure B2.1). Corks are removed from the Labline® sampler, which is slowly lowered to a depth of twice the Secchi reading and then drawn back up to just below the surface. Lowering and raising the sampler is done at a slow, continuous pace in order fill the sampler with a representative sample of the entire water column to the designated depth. This method is used for chlorophyll *a*, nutrients, total solids, suspended solids, turbidity, and phytoplankton.
- Bottom (samples collected just above the bottom of the lake): These samples are collected by lowering a Labline® (with the cork holes plugged) to just above the bottom of the lake and then pulling the attached rope to release the corks, thereby filling the Labline®. Bottom grab samples are only conducted in certain instances (i.e., special lake studies involving sampling stations near dams with deep hypolimnetic water withdrawal).

Figure B2.1: Labline® Sampler



Detailed notes on any deviation from sampling protocol are documented on the field sheets and reported to the ALMP coordinator. The ALMP coordinator will review the affected data and determine if it is appropriate to use per Section D of this document.

Sources for equipment and disposables needed for lake sampling activities are listed in Table B2.1.

Table B2.1: Sources for Equipment and Disposables

	RESPONSIBLE SOURCE		
	ALMP Coordinator	**ALMP Staff**	**Central Laboratory**
Equipment			
Field Meters: Multiparameter Meter (ex, Hydrolab Quanta or MiniSonde 4a with display and probes).		X	
Labline® composite sampler with marked rope		X	
Safety equipment Disposable gloves (nitrile or vinyl) Acid handling equipment (apron, safety glasses, spill kit, portable eye wash, ampule disposal container) First Aid kit Personal floatation devices		X	
Secchi disk		X	
Coolers/ice chests/temperature blanks		X	
Truck/van		X	
Boat and trailer		X	
Disposables			
Sample bottles			X
Sample tags	X		
Sample submission sheets	X		
pH buffers (4.0, 7.0, 10.0 s.u.)		X	
Conductivity standards (500, 1000 *u*mhos/cm)		X	
25% sulfuric acid			X
1:1 nitric acid ampules			X
Lugol's solution		X	
Distilled or deionized water*		X	
Ice		X	

* From the Environmental Sciences Section Calibration Laboratory.

B3. Sample Handling and Custody

All samples are to be handled by field staff in accordance with Sections 6-7 of the Laboratory Section QAM (Attachment 2) except for completion of Chain of Custody forms. Chain of Custody forms are not typically completed for ALMP sampling. They are only completed if sampling is conducted for enforcement purposes.

Sample Preservation

Chemical preservation of samples should occur within 15 minutes of collection. Samples should then immediately be placed in coolers with ice. Sample submissions requirements (i.e. container specifications, minimum sample volumes, preservation, and holding times) are listed in Figure 6.1 of the Laboratory's QAM (Attachment 2).

Sample Submission Forms

The ALMP Coordinator prepares sample submission forms (also called Field Lab Forms) (Figure B3.1) for each sampling month. Each sheet corresponds to one or more samples that were taken using the same sampling method (i.e., grab, photic, bottom) at the same station, date, and time. If more than one sampling method is employed at a single station visit, multiple sheets must be completed for that station. This means that for certain station visits, up to three sample submission forms may be required:

- Surface: grab samples submitted to the Central Laboratory
- Photic: photic zone composite samples submitted to the Central Laboratory
- Bottom: bottom grab samples submitted to the Central Laboratory

The flow of ALMP Sample Submission Forms is displayed in Figure B3.2.

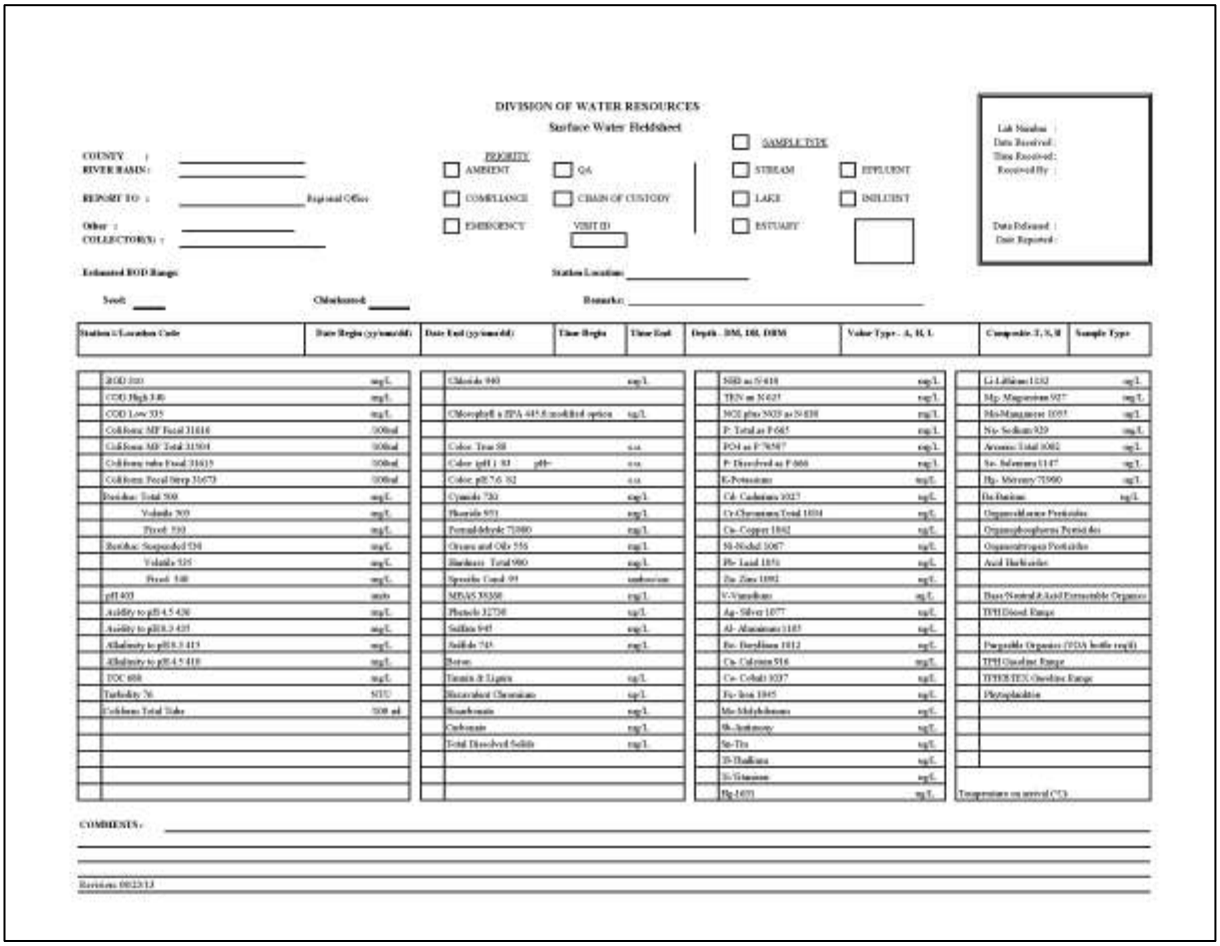
An example of a completed sample submission form is shown in Figure B3.1. Most information is pre-printed; however, the following fields must be completed by field staff using waterproof ink:

- Collector(s): Collector's first initial and last name (e.g., J. Smith)
- Date Begin: Date sampled (yymmdd)
- Time Begin: Time sampled (hhmm)
- Depth: For photic samples, depth of photic zone sample; depth of bottom for bottom grab samples; this field already completed for surface grab samples
- Sample set ID: Unique shorthand identifier allowing sample to be matched to appropriate sample tag

Sample Identification Tag

An example of a completed tag used for sample identification is shown in Figure B3.3. Tags should be filled out using waterproof ink and attached to the neck of the appropriate sample bottle with rubber bands immediately after sampling.

Figure B3.1: ALMP Sample Submission Form



DIVISION OF WATER RESOURCES
Surface Water Fieldsheet

COUNTY :
RIVER BASIN :
REPORT TO : Regional Office
Other :
COLLECTOR(S) :

PRIORITY
AMBIENT
COMPLIANCE
EMERGENCY
QA
CHAIN OF CUSTODY
VISIT ID

SAMPLE TYPE
STREAM
LAKE
ESTUARY
EFFLUENT
INFLUENT

Lab Number :
Date Received :
Time Received :
Received By :
Data Released :
Date Reported :

Estimated BOD Range:
Seed:
Chlorinated:
Station Location:
Remarks:

COMMENTS :

Figure B3.2: Flow of ALMP Sample Submission Forms

SAMPLE SUBMISSION FORMS CREATED

- Each month's forms printed by ALMP Coordinator and given to field staff.
- Includes sample set ID, station information, and all samples scheduled.
- Separate sheets for:
  - Surface grabs
  - Bottom grabs
  - Photic zone composites

Sample Submission Forms are completed in the field

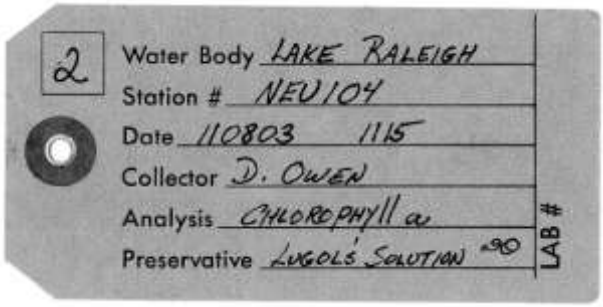
SAMPLES SUBMITTED FOR ANALYSES

Samples and completed Sample Submission Forms are submitted to Laboratory

ANALYTICAL RESULTS

Within 30 days of submittal, Sample Submission Forms and Lab Results are returned to ALMP Coordinator for review and data entry.

Figure B3.3: Completed ALMP Sample Tag



Guidance for proper completion of sample tags is listed below:

- Water Body: Station location description
- Station #: lake station number
- Date: Date sampled (yymmdd); for chlorophyll a and fecal coliform bacteria samples, the time that the sample was collected is added behind the date (hhmm)
- Collector: Name of collector (first initial, last name)
- Analysis: Name of analysis requested
- Preservative: Identification of preservation methods. This item is initialed by collector upon preservation of sample with chemical preservative.
- Sample set ID: Box in upper left hand corner; unique shorthand identifier allowing sample to be matched to appropriate sample submission sheet

Note that forms for surface (SUR) and bottom (BOT) samples have the station number followed SUR or BOT (examples: LTN008ESUR or LTN008EBOT).

Sample Transport

Immediately after sampling, labeling, and chemical preservation, samples are placed in coolers on ice. Sample submission forms are placed in a sealable waterproof bag and are taped to the inside of the cooler if samples are to be shipped. Otherwise, the forms are kept with the field staff until the coolers are delivered to the lab. Coolers are then either delivered to the lab by field staff or sealed and shipped via the NC Department of Administration's Courier Service to the appropriate lab.

Laboratory

Once samples arrive at the laboratory, support staff check the temperature blank (included in each cooler) to ensure that the samples are within the appropriate temperature range (4°C, ±2°), assign lab tracking numbers, and distribute the samples to the appropriate analytical branches.

Any samples not meeting temperature, holding time, or preservation requirements, or otherwise not submitted in accordance with the SOP are subject to rejection as per Section 7.0 of the Laboratory's QAM (Attachment 2). Laboratory staff will attempt to contact the collector by phone or email before rejecting samples. If conditionally accepted, the laboratory will document the anomaly as a "Sample Condition Upon Receipt" (SCUR) and/or Sample Anomaly Report (SAR) form and include copies with the final analytical report. Results from anomalous samples will also be reported using the appropriate qualification code.

For details of laboratory protocols for sample receipt and handling, refer to Section 7: Sample Custody of the Laboratory's QAM (Attachment 2).

B4. Analytical Methods

Field Measurements

All field parameter measurements performed in the field are listed in Table B4.1. Methods for measurement of these parameters are included in Section III of the ISB SOP (Attachment 1). Instruction manuals for the appropriate meter should also be consulted for instruction on proper meter operation.

Table B4.1: Field Measurement Method References and Reporting Levels

Parameter	EPA Method (if applicable)	Reported to Nearest…
Dissolved Oxygen	360.1	0.1 mg/L
pH	150.1	0.1 SU
Water temp	170.1	0.1 ºC
Specific conductance	120.1	1 umhos/cm
Depth		0.1 m
Secchi depth		0.1 m

Lab Analyses

A summary of methods and reporting limits are listed below in Table B4.2. More detailed information on sample preparation methods, approved method modifications, method performance criteria, precision, accuracy, MDLs and PQLs can be found in the Laboratory Section's QAM (Attachment 2; Table 5.1: QA Targets for Accuracy, Precision, and MDLs/PQLs and Section 8: Analytical Procedures).

Table B4.2: Analytical Method References and Reporting Levels

Parameter	EPA Methods	APHA Methods	Other Methods	Practical Quantitation Limit (PQL)	PQL Revision Date
pH		SM 4500-H+ B		0.01 units from 4.01 - 9.18 s.u.	3/13/2001
Conductance @ 25°C		SM 2510 B		14.9 µmhos/cm	3/30/2001
BOD_5		SM 5210 B		2.0 mg/L	3/13/2001
$CBOD_5$		SM 5210 B		2.0 mg/L	6/5/2007
COD			HACH 8000	20 mg/L	9/5/2002
Coliform, MF Fecal		SM 9222 D		1 colony/100 mL	3/13/2001
Coliform, MF Total		SM 9222 B		1 colony/100 mL	3/13/2001
Coliform, Tube Fecal		SM 9221 B		MPN/100 mL	3/13/2001
Coliform, Fecal Strep		SM 9230 C		1 colony/100 mL	3/13/2001
Total Residue		SM 2540 B		12 mg/L	6/1/2007
Total Volatile Residue	EPA 160.4			12 mg/L	6/1/2007
Total Fixed Residue	EPA 160.4			12 mg/L	6/1/2007
Total Suspended Residue		SM 2540 D		6.2 mg/L	6/1/2007
Suspended Volatile Residue	EPA 160.4			6.2 mg/L	6/1/2007
Suspended Fixed Residue	EPA 160.4			6.2 mg/L	6/1/2007
Total Dissolved Residue		SM 2540 C		12 mg/L	6/1/2007
Alkalinity to pH 8.3		SM 2320 B		1 mg/L	3/13/2001
Alkalinity to pH 4.5		SM 2320 B		1 mg/L	3/13/2001
TOC		SM 5310 B		2 mg/L	
Turbidity	EPA 180.1 Rev. 2.0 (1993)	SM 2130 B		1 NTU	3/13/2001
Chloride	EPA 300.0 Rev. 2.1 (1993)		QUIK CHEM 10-510-00-1-A$	1 mg/L	4/1/2007
Chlorophyll *a* EPA 445.0 modified option	EPA 445.0			1 µg/L*	3/13/2001
Color: True		SM 2120 C		5 color units	3/13/2001
Color: ADMI		SM 2120 E		25 color units	3/13/2001
Cyanide, Total	EPA 335.4 Rev. 1.0 (1993)$		QUIK CHEM 10-204-00-1-A QUIK CHEM 10-204-00-1-X	0.02 mg/L	4/22/2002
Fluoride	EPA 300.0 Rev. 2.1 (1993)		QUIK CHEM 10-510-00-1-A$	0.4 mg/L	4/1/2007
Formaldehyde			APHA, 1972 Method 111	0.2 mg/L	2/21/2003
HEM (Oil and Grease)	EPA 1664 A			10 mg/L	11/1/2005
HEM (Oil and Grease) sludge, sediment and solid samples			SW-846 9071 B	1000 mg/Kg	11/1/2005

Parameter	EPA Methods	APHA Methods	Other Methods	Practical Quantitation Limit (PQL)	PQL Revision Date
MBAS		SM 5540 C		0.1 mg/L	3/13/2001
Phenols	EPA 420.4 Rev. 1.0 (1993)		QUIK CHEM 10-210-00-1-A$	10 µg/L	
Silica		SM 4500-SiO2 C	QUIK CHEM 10-114-27-1-A$	2 mg/L	4/16/2002
Sulfate	EPA 300.0 Rev. 2.1 (1993)		QUIK CHEM 10-510-00-1-A$	2 mg/L	4/1/2007
Sulfide		SM 4500-S2 D		0.1 mg/L	3/13/2001
Hexavalent Chromium		SM 3500-Cr B (20th Edition)		50 µg/L	4/22/2002
NH3 as N	EPA 350.1 Rev. 2.0 (1993)		QUIK CHEM 10-107-06-1-J$	0.02 mg/L	3/1/2009
TKN as N	EPA 351.2 Rev. 2.0 (1993)		QUIK CHEM 10-107-06-2-H$	0.20 mg/L	3/1/2009
NO2 + NO3 as N	EPA 353.2 Rev. 2.0 (1993)		QUIK CHEM 10-107-04-1-C$	0.02 mg/L	3/1/2009
P, Total as P	EPA 365.1 Rev. 2.0 (1993)		QUIK CHEM 10-115-01-1-EF$	0.02 mg/L	6/1/2008
PO4 as P	EPA 365.1 Rev. 2.0 (1993)		QUIK CHEM 10-115-01-1-A$	0.02 mg/L	3/1/2009
P, Dissolved as P	EPA 365.1 Rev. 2.0 (1993)		QUIK CHEM 10-115-01-1-EF$	0.02 mg/L	3/1/2009
NO2 as N	EPA 353.2 Rev. 2.0 (1993)		QUIK CHEM 10-107-04-1-C$	0.01 mg/L	3/1/2009
Boron	EPA 200.7 Rev. 4.4 (1994)			50 µg/L	1/12/2009
Cadmium	EPA 200.8 Rev. 5.4 (1994) EPA 200.9 Rev. 2.2 (1994)$			1.0 µg/L	1/2/2007
Chromium, Total	EPA 200.8 Rev. 5.4 (1994) EPA 200.7 Rev. 4.4 (1994)$			10 µg/L	1/2/2007
Copper	EPA 200.8 Rev. 5.4 (1994) EPA 200.9 Rev. 2.2 (1994)$			2.0 µg/L	3/13/2001
Nickel	EPA 200.8 Rev. 5.4 (1994) EPA 200.9 Rev. 2.2 (1994)$			2 µg/L	3/1/2011
Lead	EPA 200.8 Rev. 5.4 (1994) EPA 200.9 Rev. 2.2 (1994)$			2 µg/L	3/1/2011
Zinc	EPA 200.8 Rev. 5.4 (1994) EPA 200.7 Rev. 4.4 (1994)$			10 µg/L	3/13/2001
Silver	EPA 200.8 Rev. 5.4 (1994) EPA 200.9 Rev. 2.2 (1994)$			5 µg/L	3/13/2001
Aluminum	EPA 200.7 Rev. 4.4 (1994)			50 µg/L	3/13/2001

EPA refers to *Methods for Chemical Analysis of Water and Wastes* , USEPA Office of Research and Development, Rev. 3/83 (unless otherwise specified). Cincinnati, OH; EPA 600/4-79-020

SM refers to *Standard Methods for the Examination of Water and Wastewater* , 18th Edition (unless otherwise specified), American Public Health Association, Washington, DC, 1992 (unless otherwise specified).

SW-846 refers to *Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods* ; 3rd edition (9/86), USEPA Office of Solid Waste and emergency Response, Washington, D.C.

HACH refers to Hach Chemical Company, PO Box 389, Loveland, CO 80537.

QUIK CHEM refers to HACH Company/Lachat Instruments, Milwaukee, WI, 53219.

* - under evaluation

$ - secondary method reference

.

B5. Quality Control Requirements

Field Activities

Field water quality instruments are calibrated for each sampling trip prior to that day's work. Meter calibrations for dissolved oxygen (D.O.), pH, and specific conductance are checked after each sampling trip to confirm that significant drift has not occurred and that the data collected is accurate and representative. If post-sampling calibration readings are beyond acceptable limits (D.O. = $\pm$0.5; pH = $\pm$0.2; conductance = $\pm$10%), the data are discounted and are not entered into the databases.

A temperature blank is included in each cooler containing samples to be analyzed by the Central Laboratory.

Laboratory Activities

Quality control for analytical samples is conducted per Section 11 of the Laboratory Section's QAM (Attachment 2).

B6. Instrument/Equipment Testing, Inspection, and Maintenance

A routine preventative maintenance program minimizes the occurrence of instrument and equipment failures. All equipment should be visually inspected for damage at the start of each sampling day and repaired or cleaned if needed before further use. Required maintenance is shown in Table B6.1.

Field Equipment

Information on equipment cleaning is supplied in Chapter VI of the ISB SOP for field meters, equipment, and vehicles (Attachment 1). Other required maintenance is shown in Table B6.1. Operator's manuals for all equipment should be consulted for manufacturer's recommendations for inspection, maintenance, and repair. Problems with meters and other equipment should be reported to the ISB Equipment Manager who is responsible for overseeing equipment maintenance and repairs.

Table B6.1: Field Equipment Maintenance

Equipment	Task	Frequency
Multiparameter Meters	Check battery level	Daily
	Inspect membrane for holes, tears, bubbles, fouling or other damage Inspect probes for damage,	Daily
Labline®	Check distance measurements on rope	Annually
	Clean and inspect Labline®	Before each sampling trip
Secchi Disk	Check distance measurements on rope	Annually

Laboratory Analytical Equipment

For laboratory equipment and instrument inspection and maintenance, refer to the Laboratory Section's QAM, Table 10.1 (Attachment 2).

B7. Instrument Calibration & Frequency

Field Meters

Field meters are inspected and calibrated before each sampling trip and at the beginning and end of each day used. Pre- and post-sampling calibration information is recorded on the Water Quality Monitoring Field Meter Calibration Sheet (ISB SOP, Figure 10). Completed calibration forms are stored in the ISB calibration room and are retained for at least five years. The specific calibration procedures are documented in Section III of the Intensive Survey Branch's SOP (Attachment 1) and in each meter's instruction manual.

Calibration standards are selected so that they bracket the range of measurements expected that day. Meters should also be checked against standards periodically throughout the day and recalibrated if needed if any of the following occur:

- Physical shock to meter;
- DO membrane is touched, fouled, or dries out;
- Unusual (high or low for the particular site) or erratic readings, or excessive drift;
- Extreme readings (e.g., extremely acidic or basic pH; D.O. saturation >120%);
- Measurements are outside of the range for which the meter was calibrated.

Standards

Traceable conductivity standards and standards for pH (buffers) are purchased. Meters currently used to measure pH require standards of 4.0, 7.0, and 10.0 S.U. Conductivity standards used are 500 and 1000 μmhos/cm at 25 °C.

Laboratory Instrumentation Calibration

For details of laboratory requirements and methods of calibration of analytical laboratory instrumentation, refer to Section 9 of the Laboratory Section's QAM (Attachment 2).

B8. Inspection/Acceptance Requirements for Supplies and Consumables

Sample submissions requirements (i.e. container specifications, minimum sample volumes, preservation, and holding times) are listed in Figure 6.1 of the Laboratory's QAM (Attachment 2).

The Central Laboratory is responsible for purchasing and quality assuring sample bottles, reagents, and chemical preservatives used by the ALMP field staff.

ALMP field staff members are responsible for visibly inspecting all sample bottles before use. Any bottles that are visibly dirty or whose lids have come off during storage are discarded. It is recommended that field staff periodically check bottles for contamination attributed to storage conditions by filling representative containers with analyte-free water (available from the Laboratory Section), adding the appropriate preservative(s), and submitting them to the laboratory for metals and wet chemistry analyses. Any container lots showing analyte levels at or above the reporting limits should be discarded.

Manufacturer's certificates of purity for chemical preservatives are retained by the Laboratory Section Support Branch. If alternative suppliers of chemical preservative are used by the ALMP, the preservatives must be ACS-grade or equivalent, and the manufacturer should provide a certificate of purity or equivalent indicating that contaminants of interest are below the Laboratory's current reporting limits. Any preservatives that show signs of contamination, such as discoloration or the presence of debris or other solids, should be not be used and should be discarded.

A list of supplies and consumables is available in Table B2.1.

B9. Acquired Data (Non-Direct Measurements)

All data will be generated through ALMP field activities and laboratory analyses, with the following exceptions:

48-hour precipitation (inches/day): Data are obtained from the State Climate Office of North Carolina via the NC CRONOS database, which is available on the Internet at: http://www.nc-climate.ncsu.edu/cronos/. There are data available from approximately 657 weather monitoring stations across NC. If an appropriate station cannot be located, field staff may obtain approximate values from local news' weather services or the National Weather Service. In both cases, data are used for relative interpretations of other parameters such as fecal coliform or turbidity that may be affected by recent runoff. Data may also be obtained from rain records maintained for reservoirs managed by the US Army Corps of Engineers.

Anecdotal lake information: Information regarding lake management activities, public complaints of water taste and odor, general water quality, and fish health are collected from lake managers, lake owners associations, public environmental groups, and water treatment facility supervisors. Other information, such as estimated aquatic weed coverage and observed algal blooms, is also collected from these sources.

Records of public complaints: Documented water quality issues are collected from public health departments and the NC Division of Health and Human Services, water treatment facility supervisors, NC Water Resources staff, NC Division of Water Resources Regional Office staff, universities, and power generation companies. Examples include letters of complaint from the public, laboratory and field studies, notices of swimming area closures, and fish consumption advisories.

Maps: USGS Quads, county map books and ALMP lake files are sources of maps used in locating lakes and stations on the lakes. Other map sources may be used. Latitude and longitude of lake stations are determined on the first sampling trip, are verified once every five years and are maintained in the AMLP database.

B10. Data Management

Field measurements and observations are recorded on Stratified Field Data Sheets and Field Observations Forms (Attachment 1; Figures 5 and 19). Completed field sheets are submitted to the ALMP Coordinator who manually enters the data into the Lakes Database. Original hardcopies of all Lake Stratified Field Data Sheets, Laboratory Analytical Reports and Lake Field Observations Forms are retained a minimum of ten years (or the most recent two sampling trips) in the "Lakes Files", which are located in the ESS building in Raleigh, NC. Table B10.1 presents the retention time for paper and electronic data generated by the Intensive Survey Branch.

Table B10.1. Retention times for Lakes Data generated by ISB.

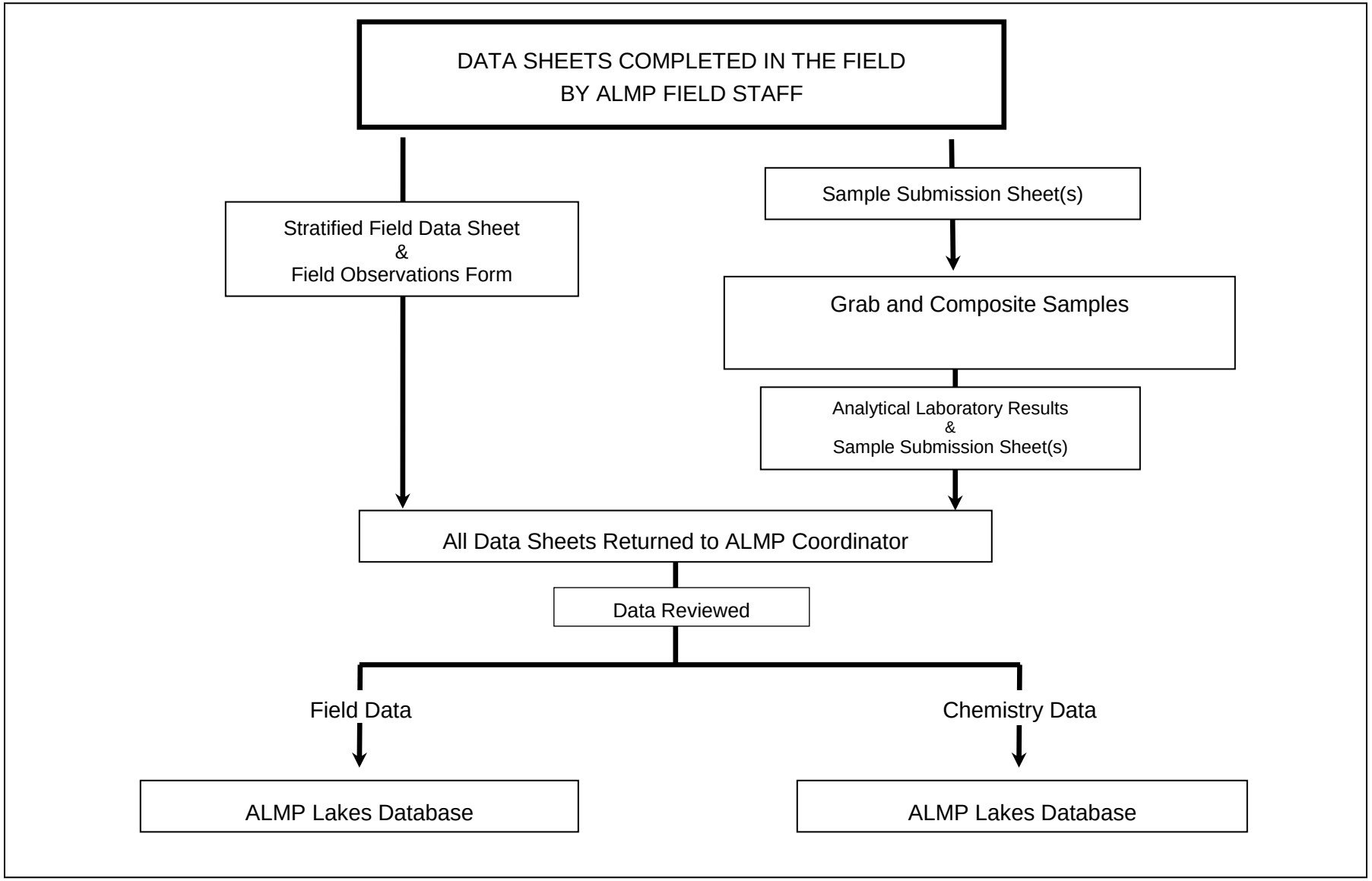
Record	Retention Time
Lake Stratified Field Data Sheet	Two (2) most recent sampling years for ambient monitoring
Laboratory Analytical Report	Two (2) most recent sampling years for ambient monitoring
Lake Field Observation Form	Two (2) most recent sampling years for ambient monitoring
Electronic Lake Monitoring Data	Indefinitely

Immediately after collection, water samples and the corresponding Sample Submission Forms (Figure B3.1) are submitted to the Central Laboratory for analyses. Within 30 days of sample submission to the Central Laboratory, the ALMP Coordinator receives the Laboratory's analytical results (including data qualifier codes). Hardcopies of the Sample Submittal Forms and any Sample Anomaly Reports (SARs) or Sample Condition Upon Receipt (SCUR) forms are also provided to the ALMP Coordinator. The ALMP coordinator reviews all analytical results as they are received and manually enters these data into the Lakes Database.

The Lakes Database is "housed" on the ESS server, which is backed up every week. The ALMP Coordinator reviews the databases for completeness, data entry errors, unlikely/impossible values, etc. as detailed in Section D of this document. Data is provided to EPA and other parties upon request as electronic files or hard copy.

An overview of the data flow for the ALMP is displayed in Figure B10.1.

Figure B10.1: ALMP Data Flow



SECTION C: ASSESSMENT AND OVERSIGHT

C1. Data Quality Assessments & Response Actions

Data quality is a high priority for the ALMP and a comprehensive assessment and oversight program is currently under development. At present, a field audit to evaluate the QA/QC of sampling efforts is in place. Also, data entered into the Lakes Database is evaluated for completeness and accuracy following the receipt of the final laboratory sample report from the Central Laboratory for the summer lake sampling effort.

Field Monitoring

Each field crew is composed on at least one member that has participated in an annual lake monitoring audit. This individual is responsible for ensuring that proper meter calibration, sampling techniques and documentation are employed. Any modifications to sampling techniques are reviewed with all staff prior to start of the sampling season. The ALMP Coordinator is responsible for overseeing training of new crew members and training of staff in new lake monitoring methods.

Field staff participates every three years in the Laboratory's Sample Submission Training and annually in the USGS National Field Quality Assurance Program (NFQA). The NFQA was created in 1979 to provide quality-assurance reference samples to field personnel who make water quality field measurements. The program verifies the proficiency of pH, and specific conductance measurements collected by water quality field analysts. Individuals failing a test are required to undergo additional training until proficiency is achieved. The ESS QA Coordinator coordinates this testing.

Further requirements are under developed.

Laboratory Activities

The Laboratory Section has a robust assessment program in place. Refer to Sections 13 and 14 of the Laboratory QAM for details (Attachment 2).

C2. Reports to Management

Reporting of issues related to data collected for the ALMP involve different approaches based on the nature of issues/deficiencies encountered in the course of the administration of this program. Methods of reporting include, but are not limited to the following:

- Oral and written notification to the ALMP Coordinator of issues related to sampling and data collection by field staff.
- Written reports regarding significant issues from the ALMP Coordinator to the Program Manager. Issues of interest to the Environmental Sciences Section as a whole are included in the Monthly Branch Update submitted by the Program Manager to the Section Chief.
- Oral and written notification to field staff and the ALMP Coordinator of significant issues related to sample submissions by the laboratories.
- Weekly oral reports on general status of ALMP to Program Manager.

A discussion of issues regarding QA assessments is included in the BAR, as needed. This discussion includes the parameter of concern, the nature of the QA issue and how the results for the parameter are affected. If the validity of the parameter results cannot be determined due to QA problems, the data are not included water quality assessments for the BAR or sent out as part of raw data requests.

SECTION D: DATA VALIDATION AND USABILITY

D1. Data Review, Verification, and Validation

Verification and validation occurs at each step of data generation and handling. It is the responsibility of field staff, laboratory bench chemists and support staff, and the ALMP Coordinator to verify that all records and results produced or handled are accurately transcribed, transmitted, and recorded. Each ALMP staff member is also responsible for ensuring that all activities (measurements, sampling, and analysis) comply with all requirements outlined in the following project documents:

- ALMP QAPP
- ISB SOP (Attachment 1)
- Laboratory Section QAM (Attachment 2)

The ALMP Coordinator performs primary review, validation, and verification duties of results reported by field staff and the Laboratory Section on an ongoing basis. This process involves comparison of new data with previously collected data and known historical water quality trends at the lake. If data are found to be unusual for a particular lake, actions may include a request to the lab to verify the data, an investigation of lake and watershed activities that could have influenced the data results, and a check of how the sample was collected to determine if sample collection and handling influenced the data results.

Field parameter data are considered invalid if post-sampling meter calibrations for DO, pH and specific conductance are beyond acceptable limits as indicated on the calibration sheet (ISB SOP, Figure 10). If meter calibrations are not within the acceptable limits, the data are discounted and are not entered in the Lakes Database.

D2. Validation and Verification of Methods

Field staff

Field staff will visually inspect the following items as they are produced to ensure accuracy and completeness:

Sample tags

Sample submission documentation

Field data worksheets

Lake descriptive data forms

Laboratory

The Laboratory Section's data verification and validation activities are described in their QAM (Attachment 2). *Section 7: Sample Custody* describes the activities involved in sample receipt and *Section 12.2: Data Verification* details the verification of analytical results.

In the event samples do not meet criteria outlined in the QAM, the Laboratory Section will indicate this using their standard Sample Condition Upon Receipt (SCUR) form, Sample Anomaly Report (SAR), and flag the subsequent result using a standardized list of qualifier codes. A full list of these codes is shown in Attachment 5.

ALMP Coordinator

The ALMP Coordinator, on an ongoing basis, performs the review, validation and verification of data results reported by field staff and the Laboratory Section. Data entry into the Lakes Database is also performed by the ALMP Coordinator.

When errors or omissions are found or suspected, corrections will be made using available hard copy laboratory reports and hand written field data forms. If these still do not contain the needed information, the field staff that conducted the sampling/measurement or the appropriate Laboratory Chemist will be contacted so they can consult their records. A determination of the source of the error is made (i.e., sample analysis error, data transcription error, etc.). If the source of the error cannot be determined or if an accurate reading cannot be obtained, the data is dropped from the Lakes Database and a comment is added to indicate why the data was not entered. Data that have been given an SAR code or a SCUR are entered into the Comments block of the database with the laboratory SAR code. These data are not used for regulatory purposes but may be used to determine if additional sampling or staff training is required depending on the nature of the code.

Data end-users

Data retrievals from the ALMP that are found to have odd or possibly incorrect values should be brought to the attention of the ALMP Coordinator. Consultation with field staff and laboratory personnel will be employed as deemed necessary to resolve data questions or issues.

D3. Reconciliation With User Requirements

A main objective of the ALMP is to provide data for use in determining lake water quality. This information is combined with other available data by the ISB and Planning Section staff to support reporting requirements such as 303(d)/305(b) reporting.

Though the major objectives of the ALMP are best served by a relatively stable monitoring schema, the system does allow for some flexibility in addressing the needs of its primary data users. Adjustments to the current ALMP can be accommodated, if deemed appropriate and sufficient resources exist, as concerns are raised. Appropriate reasons for adjustments are discussed in Section B1 of this document.

The ALMP also undergoes regular reviews; each basin's lake station locations and the indicators measured are assessed during the Basinwide Planning process. ALMP staff participates in basinwide monitoring pre-planning meetings with regional office staff prior to the beginning of each sampling season. Appropriate adjustments are made in response to needs, emerging water quality issues, and concerns identified by Regional Office staff observations and public comments received regarding lake water quality/lake watershed issues.

SECTION E: REFERENCES

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ATTACHMENTS

ATTACHMENT 1: INTENSIVE SURVEY BRANCH STANDARD OPERATING PROCEDURES

ATTACHMENT 2: QUALITY ASSURANCE MANUAL FOR THE NC DWR LABORATORY SECTION

ATTACHMENT 3: NC SURFACE WATER QUALITY STANDARDS

ATTACHMENT 4: NC DWR ALMP STATION INFORMATION

ATTACHMENT 5: NCDENR/DWR CHEMISTRY LABORATORY DATA QUALIFIER CODES

ATTACHMENT 1:

INTENSIVE SURVEY BRANCH STANDARD OPERATING PROCEDURES

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INTENSIVE SURVEY BRANCH
STANDARD OPERATING PROCEDURES MANUAL:
PHYSICAL AND CHEMICAL MONITORING

Version 2.1

December 2013

This document has been approved for release by:

Jason Green 12/10/2013

Jason Green — Date

Supervisor, Intensive Survey Branch

Dianne M. Reid 12/11/13

Dianne M. Reid — Date

Chief, Environmental Sciences Section

N.C. DEPARTMENT OF ENVIRONMENT AND NATURAL RESOURCES

DIVISION OF WATER RESOURCES

ENVIRONMENTAL SCIENCES SECTION

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INTRODUCTION

This manual contains the standard operating procedures (SOP) employed by the North Carolina Division of Water Resources (DWR) to evaluate water quality. It is intended to encompass all aspects of routine physical and chemical water quality monitoring with the occasional sediment samples. Therefore, this manual is to be considered a working, dynamic guideline for DWR personnel. Efforts to improve current procedures will continue, and the manual will be revised periodically, as needs dictate.

The primary goal of the manual is to promote the use of procedures that are consistent and reliable during field operations. All employees of the DWR staff are expected to be familiar with and to utilize these procedures as appropriate tools for water quality data collection. Because the procedures have been presented to cover a broad range of applications encountered in water quality monitoring, modifications may be necessary for specific conditions. Deviations from the procedures outlined in this manual, however, should be documented at time of collection.

These standard operating procedures apply to surface water, waste water, and sediment. The manual details procedures for sample collection and handling, as well as methods for parameters that must be measured in situ.

Procedures are referenced at the end of each section. In addition, all references are compiled in Section XIII. Mention of trade names or commercial products does not constitute endorsement or recommendation for use by the Division of Water Resources.

These standard operating procedures will assist the Division of Water Resources in its efforts to monitor the waters of the state with increased accuracy and confidence.

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FIGURES

INTENSIVE SURVEY BRANCH PROCEDURES DOCUMENT REVIEW LOG

SOP Name: **Intensive Survey Branch Standard Operating Procedures Manual: Physical and Chemical Monitoring**

Version: 2.1

Revised: December 10, 2013

Jason Green — Prepared By: — 12/10/2013 — Date

Jason Green — Branch Approval By: — 12/10/2013 — Date

Dianne M. Reid — Section Approval By: — 12/11/2013 — Date

Completed hard copy & signed original maintained by ISB Supervisor

DOCUMENT READ BY:			
Name (print):	Date Read:	Version Read:	Signature:

Completed hard copy maintained by ISB Supervisor

INTENSIVE SURVEY BRANCH SOP REVISION LOG

Date	Version Edited	Chapter	Changes	Name
12-10-13	Ver 2.0	Cover page	Updated document date & version to Decemberr 2013 and Version 2.1	Jason Green
12-10-13	Ver 2.0	Cover page & Review Log	Updated Section Chief to Dianne M. Reid	Jason Green
12-10-13	Ver 2.0	Entire	Division of Water Quality (DWQ) changed to Division of Water Resources (DWR). Intensive Survey Unit (ISU) changed to Intensive Survey Branch (ISB). Updated all headers, footers, and hyperlinks.	Jason Green
12-10-13	Ver 2.0	I. 3.3	Addition of method for use of churn splitter for sampling	Jason Green
12-10-13	Ver 2.0	II. 1.2	Updated Figure 6. – Lab sheet	Joanna Gmyr
12-10-13	Ver 2.0	II. 3.2	Updated Figure 9. – Chain of Custody Form	Joanna Gmyr
12-10-13	Ver 2.0	II. 4	Updated Figure 10. – Meter Calibration Sheet	Joanna Gmyr
12-10-13	Ver 2.0	III. 6.1	Edited Secchi Depth method to include average value & updated Figure 11 to match method	Jason Green
12-10-13	Ver 2.0	IV. 1	Addition of sample temperature blank and sample viability upon delivery	Jason Green
12-10-13	Ver 2.0	VIII. 4.1.1.g	Added Field Sheet reference to Appendix 7	Jason Green
12-10-13	Ver 2.0	Appendix	Added Appendix 7 – Flow Measurement Field Sheet	Jason Green
4-11-11	Ver 1.3	Appendix	Updated DWR guidance sheets for YSI 85, Accumet, Hydrolab, YSI Pro Plus, & YSI 6920 meters. Inserted Appendix 5 - Uncorrected DO Table	Joanna Gmyr
4-11-11	Ver 1.3	II.4 and III.1 -5	Updated field meter and calibration procedures.	Joanna Gmyr
7-15-11	Ver 1.3	1	Moved sections	Danielle Mir
10-2011	Ver 1.3	Entire	Made multiple review edits	Danielle Mir
10-2011	Ver 1.3	Entire	Removed Sediment Tox draft methods	Danielle Mir
11-2011	Ver 1.3	Entire	Updated pictures and figures	Danielle Mir
11-4-11	Ver 1.3	11	Added profile depth information, directions	Danielle Mir
11-10-11	Ver 1.3	Entire	Reformatted bullets and lists	Danielle Mir
02-11-09	Ver 8/2003	8 & 9	Put Chapter 9 as section in chapter 8	Danielle Mir
03-10-08	Ver 8/2003	Entire	Re-formatted entire document	Danielle Mir
03-10-08	Ver 8/2003	2	Updated and changed all calibration/ added QA guidance documents and current equipment type	Danielle Mir
03-10-08	Ver 8/2003	3	Changed parameter holding times & sample methods	Danielle Mir
03-10-08	Ver 8/2003	1	Added additional information on sampling types	Danielle Mir
03-10-08	Ver 8/2003	15	Removed Chapter- conductivity standards must be from certified lab	Danielle Mir
03-10-08	Ver 8/2003	5	Added methods for sediment toxicity samples	Danielle Mir
08-19-08	Ver 8/2003	2 & 11	Put chapter 11 as a section in chapter 2	Danielle Mir

12-10-08	Ver 8/2003	Entire	Changed all Figure numbers	Danielle Mir

I. CONSIDERATIONS FOR WATER QUALITY SAMPLING

The purpose for collecting water samples is to obtain a representative portion of the material or medium being evaluated. Valid results depend upon:

- Ensuring that the sample obtained is a true representative of the material or medium being evaluated;
- Employing proper sampling, handling, and preservation techniques;
- Properly identifying the collected samples and documenting their collection in permanent field records;
- Maintaining sample chain-of-custody procedures, if necessary;
- Protecting the collected samples by properly packing and transporting (shipping) them to the appropriate laboratory for analysis.

1. GENERAL WATER QUALITY SAMPLING CONSIDERATIONS

The following factors and procedures shall be considered and/or implemented in planning and conducting all water quality sampling operations. All of these factors and procedures should be considered in view of the specific objectives and scope of each individual field investigation. It is advisable to discuss sampling with the DWR Chemistry Lab during the planning process to verify and coordinate methodologies, analytical capabilities and timing of sample submittal.

1.1. Selection of parameters to be measured

The parameters to be measured are usually dictated by the purpose of an investigation and should be selected based upon required monitoring conditions (NPDES permits for example) or upon the investigator's knowledge of the problem.

1.2. Dissolved and particulate sample fractions

A sample is generally composed of dissolved and particulate fractions. When it is necessary to analyze samples for individual fractions, it is necessary to filter the sample in the field (i.e. dissolved phosphorous).

1.3. Required sample volumes

The volume of sample obtained should be sufficient to perform all the required analyses with an additional amount collected to provide for any quality control needs such as split samples or repeat examinations. DWR Laboratory sample submitting guidance document can be found at: http://portal.ncdenr.org/c/document_library/get_file?uuid=92a278e5-f75a-4e42-9be5-282ac0216b2a&groupId=38364.

1.4. Sample handling

After collection, all samples should be handled as little as possible. All personnel should use extreme care to ensure that samples are not contaminated. If samples are placed in an ice chest, personnel should ensure that the ice does not submerge the sample containers, thereby preventing cross-contamination. This is extremely important, especially if the samples are to be used in an enforcement action. Alternatives that can be used to prevent contamination include the use of frozen water

containers instead of ice or double wrapping the sample containers in trash bags surrounded with ice.

1.5. Special precautions for sampling trace amounts of contaminants

Most contaminant compounds are detected in the range of parts per billion or parts per trillion; therefore, extreme care must be taken to prevent contamination of samples. The following precautions shall be taken when trace contaminants are of concern:

1.5.1. When sampling surface waters, the aqueous sample should always be collected prior to any sediment sample collection. Sample collection should always be performed using cleaned equipment and proper collection technique.

1.5.2. Sample collection activities should proceed progressively from the least contaminated area to the most contaminated area (if this fact is known).

1.5.3. When possible, samples should be collected facing upstream to avoid contamination from sampling activities.

1.6. Procedures for identifying potentially hazardous samples.

1.6.1. Samples that are either **known** or **thought** to be **hazardous** should be identified **clearly** on both the sample tag and field sample sheet.

1.6.2. Information explaining the hazard, i.e., corrosive, flammable, poison, etc., shall also be listed.

1.6.3. If a sampling hazard is identified, only continue if a properly trained staff member is present and if appropriate safety equipment are available.

1.6.4. Follow procedures found on the ESS Fish Kill web page when sampling fish kill events: http://portal.ncdenr.org/web/wq/ess/fishkills

1.7. Collection of auxiliary data

All auxiliary data, such as flow measurements, photographs of sampling sites, meteorological conditions, and other observations, shall be entered into field records at the time samples are collected.

1.8. Time records

All records of time shall be kept utilizing local time in the military (2400 hour) time format and shall be recorded to the nearest five (5) minutes unless more precise measurements are dictated.

1.9. Transporting and shipping of samples

Samples may be hand delivered to the appropriate laboratory, or they may be shipped by common carrier. Chain of custody may be necessary during and after sample collection (Chapter II.3). All personnel must be aware that certain samples could be classified as hazardous materials and as such, could be regulated by the U.S. Department of Transportation under the Transportation Safety Act of l974. These regulations are contained in Title 49, CFR, Parts ll0-ll9 (An example would be concentrated acid, azide, etc.). A copy of these regulations is available online at: http://www.gpoaccess.gov/cfr/index.html.

2. SURFACE WATER SAMPLE SITE SELECTION

Selection of a surface water sampling location for water quality studies is based on many factors. These include but are not limited to, study objective, water use, point source discharges, non point source discharges, tributaries, changes in stream characteristics, types of stream bed, stream depth, turbulence, presence of structures (weirs, dams), accessibility, safety concerns, and personnel. When such sampling locations are located in estuarine systems, tidal effects must be considered when determining sampling locations.

Before sampling is conducted, a site assessment should be conducted to locate suitable sampling locations. Bridges and piers are normally good choices as they provide ready access and permit water sampling at any point across the width of the water body. When sampling from bridges, samples should be taken from the upstream side; however, this may alter the nature of water flow and cause sediment deposition. Additionally, bridges and piers are not always located in desirable locations with reference to waste sources, tributaries, etc. Wading for water samples is not recommended in lakes, ponds, and slow-moving rivers and streams. However, when wading for sample collections in slow-moving water bodies, it is best to work from downstream stations to upstream sampling points, especially when samples are taken in close proximity. In slow-moving or deep water, a boat is usually required for sample collections and sampling should allow for the possible presence of stratification.

3. SAMPLE COLLECTION TYPES

3.1. Grab sample

A grab sample is a sample collected over a period of time not exceeding 15 minutes. A grab sample is normally associated with water or wastewater sampling. However, soil, sediment, liquid hazardous waste samples, etc., may also be considered grab samples; no particular time limit would apply for the collection of such samples. These samples are used to characterize the medium at a particular point in time; and are generally associated with instantaneous water or wastewater flow data.

3.1.1. *Conditions when a grab sample is conducted*

a. Water or wastewater stream is not continuous (e.g., batch discharges or intermittent flow);
b. Characteristics of the water or waste stream are known to be constant;
c. Sample is to be analyzed for parameters whose characteristics are likely to change significantly with time (i.e., dissolved gases, bacteria, etc.);
d. Sample is to be collected for analysis of a parameter such as oil and grease where the compositing process could significantly affect the observed concentrations;
e. Data on maximum/minimum concentration are desired for a continuous water or wastewater stream;
f. When NPDES permit effluent monitoring specifies grab collections.

3.1.2 *Grab sample collection methods*

A grab sampler is collected at 0.15 m below the water surface. Gloves should be warn for personal safety and to prevent sample contamination.

a. Direct- A sample bottle is placed 0.15 m below the water surface while pointing the bottle mouth up current or towards the bow of a boat when lake sampling.

b. Intermediate Grab Sampling Device- These devices are any type of sampling device that holds the sample prior to pouring it into a sample bottle, and are used when sampling from a bridge or area that the water cannot be reached. The collection end is placed 0.15 m below the waterôs surface with the open end facing upstream or up current. An example is a Polyethylene Dipper (Figure 1) or other custom-made devices.

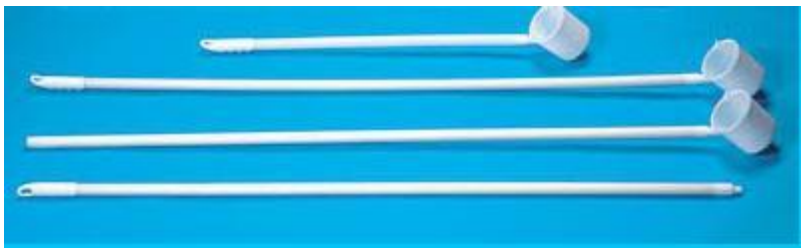

Figure 1. Polyethylene dipper typically used by DWR.

3.1.3. *Parameters that are always grab samples:*

metals	phenol
sulfide	oil and grease
volatile organics	bacterial
chlorine residual	other dissolved gases

3.2. Composite sample

Composite samples are used when average concentrations are of interest and are associated with average flow data (where appropriate). Composite sampling is employed when the water or wastewater stream is continuous or it is necessary to calculate mass/unit time loadings or when analytical capabilities are limited.

3.2.1. *Timed integrated*

A timed integrated composite sample contains discrete samples taken at equal time intervals over the compositing period. A timed composite may be collected continuously. A timed composite is collected continuously or with constant sample volume and a constant time interval between samples.

3.2.2. *Flow proportional integrated*

A flow proportional composite contains discrete samples, taken proportional to the flow rate over the compositing period. Proportional composites are collected with constant sample volume and constant time interval between samples proportional to stream flow.

3.2.3 *Area Integrated*

Area integrated composite samples are collected over a predetermined area of a waterbody, usually from the same depth. Samples are collected then composited into one representative sample.

3.2.4. *Vertical spatial composite (Photic Zone Sampling)*

Vertical spatial samples are composite samples (a.k.a. photo zone, depth-intergraded samples) taken within the photic zone. The photic zone is found between the surface and twice the secchi reading (Chapter III.6). Samples are collected by lowering and raising an integrated depth sampling device such as a Labline water sampler (Figure 2) at a steady speed to obtain a representative water sample within the photic zone. Prior to sampling, the Labline should be rinsed 3 times with station water to avoid sample contamination.

Figure 2. Labline sampler for photic zone (vertical spatial) composites.

3.3. Split sample

A split sample is a sample that has been portioned into two or more containers from a single collection device. Portioning assumes adequate mixing to assure the split samples are, for all practical purposes, identical. Devices such as churn splitters should be rinsed with ambient site water prior to field use for composite split samples, cleaned with phosphorous free cleaner after use and rinsed with deionized water before storage. Composite sample volume in the splitter should allow for ¾ of the total aliquot to be split with ¼ remainder. This prevents aeration of the sample during dispensing. Sample agitation should be performed for 2 minutes prior to sample split to ensure homogeneity of the composite. The spigot or valve should be purged prior to dispensing the first sample. As the composite volume in the churn is reduced, churning rate should increase.

3.4. Duplicate sample

Duplicate samples are collected simultaneously from the same source, under identical conditions but in separate containers.

3.5. Control sample

A control sample is collected upstream or upgrade from a source or site to isolate the effects of the source or site on the particular ambient medium being evaluated according to the study plan for that particular project.

3.6. Background sample

A background sample is collected from an area, water body, or site similar to the one being studied but located in an area known or thought to be free from the pollutants of concern. Background samples should be taken from well-mixed areas, not necessarily midstream to represent normal conditions.

3.7. Sample aliquot

A sample aliquot is a portion of a sample that is representative of the entire sample.

3.8. Scoop sample

A scoop sample is one that is taken in a non-quantitative way for identification only, such as a surface skim, a filamentous clump or rock scrape. All aquatic macrophyte samples are taken as scoop samples.

3.9. Physical Water Quality Measurements (In-Situ Field Measurements)

Physical parameter measurements recorded by a field meter such as a Hydrolab or YSI. Parameters that are considered physical water quality samples or parameters are:

Depth (m)	Temperature (°C)
Salinity (ppt)	Conductivity (us)
pH	Dissolved Oxygen (mg/L)

These may be measured at various depths depending on the water body and needs of the study being performed.

4. AUTOMATIC SAMPLERS

The Instrumentation Specialties Company (ISCO) model 2700 (Figure 3) and model 3700 wastewater samplers are portable devices designed to collect up to 24 separate sequential samples or can be programmed for composite sampling.

More complex sampling such as multiplexing, storm spaced sampling, interfacing with a variety of equipment such as flow meters, field printers, and lap top computers can also be accomplished with the 3700 model. Both sampler models must be supplied with 12 VDC power from one of four sources: an ISCO AC power pack, an ISCO nickel-cadmium battery pack, an ISCO sealed lead acid battery, or an external 12 V direct current source (such as an automotive or marine battery).

Refer to the ISCO 2700 and 3700 instruction manuals for detailed description of operating procedures. **It is important to verify the configuration of these samplers prior to placing them in the field** (Instrument Specialties Company 1988, 1991).

Figure 3. ISCO automated samplers.

I. CONSIDERATIONS FOR WATER QUALITY SAMPLING

5. MANUAL SAMPLING

Manual sampling is usually employed when collecting grab samples and immediate *in-situ* field analyses samples. However, it may also be used, in lieu of automatic - equipment, over extended periods of time for composite sampling.

5.1. Manual Sampling Technique:

The best method to manually collect a sample is to use the actual sample container. This eliminates the possibility of contaminating the sample with an intermediate collection container. **The actual sample container must always be used for collecting oil and grease and bacterial samples**.

5.1.1. If the water or wastewater stream cannot be physically reached, an approved intermediate sampling device may be used. Approved intermediate sampling devices include Labline samplers or Van Dorn type samplers. When a sample collected needs to be collected in the sample container such as grease or oil, a cage sampler can be used of the out-of-reach locations (Figure 4).

Figure 4. Cage sampler used in the DWR Ambient Monitoring Program

5.1.2. Collect the sample by lowering a properly cleaned collection vessel (bottle or intermediate sampling devise) into the water or wastewater stream. If an intermediate sampling device is used, the container employed to collect the initial sample must be rinsed three times with sample water and must be constructed of a material that meets requirements of the parameter(s) being investigated. The collection vessel may be lowered by hand or attached to a pole or rope and then lowered into the stream.

5.1.3. Some types of analyses require the use of a pump when sampling. If a pump is used, it is imperative that it be pre-purged and all components of the pump that come into contact with the liquid be properly cleaned to ensure the integrity of the sample.

5.1.4. Tip the collection container into the water or wastewater stream so that the mouth of the container faces upstream.

5.1.5. Rinse out the container via this procedure at least twice before the sample is collected (exceptions to this rinsing procedure may exist if preservatives are present in the sampling container and for certain analyses such as oil and grease).

6. SPECIAL SAMPLE COLLECTION PROCEDURES

6.1. Priority pollutants

6.1.1. Priority pollutant detection limits are usually in the range of parts per billion, thus extreme care must be exercised to ensure sample integrity.

6.1.2. All containers, composite bottles, tubing, etc., used in priority pollutant sample collection should be cleaned as described in Chapter VI.

6.1.3. When possible, the sample should be collected directly into the appropriate sample container. If the material to be sampled cannot be physically reached, an intermediate collection device may be used. This device should be a Teflon, glass or stainless steel vessel or Teflon tubing via a peristaltic type pump. The device should be cleaned as described in Chapter VI.

6.1.4. When an automatic sampler is employed for priority pollutant collection, the procedures described in Chapter I concerning collection of organic and metal samples with automatic samplers should be used.

6.2. Bacterial sampling

Samples for bacterial analysis should always be collected directly into the prepared glass or plastic sample container. Everything possible must be done to avoid contamination through physical contact with the inside of the cap or bottle and mouth of the bottle.

6.2.1. Hold the bottle near the base.

6.2.2. With cap still on, plunge the bottle, neck downward, below the surface and turn until the neck points slightly upward. The mouth should be directed toward the current.

6.2.3. Uncap the bottle and fill to within one inch of the top without rinsing

6.2.4. Recap immediately while underwater.

6.3. Immiscible liquids/oil and grease

Oil and grease may be present in wastewater as a surface film, an emulsion, a solution, or as a combination of these forms. **The designated sample container must always be used for collecting oil and grease samples.**

As it is very difficult to collect a representative oil and grease sample, the inspector must carefully evaluate the location of the sampling point. The most desirable sampling location is the point where greatest mixing occurs. Quiescent areas should be avoided. Because losses of oil and grease will occur onto the sampling equipment, the collection of a composite sample is impractical. Individual portions collected at prescribed time intervals must be analyzed separately to obtain the average concentrations over an extended period.

6.4. Volatile Organics Analyses (VOA)

Samples to be analyzed for volatile organics should be stored in the appropriate vials to prevent contamination and loss of sample. To verify proper sample container requirements, consult the DWR Chemistry Laboratory website (http://portal.ncdenr.org/web/wq/lab/staffinfo). The current methodology calls for 40 ml screw cap septum vials with a Teflon-silicone disk in the cap. The disks should be placed in the caps (Teflon side down) in the laboratory prior to the initiation of the sampling activities. Extra disks should be carried during field sampling in case of loss of the disks previously placed in the caps.

When there is no chlorine present in the sampled waterbody a 40ml VOA vial pre-preserved with 1:1 HCL by the Central Laboratory should be used for collection. A VOA sample should be preserved with ascorbic acid and 1:1 HCL whenever there is chlorine present or if it is not known if chlorine is present. Chapter 4 section 3.3.2 describes collection method used.

7. WASTEWATER SAMPLING

7.1 General considerations

Important procedures for obtaining a representative wastewater sample include:

a. Collecting the sample at a location where the wastewater is mixed. Therefore, the sample should be collected near the center of the flow channel, at a depth between 0.4 - 0.6 m total depth, where the turbulence is at a maximum and the possibility of solids settling is minimized. Skimming the water surface or dragging the channel bottom should be avoided.

b. Doing cross-sectional sampling when sampling from wide conduits or within a mixing zone. Dye may be used as an aid in determining the most representative sampling point(s).

c. If manually compositing a sample, thoroughly mix individual samples before pouring the individual aliquots into the composite container.

7.1.1. Site selection

Where applicable, wastewater samples should be collected at the location specified in the NPDES permit.

a. Influent - Influent wastewaters are preferably sampled at points of highly turbulent flow in order to ensure adequate mixing.

b. Effluent - Effluent samples should be collected at the site specified in the permit, or if no site is specified, below all treatment units including post aeration.

c. Pond and lagoon sampling - Generally, composite samples should be employed for the collection of wastewater samples from ponds and lagoons. Even if the ponds and lagoons have a long retention time, composite sampling is necessary because of the tendency of ponds and lagoons to short circuit. However, if dye studies or past experience indicate a homogenous discharge, a grab sample may be taken as representative of the waste stream; but in all cases, sampling should be consistent with permit requirements.

7.1.2 Sampling techniques

All techniques are covered in Section IV ISB Standard Operating Procedures and in the NPDES Compliance Sampling Inspection Manual.
http://www.epa.gov/compliance/resources/publications/monitoring/cwa/inspections/npdesinspect/npdesmanual.html

II. FIELD MONITORING

1. DATA SHEETS

There are two types of sheets needed for sample collection. A Field Sheet is used to document sample location and field parameters such as dissolved oxygen, temperature, pH, and secchi depth. A Lab Form is used to submit a sample(s) to the DWR Chemistry Laboratory.

1.1. Field Data Sheets (Figure 5)

These sheets have spaces for the information that identifies the station (station number, station name, date, and comments), sampler, lake observation (wind direction, rain, percent as well as providing spaces for conducting a depth profile by parameter. Data sheets can be found with the project manager (*i.e*. Ambient Lakes Coordinator).

a. Use a pen to mark on the sheets. Make sure that whatever is used is waterproof.
b. Write legibly and within the allotted space.
c. These forms are retained by the sampler for use in writing up the results or may be filed for later use.

1.2. Lab Sheets (Figure 6)

a. These forms are obtained by accessing the DWRôs Chemistry Lab website:
http://portal.ncdenr.org/web/wq/lab/staffinfo/samplesubmit/forms
b. A separate form is used for sediment, soil and tissue. Access the DWR Chemistry website to acquire the appropriate lab form. Contract labs will have their own; consult lab prior to sampling for any special requirements.
c. Lab sheets have spaces for all the information that identifies the station and sampler as well as boxes to check indicating the types of analyses to be conducted on the samples from the station.
d. The sample number used on the tags should be entered into the matching Lab Sheet. There is only one sample number per station ï it should be recorded on the Lab Sheet and all the samples related to that Lab Sheet. There is only one lab sheet per station.
e. Be sure to secure lab sheets(s) in a watertight container before shipping.
f. After analysis is complete and the information is transcribed to the lab sheet, it will be returned to the sampler.

WATERBODY: _____

COLLECTORS: _____

METER USED : _____
(Sonde SN)

WEATHER CONDITIONS:

Wind Direction: _____

Air Temperature	Wind Velocity	Rainfall (last 48 hrs)
___ < 60°	___ < 10 mph	___ None
___ 60° - 75°	___ 10 - 20 mph	___ < 1/4 inch
___ 75° - 90°	___ > 20 mph	___ 1/4 - 1 inch
___ > 90		___ > 1 inch

STRATIFIED FIELD DATA

STATION NUMBER	DATE YY	DATE MM	DATE DD	TIME (24 hour)	DEPTH (meters) X.XX	TEMP. (°C) x.x	DO (mg/L) x.x	%DO Sat x.x	pH (s.u.) x.x	COND. (umhos/cm) x	SECCHI (meters) x.x	Cloud Cover %	COMMENTS (i.e., water color, algae bloom, sedimentation, lake level fish kill or distress, macriphytes, land disturbance , etc.)

rev.4 April 2, 2012

Figure 5. Stratified Field Data Sheet

DIVISION OF WATER RESOURCES

Surface Water Fieldsheet

COUNTY :
RIVER BASIN :

REPORT TO : _____ Regional Office

Other :
COLLECTOR(S) :

PRIORITY
☐ AMBIENT ☐ QA
☐ COMPLIANCE ☐ CHAIN OF CUSTODY
☐ EMERGENCY VISIT ID

SAMPLE TYPE
☐ STREAM ☐ EFFLUENT
☐ LAKE ☐ INFLUENT
☐ ESTUARY

Lab Number :
Date Received :
Time Received :
Received By :

Data Released :
Date Reported :

Estimated BOD Range:

Station Location: _____

Seed: ____ Chlorinated: ____

Remarks: _____

Station #/Location Code	Date Begin (yy/mm/dd)	Date End (yy/mm/dd)	Time Begin	Time End	Depth - DM, DB, DBM	Value Type - A, H, L	Composite-T, S, B	Sample Type

	BOD 310	mg/L		Chloride 940	mg/L		NH3 as N 610	mg/L		Li-Lithium 1132	ug/L
	COD High 340	mg/L					TKN as N 625	mg/L		Mg- Magnesium 927	mg/L
	COD Low 335	mg/L		Chlorophyll a EPA 445.0 modified option	ug/L		NO2 plus NO3 as N 630	mg/L		Mn-Manganese 1055	ug/L
	Coliform: MF Fecal 31616	/100ml					P: Total as P 665	mg/L		Na- Sodium 929	mg/L
	Coliform: MF Total 31504	/100ml		Color: True 80	c.u.		PO4 as P 70507	mg/L		Arsenic: Total 1002	ug/L
	Coliform: tube Fecal 31615	/100ml		Color: (pH) 83 pH=	c.u.		P: Dissolved as P 666	mg/L		Se- Selenium 1147	ug/L
	Coliform: Fecal Strep 31673	/100ml		Color: pH 7.6 82	c.u.		K-Potassium	mg/L		Hg- Mercury 71900	ug/L
	Residue: Total 500	mg/L		Cyanide 720	mg/L		Cd- Cadmium 1027	ug/L		Ba-Barium	ug/L
	Volatile 505	mg/L		Fluoride 951	mg/L		Cr-Chromium: Total 1034	ug/L		Organochlorine Pesticides	
	Fixed 510	mg/L		Formaldehyde 71880	mg/L		Cu- Copper 1042	ug/L		Organophosphorus Pesticides	
	Residue: Suspended 530	mg/L		Grease and Oils 556	mg/L		Ni-Nickel 1067	ug/L		Organonitrogen Pesticides	
	Volatile 535	mg/L		Hardness Total 900	mg/L		Pb- Lead 1051	ug/L		Acid Herbicides	
	Fixed 540	mg/L		Specific Cond. 95	umhos/cm		Zn- Zinc 1092	ug/L			
	pH 403	units		MBAS 38260	mg/L		V-Vanadium	ug/L		Base/Neutral&Acid Extractable Organics	
	Acidity to pH 4.5 436	mg/L		Phenols 32730	ug/L		Ag- Silver 1077	ug/L		TPH Diesel Range	
	Acidity to pH 8.3 435	mg/L		Sulfate 945	mg/L		Al- Aluminum 1105	ug/L			
	Alkalinity to pH 8.3 415	mg/L		Sulfide 745	mg/L		Be- Beryllium 1012	ug/L		Purgeable Organics (VOA bottle req'd)	
	Alkalinity to pH 4.5 410	mg/L		Boron			Ca- Calcium 916	mg/L		TPH Gasoline Range	
	TOC 680	mg/L		Tannin & Lignin	ug/L		Co- Cobalt 1037	ug/L		TPH/BTEX Gasoline Range	
	Turbidity 76	NTU		Hexavalent Chromium	ug/L		Fe- Iron 1045	ug/L		Phytoplankton	
	Coliform Total Tube	/100 ml		Bicarbonate	mg/L		Mo-Molybdenum	ug/L			
				Carbonate	mg/L		Sb-Antimony	ug/L			
				Total Dissolved Solids	mg/L		Sn-Tin	ug/L			
							Tl-Thallium	ug/L			
							Ti-Titanium	ug/L			
							Hg-1631	ng/L		Temperature on arrival (°C):	

COMMENTS : _____

Revision: 08/23/13

Figure 6. Surface Water Lab Sheet

2. SAMPLE TAGS

A sample tag is used for most samples returned to the laboratory for analysis (Figure 7). These tags are usually attached to the sample container by a rubber band. In some cases, particularly with biological samples, the sample tag may be included with or wrapped around the sample. Sample tags should be of material that is waterproof and should be written on with indelible ink. It is very important that these tags are legible.

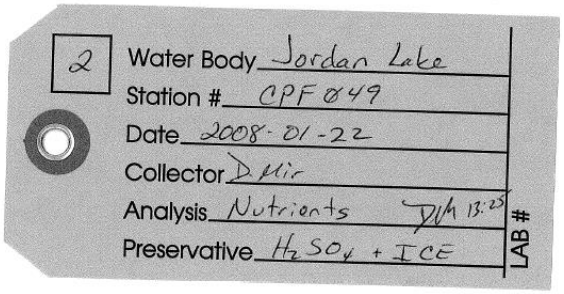

Figure 7. Completed Sample Tag

2.1 Information included on a sample tag:

- Sample number - determined based on number of stations to be sampled that day ï All samples from a station will have the same sample number. Figure 7 shows the sample number for the tag as 2.
- Water Body
- Station number
- Date(s) & time(s)
- Name of the person collecting the sample
- Types of analyses to be conducted (such as Nutrients)
- Types of preservatives used
- Sampler initial after preserving with acid

2.2 Responsibility of project leader or field investigator

The project leader or field investigator assigns the station number to be used for that location. If previous sampling has occurred at a site, that station number should be used again. This number is ordinarily a numeric code, designed for a particular study, inspection, or investigation. Ambient stations have a special numbering system. New ambient stations are identified by the Ambient Monitoring Coordinator.

The project leader or field investigator must exercise due caution to ensure that duplicate station numbers are not used during the same study. The project leader or field investigator will also always specify the type of sample collected since the same station number is used when a water and sediment sample is collected at the same location. The exact description of all stations associated with field identification or sample station numbers is documented on the field sheet.

If a sample is split with a facility, state regulatory agency, or other party, sample tags with identical information are to be attached to each of the sample containers; the facility, state regulatory agency, etc., tag shall be marked facility (actual name), state regulatory agency (actual name), etc.

3. CHAIN-OF-CUSTODY PROCEDURES

This procedure is used for samples collected as part of an investigation for legal proceedings or where it is required under the study plan. The possession of samples or other evidence shall be traceable from the time the samples are collected until they are introduced as evidence in legal proceedings.

3.1. Sample Custody

A sample or other physical evidence is under custody if it is in:

- The field investigator's actual possession, or
- The field investigator's view, after being in his/her physical possession, or
- The field investigator's physical possession and then he/she secures it to prevent tampering, or
- A designated secure area.

To simplify the chain-of-custody record and eliminate future litigation problems, as few people as possible should handle the samples or physical evidence. The field investigator is responsible for the care and custody of the samples collected until they are properly transferred to another person or facility.

3.2 Field Custody Procedures

3.2.1 *Security Seal*

a. Complete sample tags for each sample.
b. Place the lab sheets and chain of custody sheets in a Zip-loc bag and place in a cooler along with the samples.
c. Seal the coolers with filament tape and a DWR custody seal similar to the one shown in Figure 8.
d. The field investigator writes the date and their name on the seal. This requirement shall be waived if the field investigator keeps the samples in his custody from the time of collection until they are delivered to the laboratory.

3.2.2 *Chain of Custody Form*

a. Record all samples on the field form or in field logbooks and using the Chain of Custody Record (Figure 9.) available from the DWR Chemistry Lab:

   http://portal.ncdenr.org/web/wq/lab/staffinfo/samplesubmit/forms

b. For documents received during investigations, place them in large envelopes, seal with a DWR seal such that the envelopes cannot be open without breaking the seal and note the contents on the envelope. If at any time the DWR seal is broken, that fact and the reason should be noted on the chain-of-custody record and a new seal affixed. The information on the seal should include the field investigator's signature, as well as the date and time of sealing.
c. Place other physical evidence such as videotapes or other small items in zip-lock bags and affix a DWR seal so that the bag cannot be opened without breaking the seal. A chain-of-custody record should be kept with the items in the bag. Any time the seal is broken, note reason on the chain of custody record and affix a new seal.

d. Personnel shall not accept samples from other sources unless the sample collection procedures used are known to be legally defensible, can be documented, and the sample chain-of-custody can be established. If such samples are accepted, a sample tag and a DWR form, containing all relevant information and the chain-of-custody record, shall be completed for each sample.

Figure 8. DWR Chain of Custody Security Seal

Report to: _____ **SURFACE WATER SECTION** **Page** _____ **of** _____

CHAIN OF CUSTODY (COC) RECORD

NC DENR/DWR LABORATORY (check one): [ ] CENTRAL [ ] ARO

For Investigation of:

Sample collector (print name)
and DM-1 forms completed by: _____ Sample collector's signature: _____

Field storage conditions and location (when applicable):

Lab Use Only LAB NO.	STATION NO.	STATION LOCATION	DATE SAMPLED	TIME SAMPLED	NUMBER OF CONTAINERS

Relinquished by (signature):	Date	Time	Received by (signature):	Date	Time
Relinquished by (signature):	Date	Time	Received by (signature):	Date	Time
Relinquished by (signature):	Date	Time	Received by (signature):	Date	Time

Method of Shipment (circle one): State Courier Hand-delivered Federal Express UPS Other: _____

Security Type and Conditions:	Sealed by:	Broken by:

INTRALABORATORY CHAIN OF CUSTODY - Lab Use Only

LAB NUMBERS FROM	LAB NUMBERS THROUGH	NUMBER BOTTLES	ANALYSES REQUESTED	RELINQUISHED BY:	RECEIVED BY:	DATE	TIME

QA\Forms\Sample Receiving\COC form WR 4/10/01dbs Revised: 8/23/2013 ntg

Figure 9. Surface Water Section Chain of Custody Form

3.3 Transfer of Custody and Shipment

3.3.1. When transferring the possession of chain of custody samples, the individuals receiving the samples shall sign, date, and note the time that they received the samples on the field form or in the field log book. This action documents transfer of custody of samples from the field investigator to another person (e.g. to the laboratory).

3.3.2. After properly packing samples for shipment to the appropriate laboratory for analysis, secure the shipping containers using nylon strapping tape and custody seals. The seal shall be placed under the point on the tape where the ends are located and wrapped over the top of it. The seal shall be signed, dated, and the time recorded by the field investigator.

3.3.3. Samples split with a facility, state regulatory agency, or other government agency must be signed for on the Chain of Custody Form by the facility, state regulatory agency, or other government agency representative receiving the samples.

3.3.4. All samples shipped shall be accompanied by the DWR chain-of-custody form(s). The original and one copy of the form will be placed in a plastic bag inside the secured shipping container. One copy of the form will be retained by the field investigator or project leader. The original of the form will be transmitted to the field investigator or project leader after samples are accepted by the laboratory.

3.3.5. If sent by mail, the package shall be registered with return receipt requested. If sent by common carrier, a government bill of lading or air bill should be used. Receipts from post offices, copies of bills of lading, and air bills shall be retained as part of the documentation of the chain-of-custody.

4. FIELD INSTRUMENTS

Intensive Survey Branch uses a wide array of instrumentation for recording in-situ water quality parameters. Currently, Hydrolab (Hach Environmental) and YSI (Yellow Springs Instrument Co.) are the main manufacturersô used. Instructions for use, calibration, and maintenance as written by the manufacturer should always be followed. Manufacturersômanuals for all meters can be found in the ESS Calibration Lab. DWR produced a guidance sheet that outlines basic calibration, maintenance, and acceptance criteria for meters commonly used by DWR (Appendices 1-4). **All meter guidelines and guidance sheets found in this document are supplementary to and not a replacement for the manufacturer's directions.**

4.1. All field meters should be calibrated before and checked after sampling activities daily. Calibration data should be documented on a Water Quality Monitoring Field Meter Calibration Sheet (Figure 10).

4.2 In-*situ* field parameter measurements

4.2.1. *Parameters typically measured:*

a. **Conductivity** (εS/cm @ 25 °C)
b. **Dissolved Oxygen** (DO- mg/L)
c. **pH** (Standard Units)
d. **Temperature** (°C)
e. Light Attenuation (εE/m^2/s)

Additional Calibrations and Use of multiparameter Meters

4.2.2. *Battery Voltage*

a. Use the correct battery source for the particular instrument in use.
b. Battery voltage must be in an acceptable range before calibrating and using the meter (see respective manual).
c. Record both initial and terminal battery voltage on the Meter Calibration sheet (Figure 10).

4.2.3. *Depth*

a. Some meters can be calibrated to read depth by entering the number zero on the keypad while the sonde sensors are at the surface during field measurements.
b. Record all field depth measurements to the nearest tenth of a meter (if needed).

4.3 Calibrated Backup Field Meters

Although meters are maintained, failure can occur at anytime. Calibrated backup meters, meter manuals, batteries and calibration buffers/ standards are required during sampling. Inability to collect data due to a meter failure is unacceptable. See Appendices 1 ï 4 for detailed guidance on using, maintaining, and storage field meters commonly used by DWR.

Water Quality Monitoring Field Meter Calibration Sheet

Collector(s):

Study:

Sampling Location:

Meter Model:

Meter / Sonde Serial No:

	Date yy/mm/dd	Time 24hr hh:mm	Initials
Pre-Sampling Calibration			
Post-Sampling Check			

Barometer Calibration (mmHg)
**YSI Pro Plus Meters Only*

Initial Reading	Calibrated Value

Miscellaneous (Does not apply to YSI or Accumet Meters)

	Battery Level (V)		Stirrer Working?
Pre-Sampling Calibration			Y / N
Post-Sampling Check			Y / N

Battery Ranges = Surveyor: internal- 7.2-7.5V, external- 11-13V; Quanta: 4.0-4.5V

Dissolved Oxygen (mg/L)

	Temp. °C	Initial % Saturation	Barometric Pressure (mmHg)	Altitude (ft.)	D.O. Table Value	Initial Meter Reading (mg/L)	Calibrated Meter Reading (mg/L)	Calibrated % Saturation
Pre-Sampling Calibration								
Post-Sampling Check								
					Within ± 0.5 ? Y / N			

Specific Conductance (µS/cm at 25ºC)

	Dry Air [1,2] Zero (0)		Lot #: Conductivity Standard [3] Value: _____		Lot #: Calibration Check Value: _____
	Initial Meter Reading	Calibrated [4] Meter Reading	Initial Meter Reading	Calibrated [4] Meter Reading	Initial Meter Reading
Pre-Sampling Calibration					
Post-Sampling Check	Within ± 2? Y / N		Within ±10%? Y / N		Within ±10% Y / N

±10% Ranges for Sp. Cond.

Standard	Range
100	90 to 110
500	450 to 550
1,000	900 to 1,100
10,000	9,000 to 11,000
15,000	13,500 to 16,500
50,000	45,000 to 55,000

NOTE: Quanta reads in mS/cm; move decimal 3 places right for µS/cm.

[1] Dry Air CALIBRATIONS are conducted for 4a and MS5 Hydrolabs only.

[2] Dry Air CHECKS (confirmation of zero in dry air) are conducted for YSI 85, YSI 6920, YSI Pro Plus & Quanta meters.

[3] Conductivity standards are used to CHECK the YSI 85 meter and to CALIBRATE all Hydrolab meters and the YSI 6920 & YSI Pro Plus.

[4] Does not apply to Dry Air CHECKS or Conductivity Standard CHECKS (leave blank).

pH (SU)

	Lot #: Buffer #1 7.0 Buffer Temp: _____		Lot #: Buffer #2 4.0 / 10.0 Buffer Temp: _____		Slope Efficiency[5]	Confirmation Buffer 7.0
	Initial Meter Reading	Calibrated Meter Reading	Initial Meter Reading	Calibrated Meter Reading		Meter Reading
Pre-Sampling Calibration						
Post-Sampling Check	Within ± 0.2? Y / N		Within ± 0.2? Y / N			Within ± 0.1? Y / N

[5] Slope efficiency applies to Accumet meters only (does not apply to Hydrolab or YSI meters).

Comments:

Keep original on file for 5 years

Ver. 06/05/2012

Figure 10. Meter Calibration Sheet

III. FIELD PARAMETER MEASUREMENTS

1. WATER TEMPERATURE

Temperature measurements are taken by a multiparameter meter (Hydrolab or YSI) or dial Celsius-thermometer or a thermister. Below are some general considerations while collecting water temperature data.

- The meter should have a scale marked for every 0.1°C.
- Make readings with the mulitparameter meter or thermometer in water long enough to permit equilibrium.
- Temperature sensors on the Hydrolab and YSI meters are factory set and do not require recalibration.
- At least once a year check the meter thermometer against a precision thermometer certified by the National Institute of Standards and Technology (NIST).
- Temperature readings must be record as degrees Centigrade (°C) to the nearest tenth of a degree. During field use, the temperature readings should always be read when they are stable and before the other parameters are read to ensure stable readings for all parameters.

2. AIR TEMPERATURE

Refer to previous procedure, except measure the ambient air temperature above the water surface to be sampled. Do not use immersion thermometers to measure air temperature.

3. DISSOLVED OXYGEN

Dissolved oxygen analysis measures the amount of gaseous oxygen dissolved in an aqueous solution. Dissolved oxygen may be measured by electrometric methods (e.g. Hydrolab or YSI) or by chemical methods (Winkler Method).

Testing must be done immediately at the sampling location, as a grab sample, which is why electrometric methods are favored.

See Appendices 1 ï 4 for detailed guidance on using, maintaining, and storage of meters and probes commonly used by DWR. **This SOP and the attached meter guidance sheets are supplementary to and not a replacement for the manufacturer's instructions manual.** Manufacturerôs operations manuals for all meters are kept in the ESS Calibration Lab.

3.1 Electrometric Method Calibration

All field meters should be calibrated before and checked after sampling activities (at least daily). The calibration data should be entered on a meter calibration sheet (Figure 10). Detailed guidance for calibrating dissolved oxygen is provided in Appendices 1 ï 4.

3.1.1 Acceptance Criteria For DO calibration

- Calibrated meters should be compared to the DO table to ensure calibration was done correctly.
- Appendix 5 describes the calculations needed to correct for elevation and a table used at sea-level.
- Dissolved oxygen concentrations need to be calibrated within 0.5mg/L of the elevation corrected table concentration for a given temperature.

3.2. Winkler Method - azide modification (Standard Methods, 18th edition)

The azide modification effectively removes interference caused by nitrite, which is the most common interference in biologically treated effluents and incubated BOD samples. The azide modification is not applicable under the following conditions:

- Samples containing sulfite, thiosulfate, polythionate, appreciable quantities of free chlorine or hypochlorite;
- Samples high in suspended solids;
- Samples containing organic substances which are readily oxidized in a highly alkaline solution or which are oxidized by free iodine in an acid solution;
- Untreated domestic sewage;
- Biological flocs;
- Where sample color interferes with endpoint detection.

In instances where the azide modification is not applicable, electrometric methods should be employed.

Below are some general considerations while collecting dissolved oxygen data using the Winkler Method:

- Collect surface water samples in narrow-mouth glass-stoppered BOD bottles of 300 ml capacity with tapered and pointed ground-glass stoppers and flared mouths. Once analysis is complete and the information is transcribed to the lab sheet, it will be returned to the sampler.
- Avoid entrapping or dissolving atmospheric oxygen. Do not allow the sample to remain in contact with air or be agitated, because either condition may result in a change to its gaseous content.

- Where samples are collected from shallow depths (less than 5 feet) use of an APHA-type sampler is recommended. Use of a Kemmerer type sampler is recommended for samples from depths greater than 5 feet. Bleed sample from bottom of samplers through a tube extending to the bottom of a BOD bottle. Fill bottle to overflowing.
- Record sample temperature to nearest degree Celsius or more precisely.
- Reagents
  - Manganous sulfate solution
  - Alkaline iodide-sodium azide solution.
  - Sulfuric acid (H_2SO_4) concentration
  - Sodium thiosulfate solution 0.025 N
  - Starch solution
- Analysis Steps:
  1. Add 2 mL of manganous sulfate solution to sample container by holding the tip of the pipette below the surface of the liquid.
  2. Add 2 mL of alkaline iodide-sodium azide solution by holding the tip of the pipette below the surface of the liquid.
  3. Replace BOD bottle stopper, avoid trapping air bubbles, and shake well by inversion.
  4. When the precipitate settles, leaving a clear supernatant above the manganese hydroxide floc, shake again.
  5. Allow floc to settle again, at least 200 mL of clear supernate should be above the floc.
  6. Remove the stopper and add 2 mL of concentrated sulfuric acid by holding the pipette above the surface of the liquid and allowing the acid to run down the neck of the bottle, re-stopper, and mix by inversion until no floc is visible.
  7. Withdraw 203 mL of the solution into an Erlenmeyer flask.
  8. Titrate with 0.025 N sodium thiosulfate solution to a pale straw color.
  9. Add 1 mL of starch solution and continue titration to the first disappearance of blue color.
  10. Record the # of mL of thiosulfate used; where 1 mL thiosulfate = 1 mg/L DO.

4. pH (ELECTROMETRIC METHOD)

4.1. Information on pH

4.1.1. *Precision and accuracy:* ±0.2 pH unit represents the limit of accuracy under normal conditions for measurements of water and poorly buffered solutions. For this reason, report pH values to the nearest 0.1 pH unit. Calibrate instrument within 0.2 pH units of the standard pH buffer value.

4.1.2. *Calibration Reagents* - Calibrate the electrode system against standard buffer solutions of known pH. Always use fresh commercially made buffers to calibrate field meters. Buffer solution and samples should be stored in polyethylene bottles. Never pour decanted or used buffer solution back into the original bottle.

4.1.3 *Procedure* - Always follow the manufacturer's instructions for pH meter storage and preparation of electrodes. Recommended short-term storage of electrodes varies with type of electrode and manufacturer. See Appendices 1 ï 4 for detailed guidance on using, maintaining, and storage of pH meters and probes commonly used by ISB. Never store probes in DI water; tap water or pH buffer 4.0 is preferred.

Note: All field meters should be calibrated before and checked after sampling activities daily. The calibration data should be entered on a meter calibration sheet (Figure 10).

4.2. Multiparameter YSI or Hydrolab Meters

The Hydrolab and YSI meters used by ISB all have the same basic method for calibration. A training outline for each meter used by ISB is listed in Appendices 1 - 4. Copies of the manufacturerôs instruction manual are located in the ISB calibration room.

4.3. Accumet AP Series (Fisher Scientific) Handheld pH Meters

The Accumet handheld pH meter is a stand-alone pH meter (it does not measure any other parameters beyond pH). See Appendix 2 for detailed guidance on using, maintaining, and storage of the Accumet AP61 pH meter which is typically used in conjunction with the YSI 85 meter. A copy of the manufacturerôs instruction manual is located in the ISB calibration room.

5. SPECIFIC CONDUCTIVITY/SALINITY

The specific conductance (conductivity) of a solution is a measure of its ability to carry an electrical current. This ability depends on the presence of ions, their total concentration, mobility, valence, and relative concentrations, and on the temperature of measurement. Specific conductance is the conductance afforded by 1 cc (ml) of a solution of electrolyte and is reported in micromhos per centimeter (µmhos/cm). Specific conductance measurements are used in water analysis to obtain a rapid estimate of the dissolved solids content of a water sample.

5.1 Specific Conductivity Meter Calibration - Detailed meter guidelines for calibrations are listed in Appendices 1 - 4. Copies of manufacturerôs instruction manuals are found in the ISB calibration room.

Note: All field meters should be calibrated before and checked after sampling activities daily. The calibration data should be entered on a meter calibration sheet (Figure 10).

5.2 Additional Calibration Information

- *Acceptance Criteria:* Calibrate instrument within ±10% of the calibration standardôs true value.
- Always calibrate with fresh, certified conductivity standards.

6. SECCHI DISK TRANSPARENCY

A measurement of water transparency obtained by observing a specially marked, circular disk which is lowered through the water column until it is not visible. This measure of the point at which the disk is non-visible is considered the secchi depth.

6.1. Secchi disk use (Figure 11)

6.1.1. *Conditions for secchi disk readings*

a. Shaded, protected side of boat.
b. Minimal waves or ripples, if possible.
c. Do not wear sunglasses while taking the secchi depth reading.

NOTE: Any departure from these conditions should be specifically stated on the field sheet.

6.1.2. *Method*

a. Rope should be accurately graduated in meters, 0.1 meter graduations for the first meter, 0.5 m graduations thereafter. At a minimum of annually verification of correct graduation is necessary as rope may stretch with continued use.
b. Observer's eye should be 1 meter above the water surface.
c. Lower the disk into the water to the depth at which the disk disappears.
d. Lift the disk and record the depth at which it just reappears.
e. Record the average reading from previous 2 steps on field sheet as Secchi depth reading to the nearest tenth of a meter.

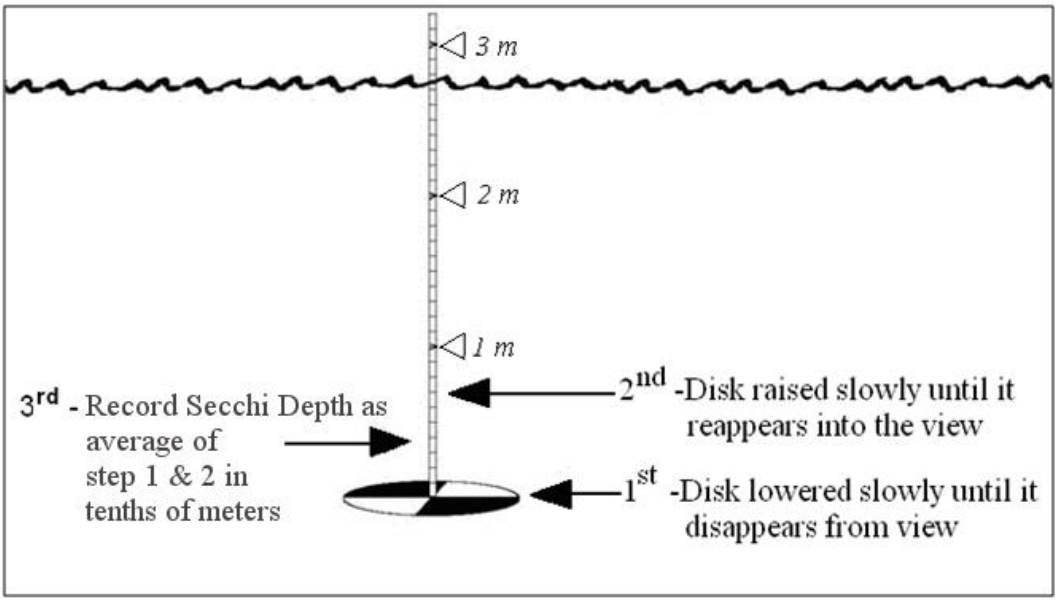

Figure 11. Secchi Disk

7. LIGHT ATTENUATION

The measurement of the decrease in light intensity through the water column as depth increases due to absorption and scattering effects of water molecules.

7.1. Light Attenuation is calculated by obtaining a vertical profile of light, using a PAR (photosynthetically active radiation) meter.

7.1.1. *PAR Meter Preparation*

a. Obtain an independent datalogger such as LI-COR LI-1400.

b. Connect a deck sensor and an underwater sensor to the LI-1400. Make sure the correct calibration factors are entered for each probe. All calibration factors are supplied by the manufacturer.

c. Place the deck sensor on the boat where it will not be shaded.

7.1.2. *Methods*

a. Lower the underwater sensor on the SUNNY, not shaded, side of the boat to a depth about 10 cm to represent the surface.

b. Once readings stabilize, record the values from both sensors (ε E/m^2/s), along with the water depth of the underwater sensor. Log the values in the datalogger.

c. Lower the underwater sensor to 0.5 m (6n), allow the values to stabilize and record the values from both sensors, along with the water depth of the underwater surface.

d. Repeat at the following schedule:
   - Shallow Sites (Ò2 m) ï Every 0.5 m interval;
   - Nominal depths (>2 <10 m) ï Every 0.5 m (near surface) and very 1 m interval to near bottom (0.5 m off-bottom)
   - Deep Sites (>10 m) ï 0.5 m (near-surface) and every 1 m interval to 10 m, than at 5 m intervals, thereafter, to near-bottom (0.5 m off-bottom)

   NOTE: Follow schedule, unless specified differently for the individual sampling project.

e. If the meter impacts the bottom, allow 2-3 minutes for the disturbed conditions to settle before take the reading.

f. If the light measurements become negative before reaching the bottom, terminate the profile readings at that depth.

8. REFERENCE POINT-TAPE-DOWN MEASUREMENT

Reference point-tape-down is a procedure for determining relative vertical distance between fixed bridge points and stage of a water body below the bridge structure.

8.1. Procedure for Reference Point-Tape-Down

a. Use a weight-tape gage consisting of a graduated (0.1 ft) steel tape to which is fastened a small cylindrical weight (dimwap) of known length.

b. Locate reference point (RP) as documented on the station location sheet. They are often located on the outer edge of bridge railings.

c. Measure by suspending the weight-tape from the reference point (measuring) to the water surface.

d. The reference point value is indicated by direct reading of the suspended tape where it intercepts the fixed reference point. Read from the top of the bevel if the reference point is beveled.

e. Record measurement and add on the length of the weight.

9. STAGE MEASUREMENTS

These procedures are for use at U.S. Geological Survey permanent stream gauging stations.

9.1 Obtaining Stage Measurements

Follow instructions in the USGS publication Stage Measurements at Gauging Stations, Book 3, Chapter A7, United States Department of the Interior, Geological Survey, 1968. http://pubs.usgs.gov/tm/tm3-a7/pdf/tm3-a7.pdf

9.1.1 *Prior to Sampling*

a. Obtain permission from the USGS district chief to read the stage measuring devices in the instrument shelters.
b. Obtain on the job training by USGS personnel as to how to read the stage measuring devices.

9.1.2 *Stage Measuring Devices*

- Staff gage
- Wire weight gage
- Electric-tape instrument
- Automatic digital recorder
- Graphic recorder (bubble meters)

IV. WATER SAMPLE COLLECTION AND PRESERVATION

1. BOTTLES AND PRESERVATION

Surface water, soil or sludge samples for submittal to the DWR Chemistry Laboratory (the Lab) must be collected using Lab and EPA approved containers, and in accordance with approved collection, preservation and holding times. The Lab maintains a website with links to the approved preservation and holding times for all parameters for which the laboratory analyzes: http://portal.ncdenr.org/c/document_library/get_file?uuid=719b475c-c4a7-44c7-86a7-1804bbd432c9&groupId=38364. Field staff are responsible for being familiar with the Labôs procedures and following them accordingly. Preservatives can be added by pipette or pre-measured vials depending on the sensitivity of parameter being measured. If a parameter is not on the Labôs website, speak with the appropriate lab staff to determine how to proceed. Any samples submitted to the Lab must be accompanied by a Lab Sheet (Chapter 2-Figure 6). Immediately after sampling, labeling, and chemical preservation, samples are placed in coolers on ice, along with a temperature blank. Once samples arrive at the laboratory, support staff check the temperature blank (included in each cooler) to ensure that they are in appropriate temperature range (4 ± 2ºC), assign lab tracking numbers, and distribute them to the appropriate analytical units. Any samples not meeting temperature, holding time, or preservation requirements or are otherwise not submitted in accordance with the SOP are subject to rejection as per Section 13: *Corrective Actions* of the Laboratory Section QAM. Laboratory staff will attempt to contact collector by phone or email before rejecting. If conditionally accepted, the laboratory will document the anomaly with a Sample Condition Upon Receipt (SCUR) and/or Sample Anomaly Report (SAR) form and include copies with the final analytical report. Results from anomalous samples will be reported using the appropriate qualification code(s).

2. COLLECTION METHODS FOR CONVENTIONAL PARAMETERS

Collection for majority of the conventional parameters can be done by the multiple methods introduced in Chapter 1, ñSample Collection Typesò The following section is an overview of the types of parameters collected by DWR along with the required sample size, bottle type, preservative method and holding time.

Note: There are some parameters that can ONLY be collected as a surface grab at 0.15 m below the surface and will be stated in the collection method statement. Although, holding times vary from hours to days, **all samples collected should be submitted to the laboratory as soon as possible.**

2.1. BOD 5 ï Day (Biochemical Oxygen Demand) - This test determines the amount of organic material in wastewater and surface waters by measuring the oxygen consumed by microorganisms in decomposing organic constituents. The test consists of the determination of dissolved oxygen prior to and following 5-day incubation of the sample at 20°C, thus establishing the amount of oxygen used.

2.1.1 *Collection method*:

a. Collect sample in a 1-liter plastic bottle.
b. Deliver within 48 hours
c. Cool to 4°C
d. For WWTP effluents, collect the sample ahead of disinfection when possible.

2.2. COD (Chemical Oxygen Demand) - measures pollution strength (Sawyer & McCarty, 1967). It is a measure of the amount of oxygen required to oxidize organic and oxidizable inorganic compounds in wastewater and surface waters.

2.2.1 *Collection method:*

a. Collect sample in a 200 ml plastic bottle
b. Acidified the sample with H_2SO_4 to pH <2.
c. Cool the sample to 4°C
d. There is a 28 day holding period.

2.3. Coliform - Fecal coliform bacteria are superior to total coliforms as indicators of possible pathogenic contamination of water. The total coliform group includes organisms, principally of the aerogenes group, that are not necessarily of fecal origin. The aerogenes may be a considerable portion of the total coliforms on occasion. They may have no sanitary significance since they can come from soils and vegetation especially grains. Essentially all fecal coliforms, on the other hand, are of fecal origin and therefore potentially are accompanied by pathogens.

2.3.1. *General Collection Methods*

a. Collect sample with a 250 ml wide-mouth sterile plastic bottle supplied by the DWR Laboratory. These bottles must contain sodium thiosulfate and EDTA reagents.
b. Coliform sample is always collected as a surface grab sample. In no case should composite samples be collected for microbiological examination.
c. Do not rinse bottle with sample, but fill it directly to within 1-2 inches from the top to allow mixing of the sample before analysis.
d. Use caution to avoid contaminating the sample with fingers, gloves, or other materials.
e. Cool to 4°C and return to lab **in less than 6 hours** from time of collection. The DWR Lab will analyze any coliform samples that are received in less than 24 hours; however, the data may not be acceptable for some uses due to extended holding time.

2.3.2. *Surface Sampling By-Hand*

a. Grab sample should be collected directly into the sample bottle.
b. Remove the bottle top to protect bottle and cap from contamination; avoid touching the inside of the bottle and cap.
c. Grasp the bottle securely near the base with one hand and plunge the bottle mouth down into the water to avoid surface scum. Position the bottle towards the current flow and away from the hand of the collector, the shore, the side of the sampling platform, or boat. The sampling depth should be 0.15m below the water surface.
d. If the water body is static, create an artificial current by moving the bottle away from the sampler while tipping the bottle slightly to allow water to enter.
e. Tip the bottle slightly upwards to allow air to exit and the bottle. Fill the bottle to within 1-2 inches of the top.
f. After removal of the bottle from the stream, tightly stopper and label the bottle.

2.3.3. *Surface Sampling by Weighted/Cage Bottle Frame* (Figure 4, pg. 19)

a. Remove the cover and lower the device to the water.
b. It is preferable to use nylon rope which does not absorb water and will not rot.
c. Swing the sampling device downstream and then allow it to drop into the water while pulling on the rope so as to direct the bottle upstream.
d. Pull the sample device rapidly upstream and out of the water, simulating the scooping motion of grab sampling.
e. Take care not to dislodge dirt or other material from the sampling platform.

2.4. Residue (Solids) - Residue refers to solid matter suspended or dissolved in water or wastewater.

2.4.1. *Residue Types*

a. Total Residue - is the term applied to the material left after evaporation of a water sample, and its subsequent drying in an oven at a defined temperature. Total residue includes nonfilterable residue and filterable residue. Also known as Total Solids.
b. Nonfilterable Residue (Suspended) - the portion of total residue retained by a filter. The concentration of other water quality parameters is related to suspended solids since the

solid structure may contain biochemical and chemical oxygen demand materials, trace metals, nutrients, pesticides, and toxic or hazardous materials absorbed on the surface. Also, known as Total Suspended Solids.

c. Filterable Residue (Dissolved) - the portion of total residue that passes through the filter. Dissolved solids consist mainly of inorganic salts, small amounts of organic matter, and dissolved gasses. Also called Total Dissolved Solids.

d. Volatile and Fixed Residue - the residue remaining after ignition for 1 hour at 550°C represents the ash or fixed solids, and the weight loss incurred is a reasonably accurate measure of organic matter or volatile solids.

2.4.2. *Collection method*: Use a 500 ml plastic bottle to collect **each** type of residue sample and cool to 4°C. The sample has a holding time of 7 days.

2.5. Alkalinity/Acidity - Alkalinity is a measure of the buffering capacity of water - the power of the water to neutralize hydrogen ions - and it is expressed in terms of an equivalent amount of calcium carbonate. Alkalinity is caused by the presence of carbonates, bicarbonates, and hydroxides. Acidity is the power of the water to neutralize hydroxy ions - and it is expressed in terms of an equivalent amount of calcium carbonate. Acidity is a result of the presence of free carbon dioxide, strong mineral acids, weakly dissociated acids, salts of strong acids, and weak bases.

2.5.1 *Collection method:* Collect sample with a 200 ml (for each parameter) plastic bottle, cool to 4°C. Holding time is 14 days.

2.6. TOC (Total Organic Carbon) - Measures the organic carbon present in water. When an empirical relationship can be established between TOC, BOD, and COD, the TOC provides a quick and convenient way of estimating the other parameters that express the degree of organic contamination.

2.6.1 *Collection method:* Collect sample with a 200 ml plastic bottle, add H_3PO_4 to pH <2 and cool to 4°C. Holding time is 28 days.

2.7. Turbidity (Clarity of Water) ï measured in Nephelometric Turbidity Units (NTU). Turbidity is an expression of the optical property that causes light to be scattered and absorbed rather than transmitted in straight lines. Turbidity in waters is a result of suspended matter such as clay, silt, finely divided organic and inorganic matter, soluble colored organic compounds, and plankton and other microscopic organisms.

2.7.1 *Collection method*: Collect sample in a 200 ml plastic bottle, cool to 4°C. The sample should be protected from light. The sample must be received by the lab in **less than 48 hours**.

2.8. Chloride - Chlorides are found in most natural waters. They may be of natural mineral origin or artificially introduced. Chloride concentrations are higher in wastewater than in raw water because sodium chloride (NaCl) is

a common article of diet and passes unchanged through the digestive system (American Public Health Association, 1992). Industrial processes also increase chlorides in wastewater effluents.

2.8.1. *Collection method:* Collect sample in a 500 ml plastic bottle, typically collected directly from the water body as a surface grab (0.15m deep). Cool to 4°C. Holding time is 28 days.

2.9. Chlorophyll *a* and Algal Biomass

2.9.1. *Chlorophyll a* - Chlorophyll *a* is the photosynthetic green, photosynthetic pigment contained in plants. The measurement of this pigment provides an estimate of algal biomass.

Collection method: Use a 500 ml wide-mouth opaque plastic bottle to collect the sample. Cool to 4°C. Sample **must** be received by the laboratory in **less than 24 hours**.

2.9.2. *Algae* - Algae are used as biological indicators of water quality. By determining the types and quantity of algae present in a water body and utilizing physical and chemical data collected at the same time, inferences can be made concerning the trophic state of a water body. Algae are sampled from the water column (phytoplankton), attached to rocks and debris (periphyton), and from floating mats (filamentous/nuisance growths). The primary type sampled by DWR is phytoplankton, although all forms of algae can be sent to the Ecosystems Branch Laboratory of the Environmental Sciences Section for analyses.

Collection method: Samples for phytoplankton should be taken with an integrated depth sampling device (Labline water sampler).

a) This device should be lowered to twice the secchi depth (approximately 1% light penetration) and slowly raised to the surface.

b) Pour sample into a 500 ml plastic disposable bottle and preserve with approximately 2.5ml of modified Lugol's solution or until a dark straw color is reached.

c) If a Labline is unavailable, a surface grab sample can be taken.

d) Scoop samples are taken only when no quantitative methods are possible or as an additional sample for ease in identification. Live samples are taken as above (Labline preferred) but are not preserved. Cool to 4°C. Send to the Lab or EU lab in **less than 24 hours**.

2.9.3. *Chlorophyll a and Algal Sample Submittal Procedure*

a. Samples should then be sent to the Central Laboratory along with nutrient and chemical samples.

b. Bloom samples should include one preserved and one unpreserved (live) phytoplankton sample along with chlorophyll *a* and nutrient samples and a completed bloom form. Bloom forms and modified Lugol's solution (for preservation) are obtained from the Ecosystems Branch in Raleigh.

c. After samples are logged in at the Central Lab and with the Ecosystems Group, they are analyzed per the Ecosystems Branch's SOP manual.

2.9.4. *Parameters collected in conjunction with phytoplankton samples.*

a. Physical Parameters- Are measured at the surface and at every meter or half meter from the surface to bottom according to depth. Parameters include:

1. Temperature
2. Dissolved Oxygen
3. pH
4. Secchi Depth
5. Conductivity
6. Salinity should be taken where appropriate.

b. Chemical samples- Include ammonia/ammonium, nitrate/nitrite, total Kjeldahl nitrogen, orthophosphate, total phosphorous, and chlorophyll *a* are required to accompany phytoplankton samples.

NOTE: Check with the lab prior to sampling for orthophosphate to ensure analysis capabilities.

c. Map showing the location of the sampling site and/ or GPS coordinates.

2.10. Color - Color in water may result from the presence of natural metallic ions (iron and manganese), humus and peat materials, plankton, weeds, and industrial wastes. True color is the color of water from which turbidity has been removed by filtration or centrifugation. The term apparent color includes not only color due to substances in solution, but also that due to suspended matter. Apparent color is determined on the original sample without filtration or centrifugation. In stream samples, unaffected by industrial wastes, usually only true color is analyzed. In some highly colored industrial wastewaters, color is contributed principally by colloidal or suspended material. Therefore, apparent color may be a more appropriate measure for samples related to industrial wastewaters.

The color value of water is extremely pH dependent and increases as the pH of the water is raised. Therefore, always measure in-*situ* pH and specify the pH at which the color is determined.

2.10.1. *Accepted Methods to Determine Color*

There are three accepted methods to determine color (USEPA, 1994): Platinum-cobalt, spectrophotometric and ADMI. Each of these methods yields different information. Their proper uses and interpretations must be reviewed to determine the appropriate test based on the purpose of the sampling.

2.10.2 *Collection method:* Use a 200 ml plastic bottle to collect a surface grab sample. Cool sample to 4°C. Sample must be submitted to the lab in **less than 48 hours**.

2.11. Chromium, Hexavalent [Cr^{+6}] - The principal chromium emissions into surface waters are from metal-finishing processes such as electroplating, pickling, and bright dipping. Uncontrolled emissions have great potential for contaminating the fresh waters with the relatively toxic form, Cr (+6). Other smaller discharges of Cr^{+6} are from the additive in circulating cooling waters, laundry chemicals, and animal glue manufacture.

2.11.1 *Collection method*: Collect sample in a 200 ml plastic disposable bottle and cool to 4°C. Sample must be submitted to the lab in **less than 24 hours**.

NOTE : Lab should be notified that this sample will be submitted for analysis prior to sample collection.

2.12. Cyanide (CN^-) - Cyanides occur in the effluents from gas works and coke ovens, from the scrubbing of gases at steel plants, from metal cleaning and electroplating processes, and from chemical industries. Most of the cyanide in water is in the form of HCN (hydrogen cyanide). Toxicities may vary markedly with pH and a given concentration that is innocuous at pH 8 may become detrimental if the pH is lowered to 6 or less. In natural streams, cyanides deteriorate or are decomposed by bacterial action, so that excessive concentrations may be expected to diminish with time.

2.12.1 *Collection method:*

a. Use two 1 liter plastic bottles collect a surface grab sample directly from the water body.
b. Add NaOH to pH>12 and 0.6g of ascorbic acid if sample contains residual chlorine.
c. Cool sample to 4°C.
d. Sample has a holding time of 14 days.

2.13. Fluoride (F^-) - Fluoride at 0.8 to 1.5 mg/l in drinking water aids in the reduction of dental decay, especially among children. Fluorides in high concentrations are not a common constituent of natural surface waters, but they may occur in detrimental concentrations in ground water. Fluorides are used as insecticides, for disinfecting brewery apparatus, as a flux in the manufacture of steel, for preserving wood and mucilage, for the manufacture of glass and enamels, in chemical industries, and water treatment. While not normally found in industrial wastes, they may be present in traces, or in higher concentrations resulting from spillage.

2.13.1 *Collection method*:

a. Use a 500 ml plastic bottle to collect a surface grab sample directly from the water body.
b. Sample must be cooled to 4°C.
c. Holding time is 28 days.

2.14. Formaldehyde - (HCHO) formaldehyde is a colorless gas with a pungent odor. It is usually stored and transported as an aqueous solution containing 37-50% formaldehyde by weight and 1-15% methanol. Formaldehyde is used in the production of urea-formaldehyde and phenol-formaldehyde resins. These resins are used in the production of plywood, particleboard, foam insulation, and a wide variety of molded or extruded plastic items. Formaldehyde is intensely irritating to mucous membranes and the National Institute for Occupational Safety and Health recommends that formaldehyde be handled as a potential occupational carcinogen. Formaldehyde is used for preserving biological specimens.

2.14.1 *Collection method*:

a. Collect surface grab sample in a 500 ml disposable plastic bottle
b. Sample must be cooled to 4°C.
c. Although no holding time is specified for this sample, it should be submitted to the lab as soon as possible.

2.15. HEM: Grease and Oil - For the grease and oil analysis; groups of substances with similar physical characteristics are determined quantitatively on the basis of their common solubility in trichlorotrifluoroethane (Freon). Grease and oils, either vegetable oil and animal fats or mineral hydrocarbons, when introduced to surface waters, are found floating on the surface, emulsified or solubilized in the water column, or settled on the bottom as a sludge. Potential contributors to oil pollution are all agencies engaged in productions, transportation, handling, and use of oil. Also, ships, railroads, civic dumps, salvage dumps, machining operations, and the most notable - garages and filling stations. Grease from animal and vegetable oils enters waterways from food processors and restaurants. Surface waters are at all times to be kept virtually free from oil or grease, not only for esthetic reasons and taste and odor problems for domestic water supply, but evidence has demonstrated both acute lethal toxicity and long term sublethal toxicity of oils to aquatic organisms.

2.15.1 *Collection method:*

a. Collect 2 liters (two 1 liter glass wide mouth mason jars, Teflon-lined caps) of sample.
b. A surface grab sample at 0.15 m deep is the only collection method.
c. Acidify the sample with HCL or H_2SO_4 to pH <2.
d. Sample must be cooled to 4°C Holding time for this sample is 28 days.

2.16. Total Hardness - Hard waters are generally considered to be those waters that require considerable amounts of soap to produce a foam or lather and that also produce scale in hot water pipes, heaters, boilers, and other units in which the temperature of water is increased materially. In general, surface waters are softer than ground waters. The hardness of water reflects the nature of the geological formations with which it has been in contact. Natural sources of hardness principally are limestone that are dissolved by percolating rainwater made acid by dissolved carbon dioxide. Industrial and industrially related sources include the inorganic chemical industry and discharges from operating and abandoned mines.

Classification of water by hardness content (Conc., mg/l $CaCO_3$) (USEPA, 1976).

Soft	0 - 75
Moderately Hard	75 - 150
Hard	150 - 300
Very Hard	300 and Up

The constituents that impart hardness to water are polyvalent cations, chiefly calcium ($Ca^{++)}$ and magnesium (Mg++). These form insoluble complexes with a variety of anions ($HCO3^-$, $SO4^-$, Cl^-, NO_3^-, SiO_3^-). By convention, hardness is reported on the basis of equivalence as mg/l calcium carbonate ($CaCO_3$).

The DWR Lab is no longer analyzing samples for Total Hardness, therefore, when a total hardness sample is required, a nitric acid (HNO_3)-preserved sample must be submitted for Ca and Mg (see section 2.23). Once the Ca and Mg results are received from the lab, total hardness is calculated using the following formula:

Total Hardness, mg/L = 2.497[Ca, mg/L] + 4.118[Mg, mg/L]

2.16.1. *Collection method:*

a. Sample must be collected as a surface grab (0.15 m from the surface) in a 500 ml plastic bottle.
b. Acidify the sample with HNO_3 to pH <2 and cool to 4°C.
c. Holding time is 6 months.

2.17. Specific conductance (Specific Electrical Conductance) - The specific conductance (conductivity) of a solution is a measure of its ability to carry an electrical current. This ability depends on the presence of ions, their total concentration, mobility, valence, and relative concentrations, and on the temperature of measurement. Specific conductance is the conductance afforded by 1 cc (ml) of a solution of electrolyte and is reported in micromhos per centimeter (µmhos/cm). Specific conductance measurements are used in water analysis to obtain a rapid estimate of the dissolved solids content of a water sample. This measurement is normally

made using a field meter; however, the following procedure can be used if necessary.

2.17.1. *Collection method*:

a. Use a 200 ml plastic bottle collected as a surface grab (0.15 m from the surface).
b. Sample should be cooled to 4°C.
c. Holding time is 28 days.

2.18 MBAS - Methylene-Blue-Active Substances - This test determines surfactants with no specificity, so the materials determined are designated as MBAS. This method depends on the formation of a blue salt or ion pair when methylene blue, a cationic dye, reacts with anionic surfactants. Surfactants are organic materials, which have the property of being surface active in aqueous solution. All surfactants have rather large polar molecules. One end of the molecule is particularly soluble in water and the other is readily soluble in oils. The surfactants include soaps, detergents, emulsifiers, wetting agents, and penetrants. Of these substances, the synthetic detergents are most important and are used in the greatest amounts. Presently, about 80 percent of all synthetic detergents are of the anionic type, and the MBAS method determines the presence of these surfactants. The most widely used anionic surfactant is linear alkylbenzene sulfonate (LAS). The detergent manufacturing industry changed to the production of LAS because it is more readily biodegradable than the older ABS (alkyl benzene sulfonates).

2.18.1 *Collection method*:

a. **The lab must be notified that this sample will be collected and submitted for analy**sis.
b. Use a 500 ml plastic bottle to collect a surface grab sample (0.15 m from the surface).and cool to 4°C.
c. Sample must be returned to the lab in **less than 48 hours**.

2.19. Phenols (C_6H_5OH) - An aromatic compound known as carbolic acid. In concentrated solution, phenol is quite toxic to bacteria and is widely used as a germicide. Phenol is obtained from coal tar and manufactured synthetically. It is used extensively in the synthesis of organic products, particularly phenolic-type resins. Phenolic wastes arise from the distillation of wood, from gas works, coke ovens, oil refineries, chemical plants, and from human and animal refuse.

2.19.1. *Collection method*:

a. Use two- 1 liter glass (phenol bottles) bottles to collect a surface grab (0.15 m from the surface)
b. Acidified the sample with H_2SO_4 to pH <2.
c. Cool the sample to 4°C
d. There is a 28 day holding period.

2.20. Sulfate (SO_4) - The sulfate ion is one of the major anions occurring in natural waters. Sulfates occur as the final oxidized state of sulfides, sulfites, and thiosulfates. Sulfates may also occur as the oxidized stage of organic matter in the sulfur cycle. Sulfates may be discharged in numerous industrial wastes, tanneries, sulfate-pulp mills, textile mills, and other plants that use sulfates or sulfuric acid. Sulfate is important to public water supplies because of its cathartic effect upon humans when it is present in excessive amounts (upper limit-250 mg/l U.S.P.H.S.). Sulfates are of considerable concern to wastewater treatment plants because of odor and sewer corrosion problems resulting from the reduction of sulfates to hydrogen sulfide (H_2S or hydrosulfuric acid in an aqueous solution).

2.20.1. *Collection method*:

a. Sample should only be collected as a surface grab sample.
b. Collect a surface grab (0.15 m from the surface) in a 500 ml plastic bottle
c. Cool the sample to 4°C.
d. Sample has a hold time of 28 days

2.21. Sulfide (S^-) - Sulfides are constituents of many industrial wastes tanneries, paper mills, chemical plants, and gas works. Sulfides are also generated in sewage and some natural waters by the anaerobic decomposition of organic matter. Sulfides react with hydrogen ions to form HS^- or H_2S. The toxicity of sulfides derives primarily from H_2S rather than from the hydrosulfide (HS^-) or sulfide (S^{-2}) ions. H_2S is very toxic and has claimed the lives of numerous workmen in sewers, but owing to the unpleasant taste and odor (rotten eggs), most persons or animals avoid consuming a harmful dose.

2.21.1. *Collection method:*

a. Samples should only be collect as a surface grab.
b. Collect three- 40 ml glass VOA vials with Teflon-lined septum directly as a surface grab (0.15 m from the surface)
c. Allow the sample to overflow the vial.
d. Add 0.1 ml of 2N zinc acetate plus 6N NaOH to pH >9.
e. cap the vial when sample is overflowing ,leaving no air space
f. Cool the sample to 4°
g. Holding time is 7 days.

2.22. Phosphorous and Nitrogen (Nutrients) - Phosphorus occurs in natural waters and in wastewater almost solely as phosphates. Evidence indicates that high phosphorus concentrations are associated with accelerated eutrophication of waters when other growth promoting factors are present, and aquatic plant problems develop in reservoirs and other standing waters at phosphorus values lower than those critical in flowing streams.

Nitrogen is one of the fertilizing elements essential to the growth of algae. Such growth is often stimulated to an undesirable extent in bodies of water that receive excess inputs of nitrogen from either point or nonpoint sources.

2.22.1 *Nutrient Types*

a. NH_3 (Ammonia) - In surface or ground waters, ammonia results from the decomposition of nitrogenous organic matter. It may also result from the discharge of industrial wastes from chemical or gas plants, from ice plants, or from scouring and cleaning operations where ammonia water is used. The conversion of ammonia to nitrites and nitrates by bacteria requires oxygen, and so the discharge of ammonia nitrogen and its subsequent oxidation can seriously reduce the dissolved oxygen levels in rivers and estuaries.

b. TKN (Total Kjeldahl Nitrogen) - Analytically, organic nitrogen and ammonia can be determined together and are referred to as Kjeldahl nitrogen, a term that reflects the technique used in their determination.

c. $NO_2 + NO_3$ (Nitrites + Nitrates) - Nitrites are quickly oxidized to nitrates. Nitrates are the end product of the aerobic stabilization of organic nitrogen. Nitrates also occur in percolating ground waters as a result of excessive application of fertilizer or leaching from septic tanks. Nitrates are seldom abundant in natural surface waters because of uptake by plants.

d. Total P (Phosphorus) - Phosphorus occurs in natural waters and in wastewater almost solely as phosphates. High phosphorus concentrations are associated with accelerated eutrophication of waters when other growth promoting factors are present.

e. PO_4 (Orthophosphate) - Orthophosphate is used as fertilizer and is applied to agricultural and residential cultivated land where it is carried into surface waters with storm runoff.

2.22.2 *Collection Methods for Unfiltered Nutrients* (NH_3, TKN, NO_2+NO_3, and Total P)

a. Use a 500 ml plastic disposable bottle for sample collected.
b. Acidify the sample with H_2SO_4 to pH<2 (to 500 ml sample add 2.0 ml 25% H_2SO_4 **Note**: Addition of an excessive amount of acid will interfere with the sample analysis
c. Cool the sample to 4°C.
d. Holding time is 28 days.

2.22.3. *Collection Method for PO_4 and Dissolved P (Filtered Nutrients)*

This water sample must be filtered in the field. A detailed Standard Operating Procedure for field filtering using a vacuum pump can be found in Appendix 6. Be careful- **do not** allow filter residue to touch filter apparatus or forceps.

a. Use a 200 ml plastic bottle for each sample.
b. **Dissolved P sample is acidized to pH <2 by adding 25% H_2SO_4.**
c. Dissolved P and PO_4 samples must be cooled to 4°C
d. **Holding time for PO_4 is less than 48 hours**
e. The holding time for Dissolved P is 28 days.

NOTE: For Turbid Samples - change filters during process.

2.23. METALS- The following metal parameters are collected in one bottle: Cd, Cr, Cu, Ni, Pb, Zn, Ag, Al, Be, Ca, Co, Fe, Li, Mg, Mn, Na, K, Ba, As, Se, Hg.

Whenever metal samples are collected the collection of field pH is essential. Metals are always collected as a surface grab

Collection method:

a. Collect 500 ml of sample in a plastic disposable bottle directly from the water body as a surface grab (0.15 from the surface).
b. Add HNO_3 to pH <2.
c. Cool the sample to 4°C.
d. Metals have a 6 month holding time with the exception of Mercury (Hg) which is 28 days.

2.23.1. *Cadmium (Cd)* - In the elemental form, cadmium is insoluble in water. It occurs in nature largely as the sulfide salt, greenockite or cadmium blend, often as an impurity in zinc-lead ores. Cadmium is used in metallurgy to alloy with copper, lead, silver, aluminum, and nickel. It is also used in electroplating, ceramics, pigmentation, photography, and nuclear reactors. Cadmium salts are sometimes employed as insecticides and antihelminthics. Cadmium salts may be found in wastes from electroplating plants, pigment works, textile printing, lead mines, and chemical industries. Cadmium has been shown to be toxic to man when ingested or inhaled.

2.23.2. *Chromium (Total Cr)* - The principal chromium emissions into surface waters are from metal finishing processes such as electroplating, pickling, and bright dipping. Other smaller discharges of chromium are from the additive in circulating cooling waters, laundry chemicals and animal glue manufacture, leather tanning, and textile dyeing. Chromium is one of the least toxic of the trace elements. Chromium is not acutely toxic to humans.

2.23.3. *Copper (Cu)* - Copper salts in natural waters are generally the result of pollution attributable to the corrosive action of water on copper and brass tubing, to industrial effluents, and algaecide. Copper salts are used in textile processes, pigmentation, tanning, photography, engraving, electroplating, insecticides, and fungicides. Because copper in concentrations high enough to be dangerous to human beings renders water disagreeable to taste, it is believed that copper is probably not a hazard in domestic water supplies. However, copper in water may be disadvantageous or detrimental for certain industrial uses. In trace amounts, copper may be beneficial or even essential for the growth of living organisms. In excessive quantities it has been found toxic to a wide variety of aquatic forms, from bacteria to fish.

2.23.4. *Nickel (Ni)* - Nickel toxicity to man is believed to be very low. Systemic poisoning of human beings by nickel or nickel salts is almost unknown. Nickel does not merit serious consideration as a water pollutant, but nickel ions may be detrimental to beneficial uses. Nickel is toxic to some plants. Nickel is used in metal plating, batteries, as a catalyst in the preparation of edible oils, and in solar energy equipment.

2.23.5. *Lead (Pb)* - Lead is a cumulative poison. The poisoning usually results from the cumulative toxic effects of lead after continuous, long-term consumption rather than from occasional small doses. Lead exists in nature mainly as the sulfide (galena). Some natural waters contain lead in solution where mountain limestone and galena are found. Lead may also be introduced into water as a constituent of various industrial and mining effluents or as a result of the action of the water on lead in pipes. Atmospheric fallout and rainout of particulate lead are considered the most significant sources of lead input into natural surface waters, especially in urban areas. Storm runoff originating in urban areas will tend to be high in lead concentration. The low solubility of lead in the aqueous phase of natural systems and the formation of stable complexes with organic matter are manifested in the low uptake by some plants and animals. There are extremely low concentrations of lead in natural bodies of water in proportion to the concentration in the beds of lakes and streams. The net effect of these sluggish dynamics is a high degree of accumulation with prolonged exposure.

2.23.6. *Zinc (Zn)* - Zinc is used extensively for galvanizing, in alloys, for electrical purposes, in printing plates, for dye manufacture, and dyeing processes. Zinc salts are used in paint pigments, cosmetics, pharmaceutics, dyes, and insecticides. Zinc is found in high concentrations in natural waters in zinc mining areas and in effluents from metal plating works. In most surface and ground waters it is present only in trace amounts. There is some evidence that zinc ions are absorbed strongly and permanently on silt with a resultant inactivation of the zinc. Zinc has no known adverse physiological effects upon man except at very high concentrations. For esthetic considerations, high concentrations of zinc in domestic water are undesirable. At 30 mg/l, zinc gives water a milky appearance and causes a greasy film on boiling. It is toward fish and aquatic organisms that zinc exhibits its greatest toxicity at much lower concentrations.

2.23.7. *Silver (Ag)* - Silver metal is used in jewelry and silverware, in alloys, for electroplating, and in the processing of food and beverages. Silver nitrate is used in photography, ink manufacture, electroplating, coloring porcelain, and as an antiseptic. Traces of silver can be expected to reach natural waters from such sources. Silver is bactericidal and toxic at low concentrations.

2.23.8. *Aluminum (Al)* - Aluminum is the third most abundant element of the earth's crust. Aluminum occurs in many rocks and ores and clays, but never as a pure metal in nature. The metal itself is insoluble, but many of its salts are readily soluble. Aluminum is not likely to occur for long in surface waters because it precipitates and settles, or is absorbed as aluminum hydroxide or aluminum carbonate. In streams the presence of aluminum ions may result from industrial wastes or more likely from wash water containing alum from water treatment plants.

2.23.9. *Beryllium (Be)* - A relatively rare element, found chiefly in the mineral beryl, this substance is not likely to occur in natural waters. Although the chloride and nitrate forms are very soluble in water and the sulfate form moderately so, the carbonate and hydroxide forms are almost insoluble in cold water. Beryllium is used primarily in metallurgy to produce special alloys, in the manufacture of X-ray diffraction tubes and electrodes for neon signs, and in nuclear reactors. Beryllium is not harmful when taken internally through the digestive tract but has been incriminated in pulmonary ailments of workers exposed to beryllium dusts.

2.23.10. *Calcium (Ca)* - Calcium is the most abundant dissolved cationic constituent of natural fresh waters. This element is widely distributed in the minerals of rocks and soils. Calcium carbonate is frequently found as a cementing agent between mineral particles of sandstone and other detrital rocks. Calcium is one of the constituents of hard water and is a scale former in hot water systems. Prevention of corrosion of cast iron water distribution systems may be obtained through controlled precipitation of calcium carbonate. Lime ($CaOH_2$), and dolomite [$CaMg(CO_3)_2$] are frequently employed as neutralizing agents in water and wastewater treatment.

2.23.11. *Cobalt (Co)* - Cobalt and its salts are used for making alloys, in nuclear technology, as pigment in the china and glass industry, and as binders in the tungsten-carbide tool industry. Cobalt has a relatively low toxicity to man, and traces of cobalt are essential to nutrition.

2.23.12. *Iron (Fe)* - Iron interferes with laundering operations, imparts objectionable stains to porcelain fixtures, and causes difficulties in distribution systems by supporting growths of iron bacteria. Iron also imparts a taste to water, which is detectable at very low concentrations. In addition to corrosion products, natural waters may be polluted by iron-bearing ground water.

2.23.13. *Lithium (Li)* - An alkali metal, it is not widely distributed in nature, being found in a few minerals and in certain spring waters. Lithium is used in metallurgy, medicinal waters, some types of glass, and, as lithium hydroxide, in storage batteries. Lithium is toxic at high concentrations.

2.23.14. *Magnesium (Mg)* - Magnesium ions are of particular importance in that they occur in significant concentration in natural waters, and along with calcium, form the bulk of the hardness reaction. Magnesium is considered relatively non-toxic to man and not a public health hazard because, before toxic concentrations are reached in water, the taste becomes quite unpleasant. At high concentrations, magnesium salts have a laxative effect, particularly upon new users, although the human body can develop a tolerance to magnesium over a period of time.

2.23.15. *Manganese (Mn)* - Manganese is essential for the nutrition of both plants and animals. Manganese is undesirable in domestic water supplies because it causes an unpleasant taste, deposits on food during cooking, stains, and discolors laundry and plumbing fixtures, and fosters the growth of some microorganisms in reservoirs, filters, and distribution systems. Manganese frequently appears in surface waters as the result of decaying vegetation, in waters with acid pH values, and acidic waters from coal mine drainage. In ground water subject to reducing conditions, manganese can be leached from the soil and occur in high concentrations.

2.23.16. *Sodium (Na*) - Sodium salts are extremely soluble in water; any sodium that is leached from soil or discharged to streams by industrial wastes will remain in solution. Sodium is the cation of many salts used in industry and as such is one of the most common ions in process wastes. Sodium in drinking water may be harmful to persons suffering from cardiac, renal, and circulatory diseases.

2.23.17. *Potassium (K)* - One of the more common elements, potassium is one of the most active metals, and for that reason it is not found free in nature but only in the ionized or molecular form. Potassium is used for fertilizers and some varieties of glass. It is an essential nutritional element, but in excessive quantities it acts as a cathartic.

2.23.18. *Barium (Ba)* - Barium ions are not normally present in natural surface or ground waters in measurable concentrations although they have been detected in a few springs and in effluents from areas where barytes, $BaSO_4$, or witherite, $BaCO_3$, are mined. Barium and its salts are used in the metallurgical industry for special alloys, in the paint industry, in cements designed to withstand salt water, and in the ceramic and glass industries. Because of possible toxic effects on the heart, blood vessels and nerves a surface water supply standard of 1.0 mg/l was established.

2.23.19. *Arsenic (As)* - Arsenic may occur in water as a result of mineral dissolution, industrial discharges, or the application of insecticides. Arsenic is toxic to humans and accumulates in the body.

2.23.20. *Selenium (Se)* - Elemental selenium is practically nontoxic, but hydrogen selenide and other selenium compounds are extremely toxic and resemble arsenic in their physiological reactions. Selenium poisoning occurs mostly among livestock, and the toxic effects appear to be associated with the consumption of high concentrations of selenium in food, such as locoweed or grains grown in soils with high concentrations of selenium, rather than from water consumption. Selenium occurs in sulfur deposits, sulfides of metals, volcanic emissions, sedimentary rocks, organic-rich soils, and coal. Selenium is used in the electronics industry, xerographic copying machines, photoelectric cells, glass and ceramics, pigment manufacture to color plastics, paints, enamels, inks, and rubber. It is also used as a component of plating solutions. It can also be found in discharges from coal-fired power plants.

2.23.21. *Mercury (Hg)* - Mercury and mercuric salts are considered to be highly toxic to humans and aquatic life. Elemental mercury is inert chemically and insoluble in water, and is not likely to occur as a water pollutant. Mercuric salts occur in nature chiefly as the sulfide HgS, known as cinnabar, but numerous synthetic organic and inorganic salts of mercury are used commercially and industrially. They are used in medicinal products, disinfectants, detonators,

pigments, and photoengraving. Many of the mercuric and mercurous salts are highly soluble in water.

3. PESTICIDES AND ORGANICS

3.1. Pesticides - Pesticides are any substance or mixture of substances intended for preventing, destroying, repelling, or mitigating insects, rodents, fungi, viruses, or weeds, and other forms of plant or animal life considered to be pests. Pesticides are categorized into three groups:

- Inorganic - arsenicals, mercurials, borates, and fluorides
- Synthetic organic - chlorinated hydrocarbons, organic phosphates, and thiocarbamates
- Natural organic - rotenone, pyrethrum, and nicotine

Pesticides may also be classified by their biological usefulness as algaecides, acaricides, fungicides, and herbicides.

3.2. Organics - All organic compounds contain carbon in combination with one or more elements.

3.3. Collection Methods

3.3.1. *Pesticides, semivolatile organics, and acid herbicides*

a. Collect each sample (Pesticides, semivolatile organic & acid herbicides) into a separate 1 gallon amber glass jug with a Teflon-lined cap.

b. The sample is collected directly from the water body as a surface grab (0.15m deep).

c. Add sodium thiosulfate and cool to 4°C.

d. Holding time is 7 days.

3.3.2. *Purgeable Organics (VOA)*

a. Collect sample into three-40 ml Teflon vials, remove cap underwater.

b. When collecting in waters with no chlorine, use vials pre-preserved by the Central Laboratory with sodium bisulfate ($NaHSO_4$).

c. The vial should be filled and capped underwater (0.15m deep) with no air space in the vial. While keeping lid and bottle under water gently rock the lid and bottle to remove air bubbles (unless bottle is pre-preserved). The volatile organics vials should be completely filled to prevent volatilization, and extreme caution should be exercised when filling a vial to avoid any turbulence that could also produce volatilization.

d. When collecting in waters where chlorine is present, first preserve an empty vial with 0.6 g of ascorbic acid before filling and capping the vial underwater. After capping the vial, remove the vial above water, uncap, and add 0.25 g of sodium bisulfate leaving no air space before recapping the vial.

e. The three vials should be placed in a Ziploc bag in the cooler. A trip blank is also required. This is a vial filled at the laboratory with appropriate bottled water and placed in a Ziploc bag in the same cooler with the other VOA vials.

f. A separate laboratory sheet is filled out for the trip blank and this sample is used to determine if any contamination has occurred of the VOA samples.

h. Cool to 4°C.

3.3. Instructions for requesting a pesticide or organic analysis

When a particular pesticide or organic is suspected or known to be present in a sample, its name should be entered on the laboratory form. With this information, the laboratory can focus immediately on the analytical methodology for determining the presence and concentration of the suspected pollutant and as a result possibly decrease the analysis time. A list of specific pesticides and organics currently analyzed at the Laboratory is available online: http://portal.ncdenr.org/web/wq/lab/ops/org

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V. SEDIMENT COLLECTION AND PRESERVATION

1. COLLECTING SUSPENDED SEDIMENT

1.1. Samplers and Applications

For more descriptive information about these samplers see references (Inter-Agency Committee on Water Resources 1965).

1.1.1. *U.S. DH-48 – When in wadable streams*

1.1.2. *U.S. DH-59 [Equal Width Increment Method (E.W.I.)]*

1.1.2.1 Used When

a. Too deep for wading but less than 15 feet deep.
b. From low bridges.
c. Velocities less than approximately 5 ft/ sec.

1.1.2.2 Sampling Tips

a. Set out safety equipment (cones, high visibility vests, etc.) as necessary and assemble sampling equipment. Note: Prior to using any sampler, it should be thoroughly cleaned and inspected.
b. Rinse sampler with distilled water before the first station and between stations to wash away any contaminants.
c. Use the upstream side of bridges if possible.
d. Go to midstream or the area where most of the flow is occurring. First sampling point must be made where the flow is greatest.
e. Lower and raise the sampler at a consistent rate with the nozzle oriented upstream to the bottom, immediately reverse it and raise to above the water surface. Repeat until jar is filled within approximately 3 inches of the top of the jar (350-440 cc). Rate must not exceed 0.4 times the mean velocity and must be fast enough to keep from overfilling.
f. If bottle overfills - discard sample, rinse bottle, and collect again. Use a smaller nozzle or a faster transit rate.
g. Raise the sampler and pour contents into a cleaned sample splitter. For cleaning instructions see USGS references. The sample splitter should be rinsed with distilled water before the first station and between stations.
h. Sample at the next sampling point and place contents into a mixing churn.

i. Ideally try for 3 sampling points, midstream and quarter points, but the situation might indicate otherwise (if maximum flow is not midstream). Sampling points should be equally spaced.

j. If more sample is needed for the churn, take a second set of samples at the same transit rate at all verticals.

k. Churn sample at a uniform rate of about nine inches per second. Disc should touch the bottom of the tank on every stroke and the stroke length should be as long as possible without breaking the water surface.

l. After churning for about l0 strokes, withdraw sub-samples and place in ½ liter bottles. As sub-samples are withdrawn, maintain churning rate. If there is a break in withdrawals, the stirring rate must be re-established before withdrawals can continue.

1.2. Variations on Suspended Sediment Sampling

1.2.1. When suspended materials in the stream are uniformly distributed, a representative sample can be obtained by sampling vertically at one location near the center of the flow.

1.2.2. Use surface or dip sampling instead of depth integrated sampling when:

- Stream velocity is too high.
- Large floating and moving submerged debris is in the stream.
- A depth-integrated sampler is not available.
- The depth of the stream is very shallow.

2. COLLECTING BOTTOM SEDIMENT

2.1. Containers and Volumes

2.1.1. *Sample Containers*

a. Use certified jars for sediment samples or as indicated by Chemistry Laboratory.

b. Use Teflon lid or parafilm between jar and lid for nutrients and all metals.

c. Tin foil can be used between jar and lid for all metals except aluminum.

d. Use Teflon lid or tinfoil for pesticides.

2.1.2 *Required volume*

- One pint of sample must be obtained for analyses of metals, nutrients, and organics.

3. BOTTOM SEDIMENT SAMPLERS, APPLICATIONS, AND PROCEDURES

For more descriptive information about these samplers see references.

3.1. Ekman grab (Figure 12)

3.1.1. *Locations Suitable for Use*:

a. Use in soft finely divided littoral bottoms of lakes, ponds, and streams that are free from vegetation (sticks, partially decayed leaves, etc.) as well as intermixtures of sand, stones, and other coarse debris.

b. Calm waters.

c. Low velocity streams.

d. Low bridges (messenger can damage spring mechanism if used from high bridges)

3.1.2. *Sampling Tips:*

a. Make sure grab is operating correctly. The grab can cause severe injury. Do not activate unit while holding.

b. If sampling from a low bridge, it may be advisable on wide streams to take 3 samples (midstream and quarter sections) and composite them to form l sample in a Nalgene mixing tub.

c. Set in open position by locking open the spring operated jaws.

d. Operating procedures are similar to those of the Petersen grab starting at step 5.2.6.

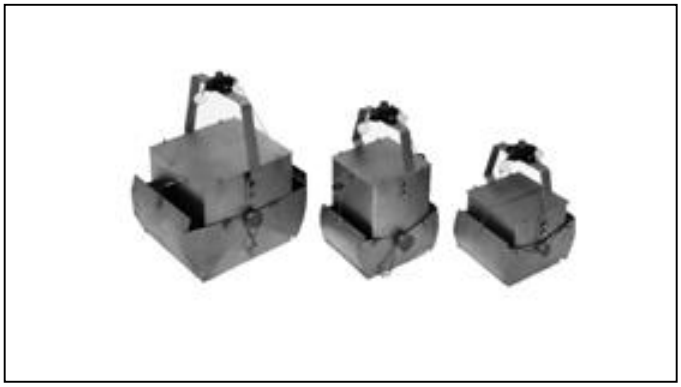


Figure 12. Ekman Grab Samplers

3.2. Petersen grab (Figure 13)

3.2.1. *Locations Suitable for Use:*

a. Hard bottoms (sand, gravel, marl, clay, etc.).
b. Strong velocities.
c. Very deep water

3.2.2. *Sampling Method and Tips*

a. Use with hoist because of its weight.
b. Make sure grab is operating correctly and rinse in water at first station and between stations.
c. Move jaws to open position, bring free end of horizontal locking bar into position in the locking notch on upper bar, insert safety pin lock.
d. Swing grab over side, remove safety pin lock, and lower slowly to bottom.
e. When grab is at the bottom, allow a moment for it to sink into the bottom then slack off on the line.
f. Resume tension on the line to close grab.
g. Pull grab to surface, swing inboard over a tub and discharge sample.
h. Place sample in jar. Approximately one pint of sample is needed.
i. If jaws of grab are jammed due to a stick, rock, or other hard object, discard sample, clean grab and sample again.

Figure 13. Peterson Grab Sampler

3.3. Ponar grab (Figure 14)

3.3.1. *Locations Suitable for Use:*

a. All types of bottoms except the hardest clays.
b. Strong velocities.
c. Very deep water

3.3.2. *Sampling Method and Tips*

a. Use with hoist because of its weight.
b. Make sure grab is operating correctly and rinse in water at first station and between stations.
c. Move jaws to open position, bring free end of the horizontal locking bar into position in locking notch on upper bar and insert safety pin lock.
d. Remove safety pin lock and lower sampler slowly.
e. When the grab is at the bottom, wait a minute to allow it to sink, and then slack off the cable.
f. Lift the sample maintaining tension and raise steadily and slowly to surface.
g. Swing inboard and open sampler over a tub to discharge sample.
h. Place sample in jar. Approximately one pint of sample is needed.
i. If an object is wedged between the jaws, discard sample, clean sampler, and sample again.
j. At the conclusion of sampling, replace the safety pin lock.

Figure 14. Ponar Grab Sampler

4. BOTTOM CORE SAMPLERS, APPLICATIONS, AND PROCEDURES

4.1. Phleger core sampler (Figure 15.)

4.1.1. *Locations Suitable for Use:*

a. Use with hoist because of its weight.
b. Use where water is too deep to use hand coring devices.
c. Sampling soft, sandy or semi-compacted sediments.

4.1.2. *Phleger Core Sampler Methods*

a. Make sure sampler and core tubes are clean and operating properly, rinse corer at first station and between stations.
b. Lower sampler to bottom, then raise off the bottom approximately one to two meters.
c. Drop sampler again to collect core.
d. Swing sampler inboard over a Nalgene tub.
e. Remove tube and core, measure out top two inches of core.
f. Place this portion of core into jar.
g. Repeat sampling until approximately one pint of sample is obtained.

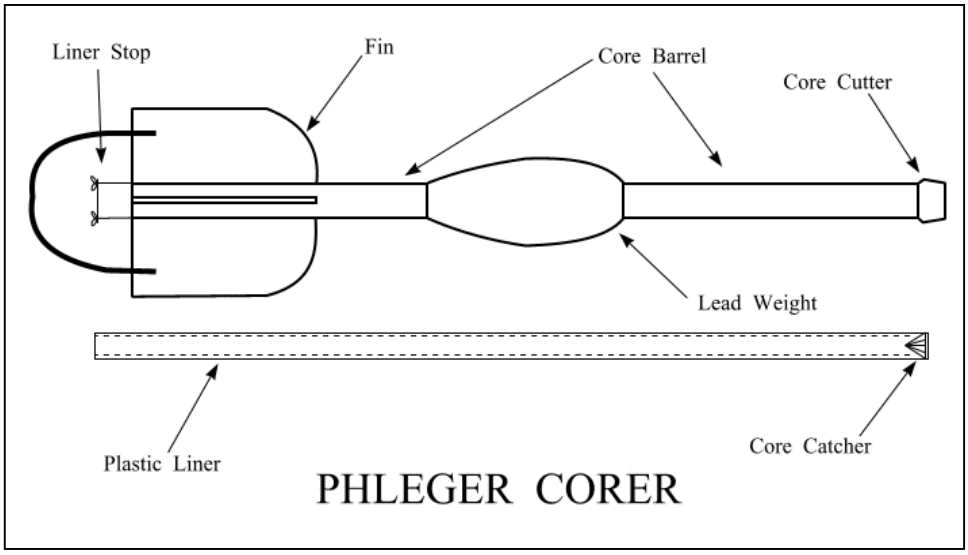


Figure 15. Phleger Corer Diagram

4.2. Wildco Light Duty Model 2414 Core Sampler

4.2.1. *Locations Suitable for Use:*

a. Use by hand or on the end of a line.
b. Where sediment is relatively soft.

4.2.2. *Wildco Light Duty Model 2414 Core Sampler Methods*

a. Make sure sampler and core tubes are clean and operating properly, rinse corer at first station and between stations.
b. Lower sampler to bottom, raise again and drop if necessary to take sample.
c. Remove plastic core, measure out top two inches of core.
d. Place this portion of core into jar.
e. Repeat sampling until approximately one pint of sample is obtained.

4.3. Hand coring deviceï for shallow water use.

4.3.1 *Procedure for hand coring device:*

a. Make sure sampler is clean before using. Rinse before first station and between stations.
b. Take sample by turning sampler into sediment.
c. Remove sampler and core.
d. Measure out top two inches of core.
e. Place this into jar.
f. Repeat sampling until approximately one pint of sample is obtained.

4.4. Hand Sampling Method

a. Face upstream in shallow, wadable streams.
b. Make sure that spoon or scoop has been thoroughly cleaned.
c. Scoop the sample directly into the jar and get a representative sample. It may be advisable to take several samples and consolidate (midstream and quarter points).

VI. STANDARD CLEANING PROCEDURES

1. GENERAL

The procedures outlined in this section are to be used by all personnel to clean sampling equipment and sample containers prior to field use. These procedures assure the standard operating procedures (SOP) for the Section; any deviation from them must be documented in field records and investigative reports.

All equipment and sample containers that are cleaned using these procedures will be tagged, labeled or marked with the following information:

- Name of person cleaning equipment or containers
- Date equipment or containers were cleaned
- Any deviation from SOP that was employed

All equipment and reusable sample containers used to collect samples will be identified at the conclusion of sampling activities. Any problems encountered with the equipment or needed repairs will also be noted. Equipment or reusable sample containers needing cleaning or repairs should not be stored with clean equipment, sample tubing or sample containers. Equipment, reusable sample containers, disposable sample containers, and sample tubing that are not used during the course of an investigation may not be replaced in storage without being re-cleaned if these materials are transported to a facility or study site where herbicides, pesticides, organic or other toxic materials are present or suspected of being present. All portions of unused coils of tubing that are returned shall be re-cleaned before being restocked. If these materials are transported to a facility in connection with a routine inspection or study where toxic or organic materials are not known or not suspected of being present, they may be placed back in storage without being cleaned.

Sufficiently clean equipment and sample containers should be transported to the field so that an entire study can be conducted without the need for cleaning equipment in the field. However, this will not always be possible when using coring equipment, dredges, buckets, water samplers, pumps and other such equipment. Field cleaning procedures are included to cover these special problems. Emergency field sample container cleaning procedures are also included; however, they should not be used unless absolutely necessary. Specific cleaning procedures are included in the following paragraphs.

2. AUTOMATIC SAMPLING EQUIPMENT

2.1. General Cleaning

2.1.1. *For All Automatic Samplers*

a. The exterior and accessible interior (excluding the waterproof timing mechanism) portions of automatic samplers will be washed with phosphate free laboratory detergent and rinsed with tap water.

b. The face of the timing case mechanism will be cleaned with a damp cloth.

c. All sample intake tubing will be discarded after use. Pump tubing should be cleaned with pesticide grade solvents.

d. New pre-cleaned, silicone pump tubing (see section on cleaning tubing) will be installed with the aluminum or Teflon tubing caps intact.

f. When using the samplers for collecting samples for metals and/or organic samples, the metal distributor tubes should not be used for this purpose.

g. The automatic samplers should not be used for collecting samples for organic analyses in the individual bottle mode since there is no way to properly clean the distributor plate to remove any residual organic compounds. The sample tubing headers may not be used to collect samples for organic analyses for the same reason.

2.2. ISCO Specific Cleaning Procedures

2.2.1. *Automatic sampler rotary funnel, distributor and metal tube*

a. Use only for non-organic sample collection using individual sequential bottles.

b. Clean with hot water, phosphate free laboratory detergent and a brush.

c. Rinse thoroughly with hot tap water.

d. Rinse thoroughly with distilled water.

e. Replace in sampler.

2.2.2. *Automatic sampler headers*

a. Rinse entire header with hot water, a bottle brush, and phosphate free laboratory detergent.

b. Disassemble header and rinse thoroughly with hot tap water, using a brush to remove particulate matter and surface films.

c. Rinse plastic portion of the header with 20 percent nitric acid. Do not use acid on metal parts.

d. Rinse thoroughly with tap water.

e. Reassemble header and rinse with distilled water.

f. Let dry thoroughly and wrap with aluminum foil.

g. Headers may not be used when collecting samples for organic analyses.

2.2.3. *Glass reusable composite containers (2 ½, 3 and 5 gallon capacities)*

a. After using, rinse with water in the field, seal with aluminum foil to keep the interior of the container wet and return to the laboratory.
b. Wash thoroughly with hot tap water and phosphate free laboratory detergent, using a bottle brush to remove particulate matter and surface film.
c. Rinse thoroughly with hot tap water.
d. Wash with 10 percent nitric acid.
e. Rinse thoroughly with tap water (at least 3 times).
f. Rinse thoroughly with distilled water (at least 3 times).
g. Rinse thoroughly with acetone (pesticide grade). Caution: Acetone must be removed before using. Residual acetone will interfere with certain analyses.
h. Rinse twice with distilled H_20. Allow to air dry,
i. Cap with aluminum foil or Teflon film.
j. Do not use composite containers used to collect samples at facilities manufacturing pesticides, herbicides or other toxic or noxious compounds. These are to be properly disposed of at the DWR Chemistry Laboratory.
k. Glass composite containers used to collect in-process wastewater samples at industrial facilities will be discarded after sampling.
l. Any bottles that have a visible film scale or discoloration remaining after this cleaning procedure are to be discarded.

2.2.4. *Glass sequential sample bottles (automatic sampler base for sequential mode)*

a. Rinse bottles in the field after using with tap water and seal with aluminum foil or cap for return to laboratory.
b. Rinse thoroughly with hot tap water.
c. Wash with 20 percent nitric acid.
d. Rinse thoroughly with tap water.
e. Place in dishwasher - phosphate free detergent cycle followed by tap and distilled water rinse cycles.
f. Replace in covered, automatic sampler base; cover with aluminum foil for storage.

2.2.5. *Bottle siphons*

a. Use a new siphon for each sampling location.
b. Pre-rinse the 3/8 inch Teflon tubing (used to make siphons for organic analyses) as in Teflon tubing cleaning instructions.

c. Flush the PVC 3/8 inch tubing used for samples other than those collected for organic analyses with sample before use.

2.2.6. *Teflon composite mixer rods*

- Use the sample cleaning procedure outlined for glass reusable composite containers above.

2.2.7. *Automatic sampler rubber pump tubing*

- Only new pre-rinsed tubing should be used for each automatic sampler set up

a. Rinse tubing with hot tap water for five minutes.
b. Rinse outside of tubing with hexane.
c. Install in automatic sampler.
d. Cap both ends of tubing with aluminum foil or Teflon film.

2.2.8. *Teflon sampler tubing (pure Teflon or Teflon lined)*

a. If required length is known pre-cut Teflon tubing or clean l00 feet coil intact.
b. Rinse outside of tubing with hexane.
c. Flush interior of tubing with hexane.
d. Air dry.
e. Cap each end of tubing with aluminum foil or Teflon tape and completely wrap the coil of Teflon tubing with aluminum foil to prevent contamination.

2.2.9. *Polyvinyl chloride sample (PVC) tubing (1/18, l/l4, or 3/8 Inch)*

a. Use only new tubing.
b. Use in selective sampling where organics are not of concern.
c. Flush the tube with sample immediately after the sampler is set up at the sampling site to remove any residues from the manufacturing or extruding process.
d. Store tubing in original container and do not removed from this container until needed.

2.2.10. *Stainless steel tubing*

Tubing will be flushed in the field with tap water after use and cleaned as follows upon return to the laboratory:

a. Wash with phosphate free laboratory detergent and a long bottle brush.
b. Rinse with hot water for 5 minutes.
c. Rinse with acetone.
d. Rinse with distilled water for one minute.
e. Air dry.
f. Rinse with hexane.
g. Completely wrap tubing, including ends, with aluminum foil to prevent contamination during storage.

3. MISCELLANEOUS SAMPLING AND FLOW MEASURING EQUIPMENT

Miscellaneous flow measuring and sampling equipment should be washed with phosphate free laboratory detergent and rinsed with hot tap water before being stored. For Lablines, rinse at least three times with distilled deionized water and cover the top of the Labline with foil to prevent contamination and to show that the Labline has been cleaned.

A different procedure is used for any equipment utilized in organic or toxics sampling.

4. STAINLESS STEEL SAMPLING EQUIPMENT

For collecting samples for organic analyses:

4.1. Follow the procedures given in the Automatic Sampler Section, Glass Reusable Composite Containers, but omit acid rinse.

4.2. Wrap equipment completely in aluminum foil to prevent contamination during storage.

5. OTHER FIELD INSTRUMENTATION

NOTE: Where available, always follow the manufacturerôs recommendations for cleaning the device (see Appendices 1-4).

The exterior of sealed, watertight equipment such as Labline Samplers and field meters should be washed with a mild detergent (liquid dishwashing detergent, for example) and rinsed with tap water before storage. The interior of such equipment may be wiped with a damp cloth if necessary. Other field instrumentation should be wiped with a damp cloth. Probes for pH, conductivity, DO, etc. should be rinsed with distilled water before storage. The desiccant in flow meters and other equipment should be checked and replaced if necessary each time the equipment is cleaned.

Keep meters clean and in good operating condition. Probes should be rinsed at the end of each sampling day, properly stored and cleaned on a regular basis.

6. ICE CHESTS AND SHIPPING CONTAINERS

All ice chests and reusable shipping containers will be washed with a mild detergent (interior and exterior) and rinsed with tap water and air dried before storage.

7. FIELD CLEANING PROCEDURES

For routine operations involving classic parameter analyses, water quality sampling equipment such as Kemmerers, buckets, DO dunkers, dredges, etc. may be cleaned with sample or tap water between sampling locations. A brush may be used to remove deposits of material or sediment if necessary. Flow measuring equipment such as weirs, staff gages, velocity meters, and other stream gauging equipment should be cleaned with tap water after use and between measuring locations. When sampling equipment (not tubing) is to be utilized for collecting organic or toxic samples, the following cleaning procedure is to be used between sampling locations:

- Clean with tap water and brush if necessary.
- Rinse with pesticide grade acetone.
- Rinse thoroughly with tap water (if available).
- Rinse with distilled water.

It must be emphasized that these procedures are only to be used in the field. All equipment will be cleaned before storage at the laboratory utilizing the procedures previously outlined.

8. VEHICLES

All vehicles used by staff should be washed on a routine basis. This routine maintenance should minimize any chance of contamination of equipment or samples due to contamination of vehicles. When vehicles are used in conjunction with hazardous waste site inspections, or on studies where pesticides, herbicides, organic materials or other toxic matter are known or suspected to be present, a thorough interior and exterior cleaning is mandatory at the conclusion of such investigations. All vehicles shall be equipped with trash bags and/or trash containers to facilitate vehicle cleaning. All contaminated trash and equipment must be kept separate from ordinary trash and must be disposed of properly on-site or on return to the facility.

9. DISPOSABLE SAMPLE CONTAINERS

All disposable sample containers will be stored in their original packing containers in a clean, dust free environment. When any packing container is opened, all disposable sample containers inside should be immediately capped if they are found uncapped.

VII. TIME-OF-TRAVEL & DYE TRACING

1. FLUORESCENT DYE

The preferred dye for use in time-of travel studies by the North Carolina Division of Water Resources is Rhodamine W. T. (20%) solution. This is a red fluorescent dye which mixes well with water and is easily detected through visual means under high concentrations and through the use of a fluorometer for concentrations to as low as 0.01 parts per billion. Rhodamine WT has properties essential for water tracing studies. Rhodamine WT is:

- water soluble,
- highly detectable-strongly fluorescent,
- fluorescent in a part of the spectrum not common to materials generally found in water, thereby reducing the problem of background fluorescence,
- harmless in low concentrations,
- inexpensive, and
- reasonably stable in a normal water environment (Wilson, Cobb & Kilpatrick, 1986).

Rhodamine dye can also be used to determine such things as short-circuiting in wastewater treatment plants, outlets from storm drains, septic tank leakage, etc.

Most of ISBôs dye studies are performed as part of a waste-load allocation model. This model requires that a stream be segmented into different reaches based upon predicted stream velocities, stream morphology, total distance of the study area, and major inputs from dischargers and tributaries. A dye sampling station is required in each of these reaches.

1.1. Safety

(MSDS is kept with dye container)

1.1.1. *Personal Protection*

a. Latex or vinyl gloves (in lab and field).
b. Goggles
c. Ventilated room
d. Apron

1.1.2. *Emergency and First Aid Procedure*

a. Inhalation:
- move to fresh air.
- Give oxygen and medical help if breathing is difficult.

b. Eye contact:
- Flush eyes with flowing water for at least 15 minutes, holding eyelids apart to irrigate thoroughly.
- Get medical attention right away.

c. Skin contact:
- Wash affected skin areas thoroughly with soap and water.
- If irritation develops, consult a physician.

d. Ingestion:
- If swallowed, dilute with water and induce vomiting.
- Get immediate medical attention.
- Never give fluids or induce vomiting if patient is unconscious or has convulsions.

1.2. Equipment - Fluorometer

1.2.1. Turner Designs Model 110 - reads dye concentrations directly in ppb. Operating instructions are contained in the Turner Designs Model 10 Operators manual, section 3-operations.

1.2.2. Turner Model 10-AU - reads dye concentrations in ppb. Operating instructions are contained in the Turner Designs Model 10-AU operating manual.

2. PRE-SURVEY

2.1. Surface Water Supplies

2.1.1. Identify all surface water supplies in or downstream from the study area.

2.1.2. Notify each water supply operator that may be affected in the study area, the DENR - Division of Water Resources regional water quality supervisor that a dye study is scheduled to be performed. Explain the reason for the study and inform the water treatment operator that DWR personnel will monitor dye concentrations at their water intake. If dye concentrations in the river exceed 10 ppb, the facility will be informed to shutdown their operation until river dye concentrations fall below 10 ppb. All efforts should be made to calculate a dye dosage that will result in a dye concentration at a water supply significantly below the 10 ppb.

2.2. Field Reconnaissance

1. Select dye sampling stations. Stations are selected based upon access, distance from the dose, and model requirements.
2. Locate all USGS gage stations in the study area or sites at which flows can be performed.
3. Determine if any dam structures that can regulate flow exist in the study area. If there is such a dam structure, a station is usually set up just upstream of the dam and an additional dose is made below the dam.

3. DYE REQUIREMENTS (ESTIMATING DOSAGE)

For Rhodamine WT 20 percent dye the dosage formula is:

$$V = 3.4 * 10^{-4} * [(Qm * L)/Vm]^{0.93} * Cp$$

Where:

V is the volume of dye, in liters
Qm is the maximum discharge in the reach, in cfs
L is the distance from injection to sampling point, in miles
Vm is the mean velocity, in fps
Cp is the peak concentrations desired in εg/l

The volume of Rhodamine WT 20 percent dye required to produce a peak concentration of 1 εg/l (ppb) can be determined from the nomograph in Figure 16 for a range of flow-reach conditions.

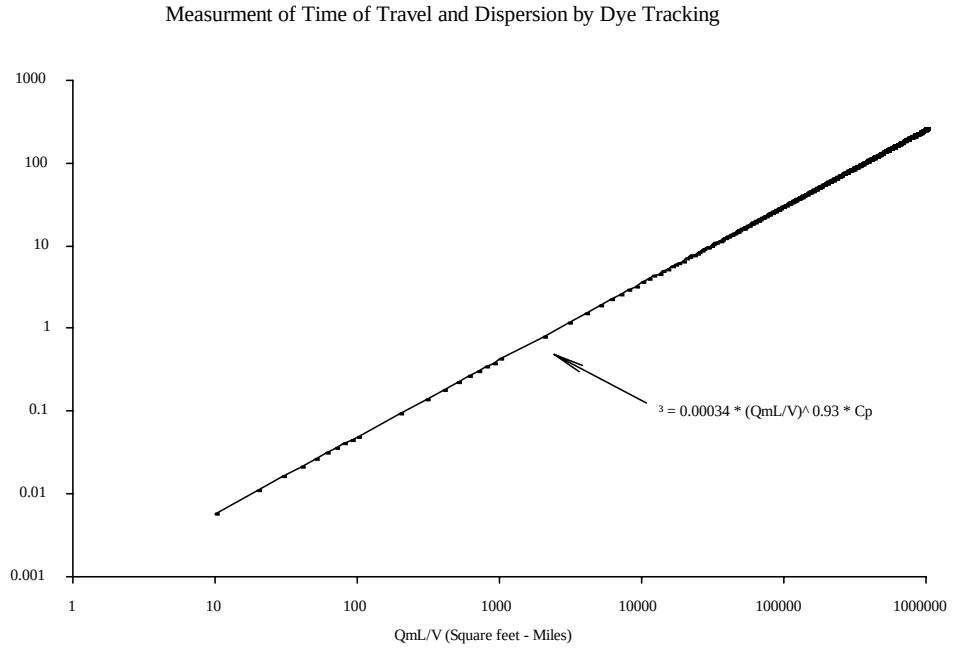


Figure 16. Nomograph for determining volume of dye necessary to produce peak concentration

4. INJECTION OF DYE

4.1. Injection Types

4.1.1. *Single Slug Injection*

a. A single slug injection of dye is usually made in the center of the thread of flow.

b. The desired quantity of dye as calculated in part 3 (above) can be poured into the stream from a container.

4.1.2. *Continuous injection*

a. The desired quantity of dye is pumped into the water column at a fixed rate for a fixed time using an ISCO sampler or a peristaltic pump. Tubing is run from the pump to the desired dye injection point. Contaminated pump lines are first flushed with stream water and then placed in plastic bags for shipment back to the lab.

5. COLLECTION OF WATER SAMPLES

Samples should be taken in pre-numbered glass bottles by a hand sampler or by ISCO samplers. Care should be taken to collect samples in the peak concentration of the dye cloud, i.e. do not sample midstream if the dye cloud is along one stream bank.

5.1. Dye Sample Collection

5.1.1. *Data recorded on field sheets* (Figure 17)

a. Station Location-Sampling Point
b. Date
c. Sample Bottle Number
d. Time
e. Name of Sampler

5.1.2. *Methods and Guidance*

a. At least one background sample is needed for measurement of background fluorescence at each site in the study reach before the dye arrives.
b. Sampling should begin early enough to determine the true dye peak.
c. Sampling should continue until a peak has been determined; and until a decreasing trend has been clearly established.

5.5.1. *Sampling Schedule*

a. The schedule for collecting samples at each sampling site is the most uncertain aspect of the plan.

b. Estimates of the time to begin sampling, time intervals between samples, and the duration of sampling must be made, which will ensure adequate definition of the dye cloud passing each site. It is better to start with more frequent sampling and decrease frequency based on sampling results when travel times are unknown.
c. An estimate of the time-of-travel between sampling sites is usually based on the cloud's movement to the first sampling site downstream of the injection site.

WATER QUALITY SECTION
INTENSIVE SURVEY
DYE TRACER STUDIES

SUB-BASIN: ___________

STREAM: ______________________________ LOCATION: __________________________________

SAMP. NO	SAMP Point	DATE	TIME	G. H. OR RP-WS	FIELD READINGS				LABORATORY READINGS				CONC (ppb)
					1X	3X	10X	30X	1X	3X	10X	30X	

COMMENTS:

Figure 17. Dye Tracer Study Field Sheet

5.6. Sample Collection Methods

5.6.1. *Hand Sampling*

a. Grab sample by hand dipping a bottle or by using a dye sampler.

b. Depth integrated sample a Labline sampler is needed.

5.6.2. *Automatic Samples (ISCO Sampler)*

a. Samples can be analyzed directly from the ISCO bottles, however, if the ISCO bottles are needed to continue sampling the samples can be transferred to numbered glass bottles.
b. Label bottle racks.
c. To reuse ISCO bottle, rinse three times using tap water or if tap water is unavailable uncontaminated stream water can be used.

6. FLUOROMETER USE

Refer to fluorometer manufacturerôs operating instructions for specific procedures and service instructions. The Turner Designs Model 10 Fluorometer has two main scales; an X1 and an X100. When the fluorometer is in the X1 position, the sensitivity of the instrument is as indicated by the range lights. When the fluorometer is in the X100 position, the sensitivity of the instrument is 100 times that indicated by the range lights.

The scale for the Turner Designs Model 10 Fluorometer is:

Scale	Range	Concentration (ppb)
X100	X10	0-1
X100	minimum sensitivity	1-10
X1	X10	10-100
X1	minimum sensitivity	100-1000

6.1. Fluorometer Usage

6.1.1. *Calibration-*

(Fluorometric Procedures for Dye Tracing, Book 3, Chapter A12, Revised 1986.)

a. Use a range of dye concentrations (ex. 1 εg/l, 10 εg/l, 50 εg/l, 100 εg/l with εg/l=ppb) to calibrate the fluorometer prior to a dye study.
b. Calibrate all fluorometer at 1 ppb ï DWR preference.
c. Calibrate fluorometer prior to taking out in the field, before running samples in the field, and before running samples in the lab

6.1.2. *Sample Collection*

a. Prepare solution standards- Dye standards of known concentrations should be prepared in accordance with the U. S. Geological Surveyôs dye tracing procedures contained in Turn the fluorometer on and allow it to stabilize for at least 10 minutes.
b. Use a distilled water blank to zero the instrument.
c. Rinse the cuvette with water from the sample bottle before running that sample. Wipe off moisture from the outside of the cuvette.
d. Run samples.
e. Record measurements on the field sheet.
f. Keep samples for future analysis, especially if peak concentration is questionable.

VIII. FLOW MEASUREMENT

1. INTRODUCTION

Stream-flow or discharge is defined as the volume rate of flow of the water including any sediment or other solids that may be mixed with it (Buchanan and Somers 1968). Stream-flow is usually expressed in cubic feet per second (cfs) and discharge flow in million gallons per day (MGD).

Several methods of determining flow are used by DWR. Most consist of wading into the stream with a top-setting flow rod and a vertical axis type flow meter shown as a propeller in Figure 18.

Other methods of determining flows (usually small, low-velocity flows) are:

- Volumetric method
- V-notched weir method
- Estimating flow mathematically method

The USGS maintains many gauging stations across the state and their stream-flow information is available in hardcopy and on-line. Discharge measurements using current meters are based on the equation:

Q=AV, where Q=Discharge, A=Area, V=Velocity

It is as important to get good depth readings as it is to get good velocity readings.

How to Calculate Flow

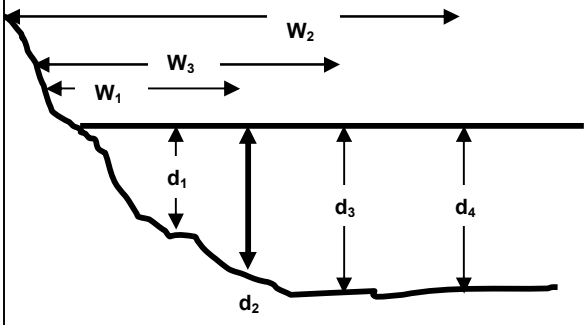
Calculating discharge from each of the width intervals:

$q_2 = v_2 d_2 (w_3 - w_1)/2$

where:
- q_2 = *discharge at width interval 2 (cfs)*
- v_2 = *velocity measure at width interval 2 (ft/sec)*
- d_2 = *depth at interval 2 (ft)*
- w_3 = *distance from the bank or initial measuring point to the point following interval 2 (ft)*
- w_1 = *distance from the bank or initial measuring point to the point preceding interval 2 (ft)*

Calculate the total discharge (flow) as the sum of each of the partial discharges:

$Q = q_1 + q_2 + q_3 + q_4$ é q_i



DWR uses several different current meters and example is shown on Figure 18. The Price meter and the pygmy meter are vertical axis type meters, which use the number of revolutions over a period of time to calculate the water velocity. The Marsh McBirney meter works on the electromagnetics of the water passing by the meter.

Depth-measuring devices are used by DWR include two types of wading rods and a cable-winch bridge board. The top-setting wading rod easily sets the current meter at the proper height. With the other type of wading rod, the depth of the current meter must be calculated and set. The bridge board is used from a bridge handrail or the gunwale of a boat in streams and rivers where the water is too deep or the current is so strong that wading is dangerous or impossible. The winch has a depth-indicating gage.

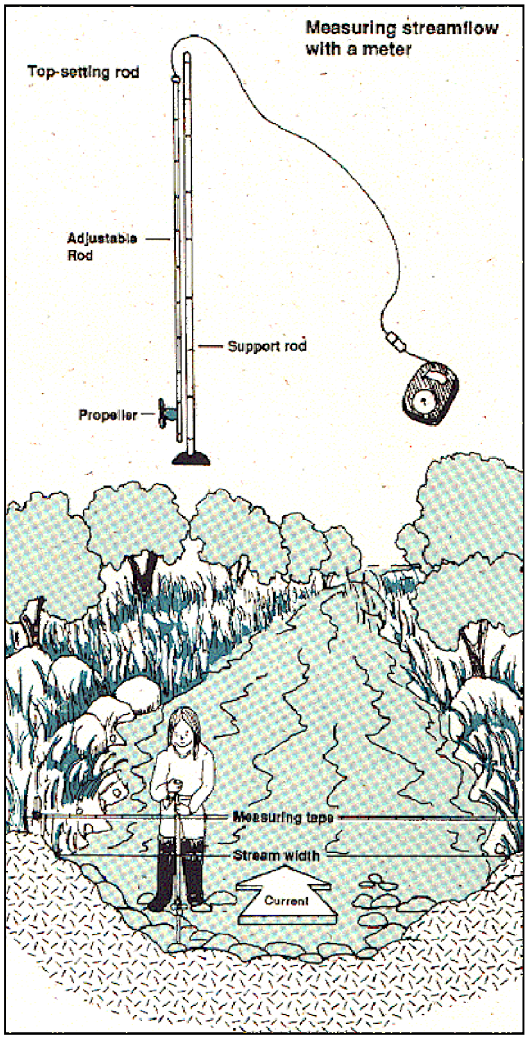


Figure 18. Instream Flow Measurement

2. ESTABLISHING AND USING A REFERENCE POINT

Before measuring any flow a stage reference point (RP) should be found or established. This point can be located on any stationary object over the water surface. Measure the distance from that point to the water surface. It is important to measure this distance before and after doing the flow measurement, as this will indicate any changes in the water level that occurred during the flow measurement.

Many bridges already have an established RP located somewhere on the bridge. These RPs have been established by the USGS and should be used if they can be located. If the RP on the bridge is not located, establish one. Make a mark on a structure over a deep pool near where the flow measurement is made. Clearly identify the mark so that others may use it also.

If many flows are to be done at a station, then a good reference point needs to be used. Each time a flow measurement is performed, record the tape down elevation. As multiple flows are compiled, a relationship between stage and flow can be graphed; eventually allowing flow estimates to be made by measuring the reference point. Occasional flows still need to be performed to make sure the tape down/flow relationship remains constant.

Use a metal tape and dimwap (weight) when measuring the tape down. Stand at the reference point. Let the tape feed out until it barely skims the water surface. Put the tape to the reference point and record the measurement. Be sure to add the length of the dimwap in the measurement.

The reference elevation should be measured to the top of the bolt, the top of the nail head, or to the top of the bridge rail (even if it is beveled). It is important to make your measurements from the exact same point each time.

3. FLOW EQUIPMENT

Price AA current meter
Price pygmy meter
Top-setting wading rod
Headset or beeper box
Stopwatch
100 foot tape measure (in 1/10 ft)
Chaining pins
Stage tape measure with dingwap
Clipboard
Flow sheets
Pencil
Cleaning cloth
Oil
Flagging
Small flathead screwdriver
Hammer and nail
Spray paint (international orange)
Torpedo weights (15 lb., 30 lb.)
Bridge board
Hand calculator

4. FLOW MEASUREMENT PROCEDURE

4.1. Flow Measurement Method

4.1.1. *Pre-Sampling:*

a. Maintain all flow equipment in good working order. Refer to USGS publication, Discharge Measurements at Gauging Stations (Buchanan and Somers 1969).

Note: The spin test is a good indicator of flow equipment readiness.

a. Check condition of flow equipment before leaving the office. An equipment checklist is helpful.

b. Gather all equipment necessary to do the flow, see list in section 3 of this chapter.

c. Select a reach of stream containing the following characteristics:

- A straight reach with the threads of velocity parallel to each other
- A stable stream bed free of large rocks, weeds, and protruding obstructions such as piers, which would create turbulence
- A flat stream bed profile to eliminate vertical components of velocity.
- Wait 15 minutes after moving rocks and vegetation prior to beginning flow measurements to allow stabilization of the stream flow.

d. Establish a stage reference point (RP) (procedure described in following pages).

e. Measure and record the starting stage.

f. Install the current meter on the wading rod, attach the headphones - try a spin test (USGS publication, Smoot & Novak, 1968). Do the headphones click once for every revolution of the current meter? Make adjustments as necessary.

g. Record pertinent information on the flow sheet (See Appendix 7). Include: stream name, date, time start, stage start, location of RP, person doing flow, person recording information, stream conditions.

h. Determine the width of the stream. String a measuring tape across the stream perpendicular to the direction of the flow. Secure the tape on each bank with the chaining pins.

i. Determine how many measurements are necessary to give an accurate total discharge. Measuring velocity at 20-30 equidistant points across the width of the stream is recommended. More measurement points should be chosen

in areas of significant depth or velocity change. Example: More measurements should be made in the area where the flow hugs one stream bank. A rule of thumb is that no area (point) being measured should contain more than 5% of the total flow of the stream.

j. Looking upstream, record from which bank (right or left) the measurements are starting. Record the location on the tape of the starting bank. Example: The left bank starts at 1.5 feet on the tape.

4.1.2. Sampling Techniques

a. Stand downstream and to the side so as not to obstruct the flow of the water to meter.

b. Record the tape measure reading of the point.

c. Measure the depth by placing the wading rod in the stream so that the base plate rests on the streambed. The depth is read from the graduated main rod and is estimated to the hundredth of a foot. Record depth readings on the flow sheet.

d. Use the upper scale of the top setting rod to set the depth of the current meter. At depths of 2.5 feet and less, the average velocity is best measured at a point 0.6 of the depth from the water surface. Using the scale at the top of the wading rod automatically sets the current meter at the desired depth. To set the depth, press the rubber button on the flow rod. This releases the smaller rod. Move the smaller rod until the foot mark on it matches the appropriate tenth marker on the zero to ten scale at the top of the larger rod.

e. At depths greater than 2.5 feet measure the velocity at 0.2 and 0.8 of the water depth (the average of these two velocities will later be recorded as a single value). The top-setting flow rod makes setting these two depths easy. Adjust the top scale readings to one half of the actual depth (this is the 0.8 reading) and to double the actual depth for the 0.2 reading.

f. Start the stopwatch and count the number of revolutions (clicks on the headphones) for at least 40 seconds. Start the stopwatch on count number 0 and stop the watch exactly on a count, not a certain number of seconds.

g. The pygmy meter is rated so that one revolution per second equals to one fps velocity. The Price meter rating is found in the top of the meter box. To use the Price meter table - count a certain number of revolutions: 1, 3, 5, 7, 10, 15, 20, 25, 30, 40 & 50. Compare to the time interval and read the velocity from the rating table. The Price meter also has a connection for measuring very high velocities where a signal is emitted from the meter for every 5 rotations instead of every single rotation.

h. Record the number of revolutions and the time (to the nearest second) on the flow sheet.

i. Move to the next point and repeat steps ñaò-ñ until velocities in at least 20 cross sections have been measured.

j. After the final measurement has been made, record the tape reading of the finishing bank.

k. Measure and record the ending stage.

l. Record the finishing clock time.

m. Replace the current meter pivot with the traveling pin and return meter to box to prevent damage while traveling.

5. BRIDGE BOARD METHOD

5.1. Bridge Board Sampling Techniques and Supplies

5.1.1. *Equipment Needs*

- Clipboard with flow sheet and pencil
- Measuring tapes
- Duct tape
- Stage tape and dimwap
- Traffic safety cones and vests
- Bridge board assembly
- Price AA current meter (with tailpiece)
- Torpedo sounding weights (15 lb., 30 lb.)
- Headphones
- Stopwatch

5.1.2. *Bridge Pre-Sampling Setup*

(Refer to Buchanan & Somers, 1969, USGS publication)

a. Assemble bridge board equipment. This involves attaching the assembled Price AA current meter and the appropriate torpedo sounding weight to the hanger end of the winch cable. The current meter's position on the hanger is dependent upon which torpedo weight is used.

b. Attach the headphone jacks to the output terminals of the winch.

c. Do a spin test to ensure that current meter is working properly.
d. Determine the width of the stream and secure the measuring tape to the upstream handrail of the bridge with duct tape. More than one 100-foot tape may be necessary.
e. Determine the distance interval of the 20 - 30 points necessary to make an accurate measurement. Example: Measure velocity every 2 feet on a 50-foot wide stream. Be ready to change the interval if velocity or depth changes significantly. Remember, no more than 5% of the flow in any one interval.
f. Fill out flow sheet information (refer to step in section 4.1.1- ñhò) of the current meter procedure).

5.1.3 *Bridge Sampling Techniques*

a. Measure the tape down from the reference point.
b. Record the tape reading from the starting stream bank.
c. Move to the first point to measure velocity. The bridge board rests on the handrail (guardrail) of the bridge.
d. Zero the winch depth indicator by lowering the current meter until the cups of the meter are half in the water. Pull the zeroing armature out and rotate until the depth indicator reads zero. Because most bridge handrails are not level, make sure to zero the winch depth indicator at each point that a velocity measurement is made.
e. Measure the depth of the water. To do this, lower the current meter until the cable goes slack. This indicates that the torpedo weight has hit something. Raise and lower the meter a couple of times to get a consistent depth reading for the bottom.
f. Add 0.5 foot to the reading to get the actual depth of the water. This accounts for the torpedo weight that hangs 0.5 foot below the current meter (which was zeroed at the cups). Record the actual depth.
g. At depths less than 2.5 feet, measure the velocity at 0.6 of the water depth (measured from the surface).
h. At depths greater than 2.5 feet, measure the velocities at 0.2 and 0.8 of the water depth.
i. Lower the current meter to the calculated depth.
j. Measure the velocity by counting clicks (revolutions) for at least 40 seconds. Refer to velocity measurements using the Price AA current meter.
k. Move to the next point and repeat steps ñdò-ñkò
l. After the last velocity is measured, record the measurement of the finishing stream bank.

m. Record the finish time.
n. Measure and record the ending stage.
o. Store all flow equipment properly.
p. Compute the total discharge.

6. BOAT FLOW MEASUREMENT METHOD

Due to the danger because of boat traffic, extreme care should be taken when setting up for boat measurements. Locations where boat traffic is minimal should be chosen and any boats in the area should be warned off.

6.1. Boat Flow Sampling Techniques and Supplies

6.1.1. *Equipment Needs*

- Boat
- Rope (> width of stream)
- Bridge board assembly
- Headphones
- Stopwatch
- Clipboard with flow sheet and pencil
- Hand calculator
- Measuring tape - 100 foot
- Duct tape
- Stage tape and dingwap
- Cross piece assembly

6.1.2 *Boat Flow Pre-Sampling Setup*

(Refer to Buchanan & Somers, 1969, USGS publication Stations for more detailed instructions)

a. Assemble bridge board equipment.
b. Make a spin test.
c. Prepare the boat for work. It takes a minimum of two people in the boat, one to operate the bridge board and one to calculate the meter depths and record the flow information. The cross piece attaches to the bow and holds the boat in position on the rope.
d. Stretch and secure the rope across the stream channel, just over the water surface and perpendicular to the flow direction. Use duct tape to attach the measuring tape to the rope (an alternative is to use a rope marked at regular intervals).
e. Fill out flow sheet information.
f. Determine the interval of sampling points.

6.1.3 *Boat Flow Sampling Techniques*

a. Record the tape measure reading at the starting stream bank.
b. Measure the velocity at first point using the following steps. The bridge board rests on the gunwale of the boat.
   - Zero the winch depth indicator.
   - Lower the current meter until slack in the cable indicates the bottom.
   - Add 0.5 foot to the reading (to correct for the weight that hangs 0.5 foot below the meter).
   - Calculate the depths to set the current meter at 0.2, 0.8 (see step section in 4.1.1- ñò) of current meter procedure).
   - Set the depth indicator at the proper depth.
   - Record the number of revolutions and the time interval at each depth.
   - Move boat to next point.
   - Repeat the steps under 6.9 until the entire stream has been measured.
c. Record the tape measure reading of the finishing stream bank.
d. Record the finish time.
e. Compute the total discharge.

7. V-NOTCH WEIR METHOD

7.1. V-Notch Sampling Techniques and Supplies

7.1.1. *Equipment Needs*

- V-notch weir (> stream channel width)
- Vertical staff gage
- Carpenter's level
- Hammers (3 lb., 8 lb. sledge)
- Straight edge
- Graduated container
- Stopwatch

7.1.2. V-Notch Flow Pre-Sampling Setup

a. Determine a good location for the weir plate. Avoid hard rock or loose sandy stream bottoms. Also avoid riffle areas where faster velocities erode the weir plate.
b. Set up the vertical staff gage. The gage should be located in the upstream pool formed behind the weir. Use the carpenter's level to make sure that all faces of the gage are level. Because of the effect of drawdown, the gage should not be located too close to the weir plate.

c. Use the sledge hammer to pound the weir plate into both banks so that the plate dams up all flow in the channel. Use the carpenter's level on all faces of the plate, it must be level. Make sure also that there is no water flowing around or under the weir plate.

7.1.3. V-Notch Flow Sampling Procedures

a. Determine the zero point of the weir (very important reading):

- Use the straight edge and level to measure a level line from the base of the angle on the weir plate back to the staff gage. Or if the distance from the top of the weir plate to the base of the angle is predetermined; then a level line is measured from the top of the plate to the gage and the distance subtracted.
- Read the water level on the staff gage just at the point where the water starts to flow through the notch in the plate.
- Let the water flowing through the V-notch stabilize.

b. Read the staff gage.

c. Determine the difference between the zero point on the staff gage and the water level flowing through the weir plate (read as the water level on the staff gage). This difference is known as head.

d. Look at the flow table for the V-notch weir. Look up the corresponding flow for the particular head height. An alternative to using the flow table is the volumetric method (procedure described in following pages).

e. Periodically clean the weir to prevent the buildup of sediments or solids around the notch. This buildup will affect the accuracy of the weir. Leaves are a problem to a V-notch weir. A leaf stuck at the base of the weir angle can cause a significant rise in water level.

8. VOLUMETRIC METHOD

8.1. Volumetric Sampling Techniques and Supplies

8.1.1. *Equipment Needs*

- Graduated container
- Stopwatch

8.1.2. *Volumetric Flow Procedure.*

a. Mark the container to a known volume (examples: 1 gallon, 1 liter).
b. Place the container under the discharge, collecting all flow.
c. Time the interval needed to fill container to the volume mark.
d. Empty the container.
e. Repeat steps ñbòñdòseveral times.
f. Average timed results.
g. Calculate the flow rate as flow volume/time. Example: 1 gallon in 15 seconds.
h. Convert flow to cfs or MGD.

9. MARSH MCBIRNEY MODEL 201 CURRENT METER

The principles of using the Marsh McBirney current meter are the same as using other current meters. The meter employs the velocity/area method of flow measurement. The sensor probe detects water velocity. The panel meter reads velocity in feet per second. The procedure for using the Marsh McBirney meter is the same as that described in Section 4 (Flow Measurement Procedure) but no calculations are needed as the flow is directly read on the instrumentôs screen.

This meter is used to measure small flows/low velocities. Because the probe has no moving parts, debris in the water has little effect on the reading. Another advantage is that the sensor probe attaches to the top-setting flow rod.

10. FLOW SHEET CALCULATIONS

10.1 Data Form

10.1.1. *Data to be Recorded on Data Sheet*

- Distance from the initial point
- Depth
- Time (in seconds)
- Revolution

10.1.2. *Calculations*

a. Columns titled, velocity (mean in vertical), area, width, and discharge are calculated values.
b. Calculate the width of each cross section. The width of the section is the sum of one-half the distance from the point of measurement to each adjacent point. Example: In the first

section the width is one-half the distance to the adjacent point plus one half the distance to the stream bank.

c. Calculate the velocity in each cross section. In depths of greater than 2.5 feet, two velocity measurements are taken in each cross section (0.2, 0.8). Average the two readings and record in column. This depends on the current meter in use:
   - The Price pygmy meter - the number of revolutions divided by the seconds.
   - The Price AA meter - The velocity is taken directly from the meter rating table. Not all Price AA meters use the same rating table-be sure that you have the correct table for the meter you are using.
   - The Marsh McBirney meter - The velocity is read from the panel as feet per second.

d. Calculate the cross-sectional area. Multiply the width times the depth.

e. Calculate the discharge of each cross section. Multiply the cross-sectional area by its velocity.

f. Calculate the total discharge of the stream. Add all the cross-sectional discharges together.

g. Record the average velocity of the stream. Divide the total discharge by the total area.

11. OPEN CHANNEL FLOW MEASUREMNT METHOD

11.1 Introduction

The following section provides a brief overview of methods for determining flow in an open channel. For more detailed information regarding open channel flow measurements, refer to the references section.

Open channel flow can be defined as flow in any channel in which liquid flows with a free surface. Open channels are generally used in moving fluids at most municipal treatment facilities, industrial waste treatment operations and in most irrigation applications. An open channel can also be a stream or a ditch. Open channel flow is typically measured by the use of a calibrated restriction device placed in the channel that affects the surface level of the liquid as it moves past the restriction. This type of open channel measuring device is referred to as a "primary" device. The known dimensions and physical characteristics of the restriction device are used to correlate a relationship between water surface level and flow. After the water level/flow relationship has been established, the flow in the open channel can be easily measured by manually sighting the height of the liquid's surface level against a calibrated scale (staff) and then referring to the appropriate rating curve or table.

The following are the most commonly used types of "primary" open channel flow measuring devices, (restriction devices):

11.1.1 Weir: a dam constructed across an open channel, over which liquid flows through an opening or notch. The most commonly used types are rectangular, trapezoidal and triangular.

11.1.2. Flume: a specially shaped open channel, designed to change the channel area or slope, resulting in an increase velocity and surface level of the liquid flowing through it. The most commonly used types are: Parshall and Palmer-Bowlus

11.2 Flow Meter

a. A flow meter is a mechanical device used to measure the liquid level in the channel and convert the level into a corresponding flow rate

b. A stage recorder is a mechanical device used to record the surface level of the liquid over a period of time

Note: Measuring flow in an open channel by means of a weir or flume is a simple function of surface level and is the most basic and inexpensive method available. However, if continuous stage or flow recording is required, then the use of a stage recorder and/or a flow meter in conjunction with the primary device may be necessary. Some of the more commonly used methods employed by these devices to determine the surface level of a liquid are floats, dipping probes, ultrasonic sensors, and bubblers.

IX. BATHYMETRY

1. PROCEDURES

Recording fathometers are used to provide bathymetric traces of water depths. Since water depths are time dependent (especially in tidal areas) the date and time of all traces should be noted. Operating manuals provide operation and calibration procedures to be followed. In particular, tide and draft adjustments provide calibration in regard to the respective tidal amplitude and sensor probe depth. All traces should be noted with transect description, chart speed, direction of travel, and pertinent reference points and then indexed to a site map. When working in tidal areas, a water stage recorder should be positioned to provide a histogram of water levels to correlate with the bathymetric trace.

During the initial setup of each survey, the fathometer calibration should be checked against a field measurement of water depth made using a graduated sounding line.

2. EQUIPMENT AVAILABLE

The following equipment is available for bathymetric surveys:

- Water level recorder and/or referenced gauging stations(s)
- Depth gauge
- Calibrated sounding line(s)

3. SPECIFIC EQUIPMENT QUALITY CONTROL PROCEDURES

Number all equipment and keep a record of maintenance and calibration procedures. Use the following steps to maintain and calibrate bathymetric measurement equipment:

3.1. Recording fathometers:

3.1.1. Calibrate and maintain according to the manufacturer's instructions before use. The chart speed should be checked against a reliable time source before the instrument is sent to the field.

3.1.2. Check daily in the field against a field measurement of water depth using a calibrated sounding line.

3.1.3. Clean daily after use and before storing.

3.2. Sounding lines are to be calibrated against steel surveyor's chain and shall be accurate to 0.1 foot.

X. WATER QUALITY VESSEL OPERATION

Water quality investigations frequently require DWR personnel to work in locations that are accessible only by boat. This necessitates that field staff be thoroughly trained in the safe operation of those boats and become familiar with the general maintenance and the particular operation of each vessel. This boating SOP provides a general operating guide to ensure that all boating and trailering activities are carried out in a safe manner and that all boats and motors are operated in a manner that reduces the frequency of repair. All field personnel should read and thoroughly understand this SOP prior to operating any DWR boat.

1. BOAT SAFETY

1.1. Supplies Needed On-Board

1. ***Fire Extinguishers*** - before operating boat, familiarize yourself with where the fire extinguisher is located. Check to make sure that it is fully charged.
2. ***Sound Producing Devices*** - boats should be equipped with a can type air horn or a manually operated whistle.
3. ***Paddles or Oars*** - all boats should be equipped with oars or paddles.
4. ***Visual Distress Signals*** - when operating boats in coastal waters, the boat must be equipped with a flare kit. The kit should include hand held flares and a flare gun for aerial type flares.
5. ***PFD's (Personal Floatation Devices)*** - **all DWR employees are required to wear life preservers at all times while on board DWR boats.** Boats will be equipped with a type 1, 2, or 3 PFD of suitable size for each person on board and a throw-able floatation device (throw cushion, flotation ring).
6. ***Lights*** - when operating a boat at night, the boat must display the front green and red navigational light and the rear beacon light. If planning to operate at night, the lights should be checked before leaving the loading area.

1.2 Safety Check

1. ***Weather*** - check weather reports before leaving shore and remain watchful for signs of bad weather. Tune into the National Weather Service Report, on a Marine radio, periodically to check weather conditions, small craft advisories, gale warnings, etc. Do not go out on the water during lightning storms.
2. ***Care and Maintenance*** - all equipment and supplies should be properly secured. Keep decks and other spaces clean, free of clutter and trash. The vessel should be free of fire hazards with clean bilges and in good condition. Inspection and required maintenance on a regular schedule will ensure the hull and superstructure remain sound.

Ensure all repairs are made properly and with marine rated parts. Always carry a toolbox and know how to make minor repairs.

3. ***Communications*** - when operating in remote areas it is always a good idea to bring along a cellular phone for cases in which assistance may be needed. Two-way radios should be used when operating with two or more boats. When operating in coastal waters always bring along either a hand-held portable marine radio or a fixed mounted marine radio.

2. FIXED MOUNT/CONSOLE TYPE BOATS

2.1. Trailering

2.1.1. *Pre-Trip Check and Preparation*

a. Install 1 Ō or 2" trailer ball to trailer hitch depending on the trailer.
b. Unscrew clamping mechanism on boat trailer tongue.
c. Back vehicle up to boat trailer, with trailer tongue directly over the center of the trailer ball.
d. Lower trailer jack so that the trailer tongue fits over the trailer ball.
e. Screw down, or tighten, the clamping mechanism (all the way) onto the trailer ball. Lock with a 2640 Master Lock.
f. Hook up both safety chains by crossing the chains and hooking to holes on the trailer hitch. Do not tow boat without safety chains.
h. Hook up brake line cable to eye bolt attached to the vehicle.
i. Plug in trailer lights and check the lights for proper operation.
j. Secure gunwhale boat strap.
k. Check the bow eye to make sure safety chain is hooked up and winch is locked down securely.
l. Periodically check the clamping mechanism on the trailer tongue to assure that it is screwed down all the way.
m. Conduct an inspection walk around the boat and trailer:
    1) Test to see if the boat motor starts before traveling.
    2) Check level of the engine oil on 4-cycle boat motors.
    3) Check the trailer tire pressure and adjust if necessary.
    4) Check the condition of the axel grease. Add grease as needed.
n. When traveling, stop and check the trailer and boat; retighten boat straps as needed. Feel the trailer bearings to see if they are hot. If hot, they probably need to be greased or replaced.
o. Trailer slowly over speed bumps and holes.

p. When backing into parking areas, do not let back of trailer come in contact with curb to avoid damaging license plate bracket or trailer lights.

2.2. Boat Launching

2.2.1. *Unloading*

a. Upon arrival at the boat ramp check the ramp to make sure it is suitable for launching including checking that the water level is high enough for proper launching.
b. **Install all the boat plugs**; check inside the bilge to make sure that the plug is installed.
c. Remove the boat strap.
d. Load the boat with equipment.
e. Unplug trailer lights
f. Make sure the motor is in the "up" position before launching the boat.
g. Keep winch "locked" until boat is in the water.
h. When the boat is in the water lower the motor and then start the motor (see motor operating instructions).
i. While one person is operating the boat another person should be manning the trailer winch.
j. Unhook winch cable from bow eye, but do not remove safety chain until the boat is running and idling.
k. If needed, back the vehicle up slightly and press the brake to bump the boat off the trailer.
l. When boat is clear from the trailer, pull the vehicle out of the ramp **slowly** and park it.

2.2.2. *Loading*

a. Slowly back the trailer into the water so that the center "guide roller" is visible above water.
b. Line up the boat with the trailer and **very slowly** ease the bow of the boat onto the center roller. If boat is off center of the trailer, back up and try again.
c. **Do not** approach trailer at a speed that will damage the boat hull or trailer if the center roller is missed!
d. The person manning the trailer winch should signal the boat operator to go left or right, or to tell the boat driver to back off if they are going to miss the center roller.
e. Once the bow is on the center roller, slowly advance the boat up onto the trailer as far as it will go. If it does not reach the stanchion then hold the boat in position until the person manning the winch can get out enough cable to hook to the bow eye.

f. Once the cable is hooked and tension is maintained then power down the motor and cut it off.
g. Winch the boat onto the trailer until the bow is snug against the stanchion.
h. Lock down the winch gear.
i. Raise the motor to the "up" position and flip down the tilt lock bar, then lower the motor until it presses against the tilt lock bar.
j. **Slowly** drive out of the boat ramp to the parking lot.
k. Remove the boat plugs.
l. Unload the boat.
m. Hook up boat strap.
n. Plug in trailer lights.
o. Make sure that all aerials and/or bimini tops are down.
p. Walk around trailer and **double check everything!**

2.3. Boat Operation

2.3.1. *Fueling*

a. Fill oil reservoir to required fill level with appropriate motor oil.
b. When fueling boats with 2 cycle outboard motors without an oil reservoir add one pint of 2 cycle outboard motor oil to the gas tank for every six gallons of gasoline. Use marine fuel stabilizer in all fuel tanks.
c. Fill tanks to their maximum fill level.

2.3.2. *Motor Operation*

a. Switch the battery "PERKO" switch to the "ALL" position.
b. Pump the gas primer ball until it is tight.
c. Lower motor into the water using hydraulic trim switch on the throttle lever.
d. When starting "cold" the choke must be engaged. To engage choke, push the ignition key in as far as it will go, then turn the key clockwise until the motor starts. If motor does not start within five seconds **do not** continue to engage the starter. Re-pump the primer ball and try again.
e. Once the motor starts disengage the choke and let the motor idle.
f. Once the motor has been given ample time to warm up, back the boat off the trailer.
g. Let motor idle down before changing from reverse to forward. Between forward and reverse, make a brief stop in neutral.
h. If working in open water with ample depth for boat running, advance the throttle to plane out the boat, adjusting the trim if necessary.

i. Once proper plane is achieved, throttle the boat back to 3/4 (approximately 4200 rpm) throttle. **Do not run the boat at full throttle.**

3. SMALL BOATS WITH PORTABLE MOTORS

3.1. Trailering

For the most part the same rules apply that were covered in the previous section with a few exceptions:

a. Install appropriate sized trailer ball to trailer hitch.

b. Flip down locking switch on trailer tongue and lock with a 2640 Master padlock.

3.2. Boat Operation

3.2.1. *Fueling*

a. Obtain gas tank from storage cabinet.

b. Make sure the tank selected is equipped with the proper fuel line connections for the motor you will be using.

c. Most of the fuel tanks have a capacity of 6.6 gallons. Leave some head space in the fuel tanks - **do not overfill**.

d. Add one pint of 2 cycle outboard motor oil for every six gallons of gasoline unless motor requires different oil and ratio.

3.2.2. *Motor Operation*

a. Select the proper motor for the boat that is to be used.

- 5 hp for Jon boat and 12' Alumacraft,
- 15 or 25 hp for 14' Alumacrafts.

b. Place outboard motor in the **center of the transom** and completely tighten the clamping screws on the outboard motor.

c. Place tank in boat and connect to motor to assure proper fitting.

d. Run a chain through the gas tank handle, then through the handle on the outboard motor, and then through the hole in the boat and lock with a pad lock.

e. Secure the motor to one side with a bungee cord to keep motor from swaying back and forth while going down the road.

f. To run: connect fuel line to motor, and pump primer ball.

g. Pull choke knob if motor is "cold".

h. Turn tiller throttle lever to "start" position.

i. Pull starter cord. If motor does not start after one or two pulls then pump the primer ball and try again.

j. Once motor starts push choke lever in and let run for about a minute then idle down with throttle lever.

k. To put motor in gear, make sure motor is idling low and pull the gear lever forward to go in a forward direction. To go in reverse, push gear lever backward to reverse position with a brief stop in neutral.

l. Once motor is in gear then throttle up the motor, once the boat is planned out back off of the throttle about 1/4 turn.

m. **Do not run the motor at full throttle, run at 3/4 throttle.**

n. To turn the motor off, press the red kill button located next to the choke lever. On some motors the kill button is located on the end of the tiller.

4. TROUBLESHOOTING: FOR ALL BOATS

4.1. Problem - **No power to starter**

a. Check to see if "PERKO" switch is on the "ALL" position.

b. Check to see if throttle lever is in neutral.

c. Check battery terminal connections.

d. Check main fuse in outboard motor.

e. Check fuses inside console.

4.2. Problem - **Motor is Turning Over But Will Not "Fire" or Start**

a. Check to see if gas line is connected to motor.

b. Check to see that primer ball has been pumped until tight.

c. Check to make sure "deadman's" or kill button is clipped.

d. Make sure air vent screw is open on gas can.

e. Check spark plug wires, replace spark plugs (they may be fouled)

4.3. Problem - **No Water is Coming Out of Flow Hole**

a. **Do not** continue running motor. Shut down and attempt to unclog flow hole with a coat hanger or similar object.

b. If problem persists, do not use boat. If out on the water when this occurs, get towed back to boat ramp.

4.4. Problem - **Extreme Cavitations or "Porpoising**"

a. Adjust trim with up and down button on throttle lever.

b. Make sure there is not an excessive amount of water in bilge.

c. Adjust the weight distribution of equipment and personnel in the boat (trim the boat up and shift weight).

XI. LAKES SAMPLING

Field data collection procedures for reservoirs and lakes differ from that of streams and rivers due to the differences in water depth and hydrology. This section focuses on procedures specific to physical and chemical water quality sampling of lakes.

1. FIELD PREPARATION

1.1. Pre-Sample Preparation

a. Preparation of the lake sampling packet.
    1. Sample tags and lab sheets must be legibly hand written with permanent black ink. Adhesive labels for the sample tags may be prepared on a laser printer.
    2. Include maps showing the locations of the sampling stations. Electronic copies of lake maps are on the ISB shared drive (Lake Maps folder).
    3. Include a copy of the Field Observation form (Figure 19).
    4. Include special instructions and point-of-contact information as needed.

b. Contact responsible parties at all publicly owned lakes several days in advance of sampling. Contact names are in the particular lakes file or in the Lakes Database. Changes in contact information will be noted and provided to the Lakes Database Administrator so that the database can be updated.

c. Confirm availability and working condition of boats, motors, vehicles, and Hydrolab/YSI.

d. Verify the lake stations on the map with the station numbers on the lab sheets and tags.

e. Always include extra bottles in case of accidents, defective bottles, and/or discovery of algal blooms or other environmental conditions that justify additional samples.

1.2. Field Equipment Needed

Aquatic Plant & Algal Bloom Report Forms	Cooler(s) with ice
Field Observation & Stratified Data Forms	Lab Sheets and Tags in sealed bag
Preservatives- Lugols solution, H_2SO_4, HNO_3	Hydrolab/YSI Meters
Labline with Rope	Camera
Sample Bottles	Calibrated backup meters
Pens and Pencils	Maps
Life Jackets	Boat Oars
Gas Tank for boat	Boat Plug & Anchor
Winter- cold weather suit (as needed)	First aid/safety box

Electric motor & 2 fully charged batteries (if needed)
Secchi Disc with line marked in 1 centimeter increments

Calibration Materials-(*e.g. Calibration and D.O. sheets, pH and conductivity standards, meter manuals).*

1.3. Field Sheets

a. *Stratified Field Sheets*: Make sure that stratified field sheets (Figure 5, page 25) are carried to the lake stations along with pens or any non-erasable ink for writing and a clipboard. Clearly write the station number, date, time, depth, dissolved oxygen, temperature, pH, conductivity, and secchi are recorded on the field sheet. At the top of the field sheet record, the name of the water body, which meter is used, and the names of the field samplers is also recorded.

b. *Field Observation* Form: In addition, a separate form is to be filled out for all of the ambient lakes (Figure 19). This form requests information about the use support status, restoration activities, weather conditions, the watershed, and lake water quality.

c. After sampling, both of these forms are filed in the current lake files in the Intensive Survey Branch. Data are entered into the lakes database within 72 hours of the lake trip.

FIELD OBSERVATION FORM Lake Name _____
Page 1 of 2 SamplersôName: _____Date: _____

WEATHER CONDITIONS

Air Temperature	% Cloud Cover	Wind Direction (from)	Wind Velocity	Rainfall (last 48 hrs)
__ <60º	__ 0-25%	_____	__ <10 mph	__ None
__ 60-70º	__ 25-50%		__ 10-20 mph	__ < ¼ inch
__ 75-90º	__ 50-75%		__ >20 mph	__ ¼ - 1 inch
__ 90º	__ 75-100%			__ >1 inch

SHORELINE AND WATERSHED OBSERVATIONS
Please describe *development* around the lake shore:

Type of Development	Density/Intensity	% of Shoreline Developed
__ Residential/Urban	__ Slight	__ 0-25%
__ Commercial/Industrial	__ Moderate	__ 25-50%
	__ heavy	__ 50-75%
		__ 75-100%

Please check *land uses* observed in the watershed:
__ Agriculture (specify if possible)
__ Crop production
__ Pasture land
__ Feedlots/Animal production
__ Forest
__ Wetlands
__ Urban/Residential
__ Commercial/Industrial

LAKE QUALITY
Please check the one statement that best describes the *physical condition* of the lake water today:
__ Crystal clear water.
__ Not quite crystal clear, a little algae/suspended sediment visible.
__ Definite algal greenness, yellowness, or brownness apparent.
__ High algal/sediment levels with one or more of the following: floating scums on lake or washed up on shore; strong foul odor; or fish kill.

Please check the one statement that best describes the *aquatic macrophyte* community:
__ None observed
__ Small amount of vegetation evident along shoreline and/or headwaters of the lake; <25% of the lakeôs total surface area covered.
__ Macrophytes extend out from shoreline well into the lake; 25-50% of the surface area covered.
__ Dense growths of several species cover more than 50% of the surface area.
__ Nuisance levels of a singe species cover more than 50% of the surface area.

Please check the one statement that best describes your *opinion* of how suitable the lake water is for recreation and aesthetic enjoyment today:
__ Beautiful, could not be nicer.
__ Very minor aesthetic problems; excellent for swimming, boating, enjoyment.
__ Swimming and aesthetic enjoyment slightly impaired because of levels of algae/sediment/or weeds (please indicate which).
__ Desire to swim and level of enjoyment of the lake substantially reduced because of algae/sediment/or weeds (please indicate which).
__ Swimming and aesthetic enjoyment of the lake nearly impossible because of algae/sediment/or weeds (please indicate which).

Figure 19. Field Observations Form

LAKE NAME: _____
Page 2 of 2

Designated Use Classification: _____

_____

Supplemental Classification: _____

USE SUPPORT STATUS
Designated uses appear to be:
__ Fully supported.
__ Fully supported, but threatened (impairment could result if pollution controls are not implemented).
__ Partially supported
__ Not supported

If uses are not fully supported, what pollutants or conditions are *causing* impairment (check all that apply):
__ Nutrients __ Noxious aquatic plants __ Other (please specify)
__ Situation __ Organic enrichment/low DO
__ Flow alteration __ Thermal modification
__ Suspended solids __ Filling and draining

If uses are not fully supported, what *sources* of pollutants contribute to use impairment:
POINT SOURCES
__ Industrial
__ Municipal
__ Municipal pretreatment
__ Other point sources (specify)

NONPOINT SOURCES
__ Agriculture (specify if possible)
__ Crop production
__ Pasture land
__ Feedlots
__ Aquaculture
__ Other (specify)
__ Silviculture
__ Construction/Land development
__ Urban runoff
__ Mining/resource extraction
__ Land disposal of waste (e.g. landfills, wastewater and sludge application, on-site septic tanks, etc.)
__ Hydrologic/habitat modification (e.g. canalization, dredging, flow regulation, etc.)
__ In-place contaminants
__ Recreational activities (e.g. motor boating)
__ Other nonpoint sources (please specify):

__ Source of impairment unknown

RESTORATION ACTIVITIES
Please describe any lake restoration or water quality management activities that have taken place:
_____
_____
_____
_____

Figure 19. Field Observations Form (continued)

2. LAKE DATA COLLECTION

2.1 Lake Physical Data Collection Methods

a. Secchi depth measurement is taken as described in Chapter III, Section 6.

b. Dissolved oxygen, water temperature, conductivity and pH are measured with a multiprobe (Hydrolab) meter beginning at the surface of the lake (0.15 meters from the surface).

c. From the surface to either the bottom of the lake or to a depth of 10 meters, physical measurements are recorded at 1- meter increments

d. Below ten meters, physical measurements are recorded at 5-meter increments until the bottom of the lake is reached

2.2. Lake Water Sample Collection

2.2.1 *Description*

a. Samples will be collected at the surface, photic zone, or at the bottom, which are described below and in detail in Chapter I.

b. If "SUR" is part of the station number, the sample is to be collected at the surface. If 'BOT" is part of the station number, the sample is collected one foot above the bottom. If "SUR" or "BOT" doesn't accompany the station number, the sample is collected in the photic zone.

c. As with all samples for laboratory analysis, a lab sheet must be completed as described in Chapter II,Section 1 of this SOP.

d. Detailed definitions of these parameters and methods of collection found in Chapter I, Section 3.

2.2.2. *Types of Typical Lake Samples*

a. **Surface Grab** samples (chloride, hardness, fecal coliform bacteria, and metals) are collected 0.15 meters below the water's surface and this can be done by hand dipping the bottle. The bottle top and bottle opening should be protected from contamination. Grasp the bottle near the base and plunge it mouth down into the water, avoiding surface scum. Position the bottle away from the hand of the collector, the shore, the side of the sampling platform, or boat.

b. **Photic Zone** ï the photic zone is defined as the column of water in the lake from the surface down to a depth equal to twice the secchi depth measurement. Photic zone samples (residue, turbidity, chlorophyll ***a***, nutrients, and phytoplankton) are collected by raising and lowering the Labline at a steady speed within the photic zone until it is full. A description of this procedure is given in this SOP in Chapter I, Section 3.2.3.

c. **Bottom sampling** (nutrients) is accomplished by inserting the two plugs in the top of the Labline and lowering it just above the bottom of the lake. This needs to be done gently as not to stir up sediments on the bottom. The plugs can be released by firmly jerking the rope on the Labline. Wait until the Labline if full before bringing it back to the surface. This can be determined by observing air bubbles from the sampler rising to the lake surface, the stopping or feeling the weight of the Labline at the end of the rope.

2.2.3 *Field Records and Information*

a. **Photographs** are to be taken of various locations on each sampled lake to record the shoreline and lake characteristics. In particular, an unusual shoreline/ watershed activity, aquatic plants, algal blooms, or other water quality issues are to be photographed (photo number and brief description and location of where picture was taken) must be made. This information along with the camera, are to be returned to the Lakes Database Administrator upon return to ISB.

b. **Comments and questions** from citizens, lake managers, water treatment plant supervisors, etc. are to be recorded (written) along with contact information and individualÕs name and title (if any). This information will be submitted to the the Lakes Database Administrator upon return to ISB.

2.3. Typical Lake Sampling Parameters

Below is a list of typical lake water sample types. Descriptions on how samples are preserved and collected are found in Chapter I, section 3, section water samples as well as more detailed descriptions can be found in this SOP in the Sample Collection Section (Chapter IV).

2.3.1. *Physical Parameters include*

Conductivity	Dissolved Oxygen (mg/L)
pH	Temperature (°C)
Secchi Depth	

2.3.2. *Chemical Parameters include*

Nutrients	Residue
Turbidity	Chloride
Magnesium	Calcium
Metals	Chlorophyll *a*

*Additional parameters may be collected based on specific lake conditions and/ or requests

2.3.3. *Biological Parameters*

a. Fecal coliform bacteria: Water samples are collected at the surface of the lake.

b. Phytoplankton: Water samples are generally collected as a photic zone sample. Bloom samples may be collected at the surface of the lake, as needed.

c. Aquatic Plants: Use the Aquatic Plant Report Form supplied by the Ecosystems Branch of the Environmental Sciences Branch and submit it along with a specimen if there appears to be problematic aquatic plants or for identification. Refer to the Aquatic Plant Report Form for collection and preservation of aquatic weeds. Include a map of the location showing where the plant specimen(s) were collected.

d. AGPT (Algal Growth Potential Test): These samples are collected after consultation with EPA since they perform the tests. The bottles (1 liter) are furnished by EPA as are the tags and coolers. The samples are collected in the photic zone and no preservative is used. The samples are shipped back to the EPA Athens, GA laboratory for analysis. The address and telephone number is: Bob Quinn, U.S. EPA, Region IV, Environmental Services Division, Athens, Georgia 30613, (706) 546-2420.

2.3.4 Lab and Field Sheets: All lab and field sheets should be **legibly** filled out with applicable dates, times, depths, etc.

a. The same time is recorded on both field and lab sheets for the same station. A field observation sheet should also be filled out and any other notable features recorded.

b. Any notes of unusual observations of lake water quality or shoreline activities that could impact water quality should also be submitted.

c. Field sheets and filed observations sheets along with camera are to be submitted to the Lakes Database Manager upon return from the field.

3. LAKE DATA MANAGEMENT

3.1 Data specific to the Intensive Survey Branch Lake Monitoring Program are warehoused in the Lakes Database. This database is maintained by the Lake Database Administrator. The responsibility of the Lake Database Administrator includes entry of data, verification data entry accuracy and reporting issues related to the functioning of the database to the ESS IT staff.

a. Physical field data are entered into the ISB's Lakes Database within 24 hours of receipt from the field sampling team.

b. Chemistry results from the DWR laboratory are entered into the Lakes Database within 72 hours of receipt from the laboratory.

c. Lake data which have been entered into the Lakes Database but not checked for input accuracy and/or completeness are designated ôPô for ôProvisionalô

d. Lake data which has been reviewed and verified for input accuracy and completeness are indicated with the designation ôAô for ôAcceptedô

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XII. SEDIMENT OXYGEN DEMAND

1. GENERAL DESCRIPTION OF SOD TEST

Sediment Oxygen Demand (SOD) is one of the more significant variables in water quality modeling evaluations for determining stream assimilative capacity. SOD data are primarily used for waste-load allocation purposes in the evaluation of receiving waters.

The SOD test involves placing an SOD chamber on the bottom sediment, securing it to prevent water infiltration and monitoring oxygen change within the chamber. A dissolved oxygen sensor inside the chamber measures the rate of decrease in oxygen that is used by organic materials in the bottom sediments over a given period of time. A standard SOD test includes seven SOD chambers of which two are water column control (blank) chambers and five are replicate SOD chambers (Figure 20). The blank chambers, used to determine water column respiration rate, have bottom plates that prevent bottom sediment from contacting the water in the chamber. The SOD replicate chambers have open bottoms allowing the internal water to circulate over the bottom sediment. The rate of oxygen change in the replicate SOD chambers minus the water column respiration of the blank chambers equals the SOD rate.

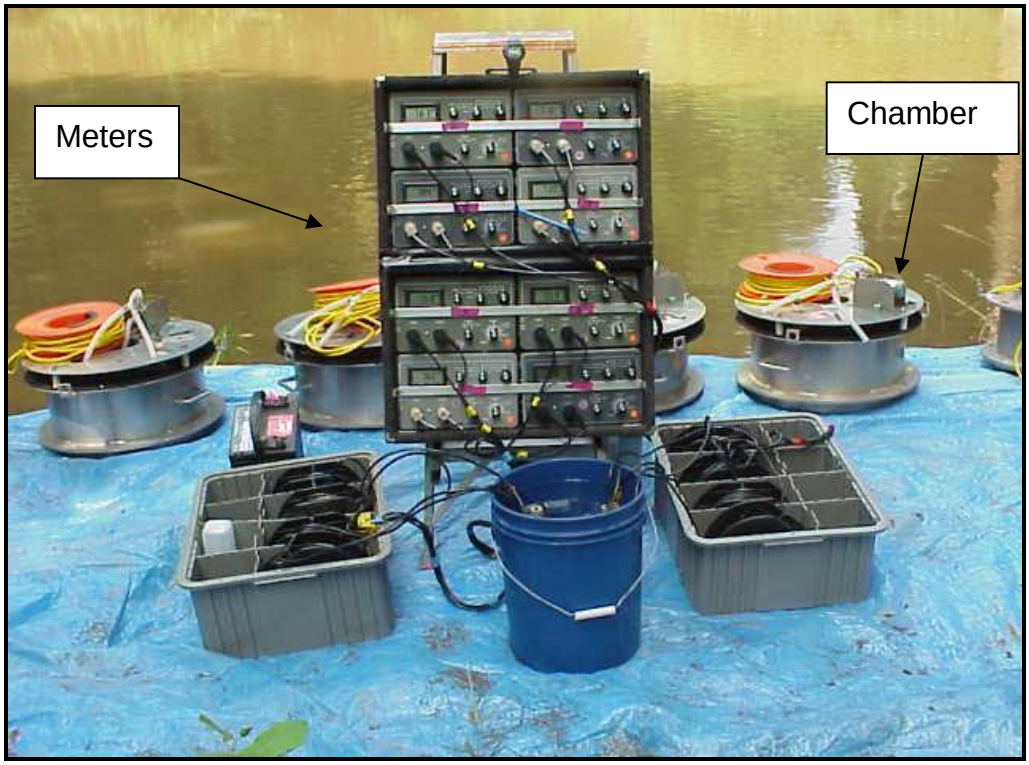


Figure 20. SOD Equipment

1.1. SOD Rate Formula:

The SOD rate for any study location is then calculated by using the SOD rate formula:

$$ß \times (K \times V) \div A = gr\ 0_2/m^2/hr.$$

where: ß = rate of change in D.O. as mg 0_2/L/min.

V = chamber volume in liters

A = chamber area in meters square

K = 0.06 (constant) converts liters to square meters

SOD rates are dependent on benthic metabolic processes, sediment particle size, stream velocity and other factors. SOD rates from 77 in-situ tests performed at locations with various substrate compositions are presented in Sediment Oxygen Demand, Processes, Modeling and Measurement (Murphy and Hicks, 1986, p. 318). An example SOD Excel Worksheet is provided at the end of this section for reference.

1.2. SOD Equipment List ï Due to the amount of gear and equipment necessary to successfully complete SOD tests, a checklist is recommended when preparing for testing. See Figure 21.

1.3. Site Evaluation ï Each site should be visited and checked out to determine if sediment is suitable in the area under investigation. Figure 22 is the SOD Site Evaluation Form that should be completed for each location.

2. FIELD CALIBRATION DISSOLVED OXYGEN METERS

An initial calibration is performed on the YSI 58 meters prior to the SOD test and a terminal calibration is performed on the meters after the test is completed. All calibration data is recorded on the SOD Calibration Forms (Figure 23). The need for accuracy is paramount for SOD evaluations due to the extremely small increments of change in D.O. measured during the test (+/- 0.01 mg/L). Because of the number of meters being calibrated on site and the extreme accuracy required for SOD testing, initial and terminal calibration procedures in this section vary from other D.O. meter calibration methods in this document. SOD meters are calibrated using the Modified Winkler Azide method as opposed to saturated air calibration methods.

Figure 21. SOD Equipment List:

CHAMBERS:
___ FIVE REP CHAMBERS ALUM
___ TWO BLANK CHAMBERS ALUM
___ ONE CLEAR BLANK CHAMBER
___ TUBES ON CHAMBERS
___ FLOW RESTRICTORS IN TUBES
___ SPACERS ON CHAMBER
___ BATTERY CLIPS ON DC LEADS
___ RUBBER SEALS OK
___ SILICON SEALS OK
___ TEST PUMPS
___ CHAMBER COLLARS
___ 3 BATTERIES MINIMUM (CHARGED)
___ CHAMBER HARNESS AND FLOATS
___ STOPPERS -#1 & #11½

METERS:
___ DO METERS
___ NEW MEMBRANES ON PROBES
___ CONDO METER
___ MEMBRANES & ELECTROLITE KIT
___ 100ôCABLES WITH PROBES
___ EXTRA 50ôCABLE AND PROBE
___ CALIBRATION & SOD FIELD SHEETS
___ COPPER BATTERY BARS
___ STAND FOR METERS (BOAT OR BANK)
___ ñCòCLAMPS (LARGE) - BOAT METER STAND
___ BUNGIES FOR METER STAND
___ BOARD FOR METER STAND MOUNT

WINKLER
___ WINKLER KIT (CHECKED OUT)
___ EXTRA CHEMICALS
___ BURET AND GLASSWARE
___ EXTRA BURET AND GLASSWARE
___ BUCKET FOR CALIBRATION
___ WATER CALIBRATION

____BURET STAND
____BURET WIRE
____STARCH

PERSONAL EQUIPMENT
____RAIN GEAR
____BOOTS
____WATCH
____COOLER AND ICE
____WASH WATER/SOAP
____INSECT REPELLANT
____SUN SCREEN
____HAT
____SUN GLASSES
____FOOD/DRINKS/WATER

MISC. EQUIPMENT
____CAMERA AND ACCESSORIES
____SEDIMENT JARS AND TAGS
____SOD TOOL BOX
____MAPS
____CALCULATOR/PENCILS
____TARPS
____FIRST AID KIT
____MACH, SHOVEL
____CHAIRS, BOX
____ROPES FOR BANK OR BOAT
____CELL PHONE
____COLORED TAPE
____BATTERY TESTER
____ñCòCLAMPS (SMALL) FOR REP LIDS
____FIELD LOG FOR SOD TEST
____PLASTIC CRATES

SOD SITE EVALUATION

Site Location - _____

_____

Date: _____ Time: _____

Site Description - _____

_____

_____

_____

Topo Map # _____ % From Right Bank (facing US) _____

Weather - _____

_____

Bank Description - _____

_____

Depth - _____ Velocity (fps) -_____

Sediment Description - _____

_____

Bottom Topography - _____

Water Description (turbid, clear, etc.) _____

_____

Site Schematic:

Figure 22. SOD SITE EVALUATION FORM

All meters are to be air calibrated prior to field operations (and on-site calibration) to assure all meters are functioning properly and stabilized.

The procedures are as follows:

2.1. Calibration Procedures

2.1.1 *INITIAL CALIBRATION Method*

a. Turn off all electronics (cellular phones, depth finders, etc.) prior to reading and calibrating meters.

b. Connect the D.O. probe to the probe receptacle of the YSI 58 meter and screw the retaining ring finger tight.

c. Connect the D.O. stirrer to the stirrer receptacle of the YSI 58 meter and screw the retaining ring finger tight. Check the stirrer battery condition by turning the stirrer switch to its spring-loaded battery check position. The warning LOBAT will indicate when approximately 5 hours of battery life remain.

d. Zero the instrument. Set the function switch to ZERO and adjust the display to read 0.00 with the O_2 ZERO control.

e. Switch to the 0.01 mg/l position and wait at least 60 minutes for the probe to polarize. Allowing additional time to re-polarize the probe is necessary whenever the meter has been turned off or the probe has been disconnected.

f. After the 60 minute wait, turn the function switch to ZERO and readjust the O_2 ZERO control to 0.00 if necessary. The meter is now ready to calibrate.

g. Calibration - Meters are calibrated using the Winkler azide method as described in this SOP in the Field Measurements Chapter III - section 3.2.

h. The D.O. probes are placed in a container of tap water with a relatively stable temperature. A minimum of four Winkler tests are then performed on the tap water. Three of the four resulting Winkler values must be within a 0.1 mg/l range. If the values are not in the 0.1 mg/l range, the Winkler tests should be repeated until the values are within the + or - 0.1 range. The three Winkler values are then averaged to provide an initial calibration value.

i. After the probes have stabilized in the container of tap water, the function switch is set to 0.01 mg/l, the meters are then adjusted to the initial calibration value by turning the O_2 CALIB control. The meters are now calibrated.

j. Leave the instrument on throughout the test to avoid re-polarizing the probe. Reactivate the stirrer approximately 2 minutes before each reading and turned off after the reading.

k. Obtain a bottom salinity reading using a YSI Model 33 S-C-T Meter. If salinity is present, the SALINITY knob on the YSI Model 58 D.O. Meter is adjusted accordingly.

l. Upon completion of the SOD test, perform a terminal calibration on all YSI 58 D.O. meters used. All terminal calibration data is recorded on the SOD terminal calibration form (Figure 23).

Note: If SOD tests are performed in coastal areas where tidal influence may cause salinity values to fluctuate during the test, salinity readings should be taken frequently and salinity adjustments made to the YSI 58 D.O. meters.

2.1.2 *TERMINAL CALIBRATION Method*

a. The D.O. probes are placed in a container of tap water with a relatively stable temperature and allowed to stabilize. A minimum of 4 Winkler tests are then performed on the tap water. The resulting Winkler values must be within a 0.1 mg/l range. If three of the four values are not in the 0.1 mg/l range, the Winkler tests should be repeated until the values are within the range. The Winkler values are then averaged to provide a terminal calibration value.

b. After the Winkler bottles have been filled with the tap water, turn on the stirrers, wait one minute and then record the D.O. and temperature readings.

c. Each D.O. reading should be within a 0.1 mg/l range from the average Winkler calibration value.

3. QUALITY ASSURANCE

3.1. Procedure

a. Complete the Pre-Sampling Calibration, Post-Sampling Calibration Check, and SOD Worksheets (Figure 23) on-site during each SOD test.

b. Perform Winkler tests per this SOP ï Chapter III- section 3.1 ï azide modification.

c. Perform a minimum of three Winkler titrations for Initial Calibration and Terminal Calibration.

d. Winkler values must be within a 0.1 mg/l range. If any value is outside the 0.1 mg/l range, then additional Winkler tests are performed until the values are within the range.

e. The terminal YSI 58 D.O. Meter reading should be within a 0.1 mg/l range from the average terminal Winkler calibration value.

f. A minimum ambient bottom D.O. of 2.0 mg/l is required to perform an SOD test (Murphy and Hicks, 1986).

g. Chamber velocities must be in a 0.08 to 0.12 ft/sec. range (Howard, 1988).
h. Take detailed field notes during the SOD test including a site description.
i. Conduct a pre-check to each SOD study to provide information on the study feasibility and station characteristics. During the -check sediment samples are generally collected to determine bottom characteristics.

Figure 23. Sediment Oxygen Demand Calibration Worksheet

SEDIMENT OXYGEN DEMAND CALIBRATION WORKSHEET

STUDY AREA	STATION
DATE	STAFF ON SITE

ALL METERS ZERO PRIOR TO CALIBRATION (YES NO)	CALIBRATION NOTES:
MEMBRANES VISUALLY CHECKED PRIOR TO CALIBRATION (YES NO)	
MEMBRANES LAST REPLACED	
BATTERIES LAST REPLACED	
CALIBRATOIN METHOD (SATURATED AIR WINKLER)	
CALIBRATION PERFORMED BY	
SALINITY	

INITIAL CALIBRATION

TIME OF INITIAL CALIBRATION
WINKLER READINGS: (A) (B) (C) (AVERAGE)

METER	AMB	BLANK O	BLANK OO	CLEAR	1	2	3	4	5	A	B	C
READINGS BEFORE CAL												
WINKLER												
DIFFERENCE												
ADJUSTED												

TERMINAL CALIBRATION

TIME OF INITIAL CALIBRATION
WINKLER READINGS: (A) (B) (C) (AVERAGE)

METER	AMB	BLANK O	BLANK OO	CLEAR	1	2	3	4	5	A	B	C
READINGS BEFORE CAL												
WINKLER												
DIFFERENCE												

4. CHAMBER DEPLOYMENT

After the D.O. meter calibration procedure is complete, the SOD chambers are prepared for the test. All chambers are prepared as follows prior to being placed into the water (Lawhorn, 1988).

4.1. Setting up Chambers

4.1.1. *Chamber Preparation*

a. Place the lids on replicate chambers in the up position with spacers located between the lid and lid companion ring. Wing nuts should be tight enough to hold the spacers in place but not so tight as to hamper removal after the chamber has been set in place on the bottom.

b. Insert the water sampling port stoppers on each chamber lid (size #1, two on each lid).

c. Open the monitoring probe port on all chamber lids (no stoppers).

d. Inspect the replicate chamber lid gaskets for damage or debris that could prevent a watertight seal.

e. Inspect the seals on the blank chambers for damage.

f. Clip the harness ropes to each chamber.

g. Open the water intake ports located on the bottom of the blank chambers (no stoppers).

h. Disconnect the return pump tubing from the chamber lid male connectors.

4.2. *Boat operation only:*

a. Hang the chambers in sequential order along the gunwale with the chamber lids several inches below the surface of the water.

b. Tie the chamber harness to a gunwale cleat.

c. Situate the boat over the bottom where the chambers will be placed.

d. Do not allow the chambers to disturb the bottom sediment.

4.3. *Land operation*:

Chambers are placed on the stream bank in the order that they will be deployed. This will prevent harness ropes, pump cables and probe cables from becoming tangled during the chamber deployment and the SOD test.

4.4. *Chamber deployment:*

a. Blank chambers are deployed first because sufficient time is required to replace surface water trapped inside the chamber with ambient bottom water prior to initiating the SOD test.

b. When deploying blank chambers in soft sediment, place the chambers in an area away from the area that the replicate chambers will be deployed in order to avoid stirred up sediments from being drawn into the chamber through the open probe port.

c. One clear polycarbonate and acrylic blank chamber is used in addition to the conventional aluminum blank chambers to provide an indication of whether or not photosynthesis is occurring in the water column. The mechanical functions and the deployment procedure for the clear blank chamber are identical to that of the aluminum blank chambers. In cases of high flow a weighted band should be placed around the clear chamber to prevent it from being washed away.

d. Each blank chamber must be filled with surface water that enters through the two filling ports located on the bottom plate of the chamber.

e. After the blank chamber is filled at the surface and prior to lowering the chamber, two #11½ stoppers must be inserted into the filling ports. Surface water is used to fill the blank chamber to create negative buoyancy so the chamber can be lowered to the bottom.

f. After the filling port stoppers are in place, the chamber is agitated to dislodge any air that is trapped under the lid. The trapped air will exit through the probe port.

g. The chamber is then lowered to the bottom.

h. When the blank chamber is on the bottom, the pump is turned on. Unlike the replicate chambers, the lid and bottom of the blank chambers are permanently sealed thus no water exchange occurs when the chamber is lowered to the bottom. Surface water must be purged from the chambers by operating the pump with the tubing disconnected from the male adapters on the lid while the chamber is on the bottom. Bottom water is drawn into the chamber through the open probe port while the surface water is purged through the disconnected return tubing. With the two return pump tubes disconnected, the chamber will purge surface water and draw in bottom water.

i. A light tapping on the pump housing and tubing will dislodge air bubbles trapped in the pump system.

j. The pump is then allowed to run while the other chambers are being deployed.

k. This procedure is repeated for each blank chamber.

4.5. *Replicate Chamber Deployment*

a. After the blank chambers are deployed, each replicate chamber is slowly lowered to the bottom substrate prior to setting the chamber.

b. Set the replicate chambers out in downstream to upstream order to prevent sediment disturbance and any silt that may have been disturbed from settling on areas where other chambers will be placed.

c. If the chamber location is unsatisfactory because of debris, or other bottom characteristics that would prevent the chamber from sealing then the chamber is carefully relocated. In addition, if the chamber location is atypical of the general stream area, the chamber or possibly the station should be relocated.

d. After the replicate chamber is placed in a satisfactory location on the bottom, carefully examine the sediment/flange seal and the sediment/inner core seal to assure that ambient water infiltration will not occur during the test.

e. The replicate chamber lid is then lowered by loosening the four wing nuts and removing the PVC spacers. The lid must be lowered very slowly as not to create a pressure wave and stir up silt inside the chamber. If silting occurs in the chamber, initial D.O. readings will be erratic and a longer period will be required for SOD rate stabilization (see: Section 5. Procedure for Recording SOD Data).

f. Replace the spacers between the companion ring and stainless steel washers. The wing nuts are then tightened and the gasket forms a watertight seal.

g. Activate the pump and lightly tap the pump housing and tubing to dislodge air trapped in the pump system.

h. Turn off the pump and allow any silt that may have been suspended to resettle before starting the test.

i. Reconnect the return tubing.

j. Repeat this process until all replicate chambers are deployed.

4.6. Once all replicate chambers are in place, insert DO probes into the probe ports beginning with the first blank chamber deployed and ending with the final replicate chamber.

4.7. During the D.O. probe installation, replicate chamber pumps can be turned on and a final check of the chamber and pump tubing can be performed.

4.8. In addition to the D.O. probes located inside the SOD chambers, one D.O. probe is placed on the outside of a chamber to record ambient D.O. values.

4.9. When an SOD test has been completed, chambers can usually be lifted from the bottom using the harness ropes.

5. RECORDING SOD FIELD DATA.

5.1. After SOD Chambers and Probes are installed

5.1.1 Readings

a. Stirrers are activated approximately 2 minutes prior to reading meters and turned off after the data is recorded.

b. All meters (including the ambient meter) are read at 15 minute intervals. For each chamber, D.O., temperature, and the change in D.O. per 15 minute time period is recorded on the SOD field sheet form (Figure 24).

c. D.O. readings from the replicate chambers will usually decrease at a relatively similar rate. Typically, if relatively uniform decreases in D.O. are observed in the replicate chambers after stabilization, a sufficient SOD rate can be calculated from approximately 2 hours of testing (Murphy and Hicks, 1986).

d. A minimum oxygen reduction of 0.4 mg/l is required before an SOD test should be terminated. This situation is not typically encountered and would provide an extremely low SOD rate indicating little organic content in the sediment.

e. SOD tests with very slow oxygen uptake rates may be less reliable due to an extremely small amount of oxygen depletion over a greater period of time. Since longer tests are necessary when slow oxygen uptake is occurring, the potential for meter calibration drift increases.

f. See Figure 25 for an example of completed SOD worksheet.

5.2 Recording Errors

5.2.1 *Erratic D.O. Readings Troubleshooting*

If observed in replicate chambers the following are possible problems:

a. Initial D.O. readings may be erratic if sediment was disturbed during chamber placement on the bottom. This problem occurs often at stations where sediment consists of soft mud or a silt-like composition and is usually observed in all of the replicate chambers. For this reason, several of the initial D.O. readings may be omitted from the SOD rate calculations. The readings will usually stabilize as the suspended particles in the chamber settle out, (generally about 15 to 30 minutes, 1 to 2 readings).

b. If D.O. readings from all chambers do not stabilize after 30 minutes, it may indicate that the chambers are sinking into the soft sediment causing the circulation diffusers to become close to the sediment and continually disturbing the silt. If this occurs, the chambers must be reset on the bottom and a chamber collar must be placed around the bottom chamber

flange to prevent the chambers from sinking. Chamber collars are flat, thin pieces of material, that increases the surface area of the chamber flange and prevent the chamber from sinking into soft sediment.

c. If D.O. readings from a replicate chamber do not stabilize and begin to decrease after the other chambers have stabilized, it may indicate that the chamber was not initially sealed and ambient bottom water is leaking into the chamber via the ports, gasket seal, pump tubes or the sediment flange seal. The chamber must be reset and the seal integrity reconfirmed.

d. On occasion, ambient water will begin leaking into a chamber. Chamber leaks (blowouts) are the most frequent problem encountered in SOD tests. This problem is easily recognized when D.O. values in a chamber that have been steadily decreasing suddenly begin to rise rapidly. However, if the chamber leak is small, the rate of decrease in D.O. may only be slowed, resulting in an unrealistically low rate for the chamber. For these reasons, the rate of D.O. change in each chamber must be carefully evaluated and recorded for each 15 minute time period during the SOD test.

   If a chamber leak is detected the following options may be considered:

   - Stop the leak and restart the test for that chamber; or
   - Delete the data from that chamber from the SOD test; or
   - Terminate entire test, if sufficient data has been recorded to establish a reliable linear regression.

e. If the D.O. in a chamber falls much more rapidly than in the other chambers, it may indicate that the chamber has been inadvertently placed on organic debris such as decaying leaves or other organically rich deposits that may be uncharacteristic of the area. The chambers must be placed on sediment that is typical for the station area. If this problem is encountered, the chamber should be relocated or the data deleted from the SOD test.

   The validity of SOD test data is dependent on locating the test site at an area that is typical of the water body being studied. If the chamber location is atypical of the general stream area then the chamber or possibly the station should be relocated.

f. When other obvious D.O. or temperature problems occur during the SOD test, it is usually the result of meter or probe malfunction and can be detected by the terminal calibration results.

6. METER AND PROBE PREPARATION

6.1. Procedure

a. Check all D.O. meters, cables and probes to assure proper functioning **before** the survey.

b. Evaluate the YSI 58 instrument batteries and replaced if necessary. Stirrer batteries should be checked to assure that batteries are adequate to complete SOD test.

c. Replace all D.O. probe membranes prior to each SOD survey. After the membrane has been changed, a minimum of 24 hours should be allowed for the probe to equilibrate before it is used for an SOD test. YSI Standard Membranes should be used.

7. SOD CHAMBER VELOCITY TEST

SOD rates are directly related to the sediment/water interface velocity, therefore specific and consistent velocities must be maintained in all chambers for accurate SOD testing. EPA recommends a constant chamber velocity of 0.1 ft/sec and an acceptable range of 0.08 to 0.12 ft/sec (Howard 1988). To maintain this velocity range, DWR uses a flow restrictor placed in the chamber pump tubing to reduce pumping velocity. The restrictor is 1" long, made from brass stock, and has a 7/64" opening in the center to allow a desired velocity of water.

All SOD chambers are periodically tested in the lab to ensure that velocities remain constant after repeated field use and pump wear. Velocity tests are performed using a Marsh McBirney Magnetic Flow Meter Model 201. The Marsh McBirney meter is factory calibrated. Chamber velocity tests procedures are as follows:

7.1. Velocity test procedures for replicate chambers:

a. Insert a # 11½ stopper in the monitoring probe port and two # 1 stoppers in water sampling ports. All pump tubing should be connected and the chamber lid must be tight against the chamber companion ring.

b. Place the chamber upside-down on a support in a manner that will allow access to the monitoring probe port. The support should not touch the pump tubing or alter the pump flow in any manner. (The chamber and support should be located over a sink or other acceptable area where the test water can be easily drained).

c. Fill the chamber with water to the cutting ring flange (normal water/sediment interface).

d. Turn the pump on. It may be necessary to add more water to fill pump and pump tubing after the pump is turned on and to tap the pump and tubing to dislodge trapped air. Place the Marsh McBirney probe 2 inches below the surface of the water halfway between the outer and inner chamber wall. Allow the

water circulation in the chamber to reach the maximum velocity (approximately 15 minutes).

e. Read the Marsh McBirney Meter. The velocity in the chamber should be within a range of 0.08 to 0.12 ft/sec.

f. If the velocity is not constant or out of the acceptable range, check the following:

- Probe orientation or placement in the chamber.
- Restrictions in pump tubing (7/64" brass restrictor may be blocked).
- Air bubbles could be locking the pump or altering flow.
- Pump may be damaged and not pumping maximum flow.
- Check pump battery voltage output (12 volt)
- Check velocity meter calibration.

7.2. Velocity Tests for Blank Chambers:

a. Insert two # 11½ stoppers into the filling ports on the bottom of the blank chamber. All pump tubing should be connected.

b. Place chamber right side up on a support in a manner that will allow access to the filling ports.

c. Fill the chamber completely with water.

d. Turn the pump on . It will be necessary to add more water to fill pump and pump tubing after the pump is turned on and to tap the pump and tubing to dislodge trapped air. Place the Marsh McBirney probe through the D.O. probe monitoring port at a depth of 2 inches. Allow the water circulation in the chamber to reach the maximum velocity (approximately 15 minutes).

e. Read the Marsh McBirney Meter.

f. Use the same trouble shooting procedures as with the replicate chambers if problems are encountered.

8. LEAK TEST FOR SOD CHAMBERS

SOD chambers must remain watertight during the SOD test to prevent ambient bottom water from entering the chamber and invalidating the test. The exchange of ambient bottom water and chamber water can occur by two means, by leaking between the sediment and chamber cutting edge or by leaking through any of the normally sealed chamber gaskets, stoppers, fittings and tube connections. Chamber leaks at the sediment/chamber interface generally occur as a result of sediment or sand washing out from around the chamber due to scouring and are usually detected during the test. Leaks through chamber seals, other than the sediment/chamber interface can be detected during the Chamber Velocity Test (Section 7). Note: leak test is under worst case conditions because chamber water (inside/outside) is equalized during the test. Procedures for leak testing SOD chambers are as follows:

1. Insert a # 11½ stopper in the monitoring probe port and # 1 stoppers in water sampling ports. All pump tubing should be connected and the chamber lid must be tight against the chamber companion ring.
2. Place the chamber upside-down on a support in a manner that will allow access to the monitoring probe port. The support should not touch the pump tubing or alter the pump flow in any manner. The chamber and support should be located over a sink or other acceptable area where the test water can be easily drained.
3. Fill the chamber with water to the cutting ring flange (normal water/sediment interface for replicate chambers and to bottom plate on blank chambers).
4. Turn the pump on.
5. If water leaks out, repair or replace the seal and repeat the leak test.

9. THREE POINT ANCHOR TECHNIQUE

If a boat operation is necessary to perform a SOD test, care must be taken to provide maximum stability and minimize wave action and horizontal swing over the bottom. Movement of the boat by wave action or swing on a single anchor line will result in chambers being lifted and the SOD test terminated. This problem can be avoided by using the following three-point anchor technique:

1. After the boat is on station, align the bow into the current.
2. Set bow anchor on SOD boat allowing a minimum scope of 3 times depth. More scope may be necessary if strong current or winds are present.
3. Use support boat to set aft port and aft starboard anchors (minimum scope 3 times depth).
4. Anchors should be oriented in a 3-point (tripod like) pattern with the SOD boat in the center.
5. After all anchors are set, the lines should be tightened as much as possible and cleated to provide maximum stability and minimize horizontal movement of the SOD boat.
6. While anchoring, care should be taken not to disturb the sediment where the SOD test is to be performed.
7. It is potentially dangerous to anchor with the stern of the boat facing upstream if current, waves or bad weather exists. The 3-point anchor method should not be used in areas affected by strong tidal current unless the test can be completed prior to the turning of the tide.

Figure 24. SOD Field Sheet

STUDY AREA																					
STATION LOCATION																					
DATE		SEDIMENT TYPE				DEPTH				CHAMBERS DIVER DEPLOYED? YES/NO				VELOCITY FT/SEC (at bottom)							
PERSON(S) READING METERS						STAFF ON SITE								BOAT SOD/BANK SOD							
TIME	MIN.	AMB		BLANK O		BLANK OO		BLANK CLEAR		REP 1		REP 2		REP 3		REP 4		REP 5		BACK-UP	
		DO	TEMP	DO	TEMP	DO	TEMP	DO	TEMP	DO	TEMP	DO	TEMP	DO	TEMP	DO	TEMP	DO	TEMP	DO	TEMP
	START																				
	15																				
	30																				
	45																				
	60																				
	75																				
	90																				
	105																				
	120																				
	135																				
	150																				
	165																				
	180																				
	195																				
	210																				
	225																				
	240																				
	255																				
	270																				

TIME	MIN.	Change in DO	Change in DO	Change in DO	Change in DO	Change in DO	Change in DO	Change in DO	Change in DO	Change in DO	Change in DO
	START										
	15										
	30										
	45										
	60										
	75										
	90										
	105										
	120										
	135										
	150										
	165										
	180										
	195										
	210										
	225										
	240										
	255										

Figure 25. Example of SOD Excel Worksheet for Determining Average SOD Rates.

Rocky River near Aquadale
STATION LOCATION: Rocky River at Hwy 52
SUB-BASIN 03 07 13 COUNTY - Stanley

STAFF ON SITE: QUIDLEY, WILLIAMS, FISHER, MEDLIN
DEPTH AT SITE: 2 feet
BOTTOM SEDIMENT: SAND

TEST CONDUCTED FROM RIGHT BANK
AVERAGE AMBIENT WATER TEMP 24.4°C
SOD TEST FOR SOD CALIBRATION

		BLANK CHAMBERS		REPLICATE CHAMBERS					
	AMB	BLANK 0	BLANK OO	REP 1	REP 2	REP 3	REP 4	REP 5	Extra Chamber
NOTES									not used
AVERAGE T°C	24.3	24.3	24.4	24.4	24.3	24.4	24.3	24.4	
D.O. Range	7.81 - 8.02	7.79 - 7.86	7.87 - 7.97	6.34 - 7.80	6.61 - 7.63	6.78 - 7.80	6.69 - 7.60	6.61 - 7.47	
Test Time in min.									
0	7.99	7.86	7.97	7.80	7.63	7.80	7.60	7.47	
15	8.02	7.85	7.96	7.63	7.49	7.68	7.48	7.35	
30	8.00	7.84	7.95	7.47	7.37	7.56	7.37	7.23	
45	7.94	7.83	7.94	7.30	7.24	7.45	7.26	7.10	
60	7.83	7.82	7.93	7.14	7.12	7.34	7.17	7.02	
75	7.81	7.82	7.93	6.98	7.02	7.23	7.07	6.95	
90	7.90	7.81	7.91	6.83	6.91	7.12	6.98	6.87	
105	7.91	7.80	7.89	6.67	6.81	7.01	6.88	6.77	
120	7.93	7.80	7.88	6.50	6.71	6.89	6.78	6.68	
135	7.90	7.79	7.87	6.34	6.61	6.78	6.69	6.61	
RATE OF WATER COL (ß) mg/L/min		-0.0005	-0.0007						
RATE OF REPLICATE (ß) mg/L/min				-0.0108	-0.0073	-0.0075	-0.0067	-0.0063	
		-0.0006	AVG. RATE OF WATER COL (ß) mg/L/min	-0.0006	-0.0006	-0.0006	-0.0006	-0.0006	
RATE CALCULATION			REPLICATE RATE (-) AVG WATER COL RATE mg/L/min	-0.0102	-0.0067	-0.0069	-0.0061	-0.0057	
ß x (K x V) ÷ A = gr O2/m2/hr			SOD RATE gr/m2/hr	-0.1113	-0.0734	-0.0756	-0.0664	-0.0620	
ß = Rate of change in D.O.			gr/m2/day	-2.6713	-1.7624	-1.8147	-1.5937	-1.4884	
as mg O2/L/min									
v = Chamber volume in liters									
A = Chamber area in meters square									
K = .06 (Constant)									

AVERAGE SEDIMENT OXYGEN DEMAND (rate calculated for 3 hours)

-0.0778 gr/m2/hr

-1.8661 gr/m2/day AT AMB. TEMP 24.4°C

-1.4145 CORRECTED TO 20°C (COEF 1.065)

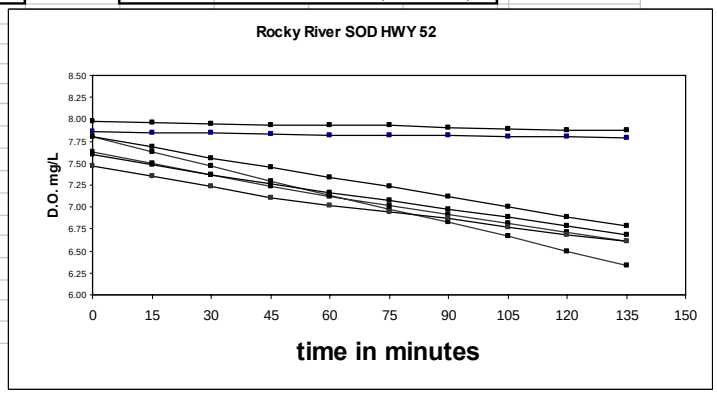
-0.0072 gr/ft2/hr

-0.1734 gr/ft2/day

FIELD STAFF :

CALCULATIONS PERFORMED BY:
Harold Quidley

CALCULATIONS CHECKED BY:
Ed Williams



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APPENDICES

Appendix 1: DWR's Hydrolab Mulitparameter Guidance Sheet

DISSOLVED OXYGEN

HYDROLAB with CLARK CELL D.O. SENSOR
(DOES NOT APPLY TO LDO SENSORS)

CALIBRATION

Clark Cell Dissolved Oxygen (D.O.) Calibration for Hydrolab Meters:

(% AIR CALIBRATION IN WATER-SATURATED AIR)

1) Secure probe to work surface and inspect membrane for tears and debris.
2) Rinse calibration cup with deionized water and attach to probe.
3) Fill calibration cup with tap water until water is just level with the O-ring used to secure the D.O. membrane. Do not cover the membrane.
4) Remove any water droplets from D.O. membrane with the corner of a chem-wipe or a lint-free cloth.
5) Place inverted lid (concave upward) on top of calibration cup. The lid should not completely seal or cover the cup (lid should be slightly tilted inwards on top of the cup, leaving a small gap or opening).
6) Wait 5 to 10 minutes for readings to stabilize.
7) Once readings are stable, record the following values on the calibration sheet: "Temperature", "Initial % Saturation", and "Initial Meter Reading (mg/L)".
8) Record "Barometric Pressure" and "Altitude" on the calibration sheet.
   These values are available on the *Dissolved Oxygen Table* for your corresponding regional office. All calibrations should be performed in a controlled environment (field calibrations are not recommended).
9) Use the *Dissolved Oxygen Table* for your location to find the "D.O. Table Value" (based on the temperature displayed on the meter), and record value on calibration sheet.
10) Follow menu prompts to calibrate for Dissolved Oxygen, Percent Saturation, which is displayed as "DO%:SAT".
    * **NOTE: Dissolved oxygen should always be calibrated using % saturation.**
      Calibrations based on "mg/l" require a water sample with a known D.O. concentration (Winkler titration must be performed).
11) When prompted, enter the barometric pressure for your location in millimeters of Mercury (mmHg). The unit should display "CALIBRATION SUCCESSFUL!"
12) On the calibration sheet, record the displayed mg/L value as "Calibrated Meter Reading" and the % SAT value as the "Calibrated % Saturation" value.
    * **NOTE: "D.O. Table Value" and "Calibrated Meter Reading" value should be within ±0.5 mg/L of each other.**

Terminal Calibration Check (Post-Sampling Meter Check)

a. Repeat calibration steps 1 thru 9.
b. The post-sampling "D.O. Table Value" and the post-sampling "Initial Meter Reading" should be within ±0.5 mg/L of each other.

MAINTENANCE

Sonde Storage - Calibration/Storage Cup:
Store sonde and sensors in the Calibration/Storage Cup filled with pH 4 buffer solution.

Clark Cell D.O. Membrane Replacement:

D.O. membrane should be replaced when calibration is impossible, if calibration drift occurs quickly or frequently, or if membrane is dry or damaged.

1) Remove O-ring and shake out old electrolyte.
2) Rinse sensor cavity with deionized water.
3) If gold cathode appears tarnished, dry and lightly buff with a pencil eraser or Kimwipe until gold is bright.
4) Refill with fresh D.O. electrolyte (2M KCl) until a meniscus forms. Remove any bubbles trapped in electrolyte.
5) Replace membrane and secure with O-ring. Inspect O-ring for any tears or breaks; replace as needed.
6) Trim excess membrane.
7) Allow membrane to soak overnight in tap water before calibrating.

D.O. Circulator Maintenance (Remove dirt and debris build-up from inside circulator impeller shaft):

1) Use a flat-head screwdriver to remove impeller screw.
2) Clean dirt and debris from the screw, impeller, and inside the impeller shaft.
3) Replace screw and re-attach impeller to circulator with flat-head screwdriver.

Frequency of cleaning will depend on use and environment.

DISSOLVED OXYGEN

HYDROLAB with LUMINESCENCE D.O. (LDO) SENSOR
(DOES NOT APPLY TO CLARK CELL SENSORS)

CALIBRATION

Hach Luminescence Dissolved Oxygen (LDO) Calibration for Hydrolab Meters:

(% AIR CALIBRATION IN AIR-SATURATED WATER)

1) Fill a 1-liter bottle half-full of tap water. Water bottle should remain open (no cap or seal) for at least 12 hours (overnight) to allow water to equilibrate to ambient temperature and atmospheric pressure.
2) After 12 hours have passed, use a thermometer to confirm that the water in the bottle is close to room temperature.
3) Seal/cap bottle and shake it very vigorously for 40 seconds to saturate the water with air.
4) With sonde positioned with sensors facing upward, pour the water into the calibration cup such that the LDO sensor cap and the temperature sensor are completely submersed (water should come close to the top of the calibration cup).
5) Completely cover the top of calibration cup with the inverted lid (do not tightly seal the cup).
6) Wait 10 minutes for readings to stabilize. If temperature changes more than ±0.5 ºC during calibration, recalibration of the sensor is recommended.
7) Once readings are stable, record the following values on the calibration sheet: "Temperature", "Initial % Saturation", and "Initial Meter Reading (mg/L)".
8) Record "Barometric Pressure" and "Altitude" on the calibration sheet.
   These values are available on the *Dissolved Oxygen Table* for your corresponding regional office. All calibrations should be performed in a controlled environment (field calibrations are not recommended).
9) Use the *Dissolved Oxygen Table* for your location to find the "D.O. Table Value" (based on the temperature displayed on the meter), and record value on calibration sheet.
10) Follow menu prompts to calibrate for Dissolved Oxygen, Percent Saturation, which is displayed as "LDO%:SAT".
    * **NOTE: Dissolved oxygen should always be calibrated using % saturation.**
      Calibrations based on "mg/l" require a water sample with a known D.O. concentration (Winkler titration must be performed).
11) When prompted, enter the barometric pressure for your location in millimeters of Mercury (mmHg). The unit should display "CALIBRATION SUCCESSFUL!"
12) On the calibration sheet, record the displayed mg/L value as "Calibrated Meter Reading" and the % SAT value as the "Calibrated % Saturation" value.
    * **NOTE: "D.O. Table" value and "Calibrated Meter Reading" value should be within ±0.5 mg/L of each other.**

Terminal Calibration Check (Post-Sampling Meter Check)

a. Repeat calibration steps 1 thru 10.
b. The post-sampling "D.O. Table Value" and the post-sampling "Initial Meter Reading" should be within ±0.5 mg/L of each other.

MAINTENANCE

Sonde Storage - Calibration/Storage Cup:
Store sonde and sensors in the Calibration/Storage Cup filled with pH 4 buffer solution.

Hach LDO Sensor Cap Replacement:

Replace once a year or when cap is damaged.

1) Unscrew old sensor cap from end of probe.
2) Carefully dry clear plastic window at the end of probe with cotton swab.
3) Place cap seal and o-ring on the probe tip.
4) Screw new sensor cap onto probe so that the o-ring seal is compressed. Do not over-tighten cap.
   No water or moisture should be present between sensor cap and clear plastic window at top of probe.
5) Do NOT use alcohol or any organic solvent solutions to clean the Hach LDO sensor. These solvents will damage the plastic sensor cap.

SPECIFIC CONDUCTANCE

HYDROLAB

CALIBRATION

THREE-STEP SPECIFIC CONDUCTIVITY PROCEDURE:

I. "Dry Air" (ALWAYS ZERO):

The "Dry Air" step is a **check** for the Quanta meters only, and a **calibration** for the Hydrolab 4a and MS5 meters.

1) Attach calibration cup to probe. Fill calibration cup half-full with deionized water and seal with lid. Shake probe to rinse. Repeat.
2) Secure probe to work surface, and remove calibration cup.
3) Dry the inside of conductivity sensor slot thoroughly.
4) Record displayed value as "Initial Meter Reading" in the "Dry Air" section of the calibration sheet.
   If the reading is not within ± 2, follow cleaning procedure, and repeat calibration procedure.

If using a Quanta, proceed to step 8.

Steps 5-7 are for Hydrolab 4a and MS5 meters only:

5) Follow menu prompts to calibrate for specific conductance.
6) When prompted, enter "0" (zero) as specific conductance standard. Display should read "CALIBRATION SUCCESSFUL!"
7) Record displayed value as "Calibrated Meter Reading" in the "Dry Air" section of the calibration sheet.

II. Conductivity Standard:

Calibrations should be performed using **fresh, certified** conductivity standards that bracket the range of measurements to be taken that day. Record the standard's "true value" (found on the certificate of analysis or bottle label) and lot number (also called analysis number) on the calibration sheet. Traceable® Certificate of Analysis for Conductivity Solution sheets (one certificate for each lot number) should be retained and stored in a notebook.

8) Re-attach calibration cup to probe. Fill cup half-full with deionized water. Seal cup with lid and shake probe to rinse. Repeat.
9) Rinse sensors with small amount of fresh conductivity standard. Discard rinse.
10) Fill calibration cup with conductivity standard to within a centimeter of the top of the Calibration Cup. (Pour standard down the interior side of the cup to avoid trapping bubbles.)
11) Wait approximately 1 to 3 minutes for readings to stabilize.
12) Record displayed value as "Initial Meter Reading" in the "Conductivity Standard" section of the calibration sheet.
13) Follow menu prompts to calibrate for Specific Conductance. When prompted, enter the value of the standard. Unit will display "CALIBRATION SUCCESSFUL!"
14) Record displayed value as "Calibrated Meter Reading" in the "Conductivity Standard" section on the calibration sheet.

III. Calibration Check:

15) Rinse with deionized water and wipe dry with a chem-wipe or a lint-free cloth. Confirm that the meter display is reading 0 (zero) µS before going to the next step.
16) Repeat steps 9-11 with a fresh conductivity standard with a value different from the one used in the previous steps. Choose a standard that will give the best range of values for the anticipated conductivity of the samples to be collected.
17) Record displayed value as "Initial Meter Reading" in the "Calibration Check" section of the calibration sheet.

Terminal Calibration Check (Post-Sampling Meter Check)

a. Repeat calibration steps 1 thru 4, and record value in the "Dry Air" section on the calibration sheet.
   For the "Dry Air" check, displayed value should be between –2 and 2 µS.
b. Repeat calibration steps 8 thru 12, and record value in the "Conductivity Standard" section on the calibration sheet.
   "Conductivity Standard" value should be within ±10% of the standard.
c. Repeat step 15 thru 17, and record value in the "Calibration Check" section on the calibration sheet.
   "Calibration Check", value should be within ±10% of the standard.

MAINTENANCE

Cleaning Conductivity Sensor:

Conductivity cell should be cleaned frequently (in addition to rinsing with DI water after field use).
A clean cell is imperative for accurate readings.

1) Use a cotton swab and mild soap to remove any films or deposits on the sensor.
2) Rinse sensor with deionized water.

pH | **HYDROLAB**

CALIBRATION

TWO-POINT PH CALIBRATION REQUIRED:

1st Calibration Point (always start with 7 buffer):

1) Attach calibration cup to probe. Fill cup half-full with deionized water. Seal cup with lid and shake probe to rinse. Repeat.
2) Rinse sensors with small amount of 7 pH buffer. Discard buffer rinse.
3) Secure probe to work surface.
4) Fill calibration cup with **fresh** 7.0 buffer to within a centimeter of the top of the calibration cup. Wait at least 2 minutes for stabilization.
5) Record displayed value as the "Initial Meter Reading" under Buffer # 1 on the calibration sheet.
6) Follow menu prompts to calibrate for pH. When prompted, enter "7.0" as the value of your standard. Unit will display "CALIBRATION SUCCESSFUL!"
7) Record the displayed value as "Calibrated Meter Reading" for Buffer #1 on the calibration sheet.

2nd Calibration Point:

8) Rinse sensors with deionized water.
9) Rinse sensors with small amount of a pH buffer that is similar to the anticipated pH of the samples to be collected. Discard buffer rinse.
10) Fill calibration cup with **fresh** buffer to within a centimeter of the top of the calibration cup. Wait 1 to 3 minutes for solution to stabilize.
11) Record displayed value as the "Initial Meter Reading" for Buffer # 2 on the calibration sheet.
12) Follow menu prompts to calibrate for pH. When prompted, enter the value of Buffer #2 (also called the slope buffer value).
    Unit will display "CALIBRATION SUCCESSFUL!"
13) Record the displayed value as "Calibrated Meter Reading" for Buffer #2 on the calibration sheet.

Confirmation Buffer:

14) Rinse sensors and calibration cup with deionized water.
15) Rinse sensors with small amount of 7 buffer. Discard buffer rinse.
16) Fill calibration cup with 7 buffer to within a centimeter of the top of the calibration cup. Wait 1 to 3 minutes for solution to stabilize.
17) Record the displayed value as the "Meter Reading" under "Confirmation Buffer 7.0" on the calibration sheet
18) Confirm that the "Meter Reading" value is within ± 0.1 of the buffer value (between 6.9 and 7.1).

Terminal Check (Post-Sampling Meter Check)

a. Repeat steps 1 thru 5 (for 7 buffer); record displayed value on the calibration sheet.
   This value should be within ±0.2 of 7 (for 7 buffer).
b. Repeat steps 8 thru 11 for Buffer #2. Record value on calibration sheet.
   Value should be within ±0.2 of Buffer #2.

The "Confirmation Buffer" step is not required for post-sampling meter checks.

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pH | **HYDROLAB**

MAINTENANCE

Indicators that maintenance is needed include:

- Unable to calibrate
- Slow response
- Erratic readings
- Clogged reference junction
- Black reference junction
- Coated glass bulb

Maintain as directed below. The pH electrolyte should be changed at least 3 to 4 times a year, or as needed.

pH Reference Electrode Maintenance: (pg 45 - Hydrolab MS 5 User Manual, Feb 2006 ed. 3)

Check the reference electrode regularly to confirm flow through the Teflon junction. To test for flow through the junction, press lightly on the top of the reference electrode. A bead of electrolyte should wet the Teflon junction. Maintain as directed below.

1) Remove the pH reference sleeve, and discard old electrolyte.
2) Drop two KCL salt pellets into reference sleeve. Refill the sleeve (to the top) with electrolyte, which is provided in the maintenance kit (3M KCl, saturated with AgCl).
3) With the sensors pointed down, gently push the reference sleeve back onto its mount, until the sleeve just covers the O-ring located on the mount.
4) Turn probe so that the sensors point up and push the sleeve the rest of the way onto its mount.
   Air and electrolyte should flow through the Teflon junction.
   If it does not, repeat steps 1-4.
   If the second attempt fails, replace the old junction.
5) Rinse with tap water.

Cleaning pH Glass Electrode:

Check the glass bulb regularly for a dirty film or scratches. Clean as directed below.

1) Wet a cotton swab with a mild soap solution.
2) Gently swab the pH glass electrode.
3) Rinse electrode with tap water.

Appendix 2: DWR's YSI 85 and Accumet Guidance Sheet

DISSOLVED OXYGEN — **YSI 85**

CALIBRATION

D.O. Calibration for YSI-85 Meters:
(% AIR CALIBRATION IN WATER-SATURATED AIR)

1) Inspect membrane. Membrane should be taut, flat, and free of tears and debris.
2) Confirm that sponge in the calibration/storage chamber is moist (not soaking wet). Dry the probe and sides of calibration chamber with a lens cloth.
3) Insert probe into the calibration/storage chamber. Make sure there are no water droplets on the membrane.
4) Turn meter "ON" and press "MODE" until dissolved oxygen is displayed in "%".
5) Wait approximately **15 to 30** minutes for readings to stabilize.
6) Once readings are stable, record the following displayed values on the calibration sheet: "Temperature", "Initial % Saturation", and "Initial Meter Reading (mg/L)" (press mode to switch from % Saturation to mg/L). Then press mode until % Saturation is again displayed on the screen.
7) Record "Altitude" and "Barometric Pressure" on the calibration sheet.
   These values are available on the *Dissolved Oxygen Table* for your corresponding regional office. All calibrations should be performed in a controlled environment (field calibrations are not recommended).
8) Use the *Dissolved Oxygen Table* for your location to find the "D.O. Table Value" (based on the temperature displayed on the meter); record value on calibration sheet.
9) Press and release the "Up" and "Down" arrow buttons at the same time.
10) Use the arrow buttons to find the local altitude (to the nearest 100 ft) and press "ENTER".
11) "CAL" will be visible in the lower left of the display. The current % reading should be visible on the main display. Press "ENTER".
12) "SAVE" will be displayed, and the unit will automatically return to the Normal Operation Mode. % Saturation will be displayed in the main screen. Record value as "Calibrated % Saturation".
13) Press mode until mg/L is displayed. Record the displayed value as the "Calibrated Meter Reading (mg/L)" on the calibration sheet.
    NOTE: "D.O. Table Value" and "Calibrated Meter Reading" value should be within ±0.5 mg/L of each other.

Once calibrated, the YSI-85 should remain "ON" until terminal calibration checks are completed at the end of the sampling day; otherwise, meter calibrations may be compromised.

Terminal D.O. Calibration Check (Post-Sampling Meter Check)

a. Repeat calibration steps 1 thru 8.

b. The post-sampling "D.O. Table Value" and the post-sampling "Initial Meter Reading" should be within ±0.5 mg/L.

MAINTENANCE

Probe Storage - Calibration/Storage Chamber:
Store probe in the Calibration/Storage Chamber filled with a small, moist, clean sponge.

D.O. Membrane Replacement:
Replace membrane if: calibration is impossible; readings are erratic; or membrane is damaged.

1) Remove the probe sensor guard.
2) Remove and discard old membrane cap.
3) Rinse the sensor tip with distilled or deionized water.
4) Prepare electrolyte solution (Na_2SO_4, KCl) according to the directions on the bottle (included in Membrane Cap Kit).
   When a new electrolyte solution is prepared, record the preparation date (in permanent ink) on the side of the solution bottle. Discard electrolyte solutions 12 months after the recorded preparation date.
5) Fill new membrane cap half-full with electrolyte solution.
6) Screw membrane cap onto the probe (small amount of electrolyte should overflow).
7) Re-attach the probe sensor guard.

Cleaning Dirty, Tarnished Silver Anode and Gold Cathode:

1) Remove membrane and soak probe overnight in 3% ammonium hydroxide (NH_4OH).
2) Rinse sensor tip with deionized water.
3) Use 400 or 600 grit wet/dry sandpaper to clean and polish the anode and cathode.
4) Rinse with deionized water.
5) Install new membrane.
6) Turn meter "ON" and allow unit to stabilize for at least 30 minutes to 3 hours before calibrating.

It may take several hours for the meter to stabilize.

YSI 85 - Accumet 8-15-07.doc Page 1 of 4 YSI-8/15/2007

SPECIFIC CONDUCTANCE

YSI 85

CALIBRATION CHECK

"Dry Air" Check (Zero):

1) Turn meter "ON".
2) Press "MODE" to advance to Specific Conductance.
   "°C" should be flashing on the display.
3) The displayed value should be within ± 2 µS of zero. Record displayed value as "Initial Meter Reading" in the "Dry Air" section of the calibration sheet. If the displayed value is not within the range of -2 to 2 µS, clean, rinse and thoroughly dry the conductivity cell.

Note (YSI 85 Meters): You are only checking the meter's calibration (as opposed to actually calibrating the meter); therefore, no value should be recorded as the "Calibrated Meter Reading" on the calibration sheet.

Check using Conductivity Standard:

Conductivity calibrations should be checked using a standard that is similar to the anticipated measurements to be collected in the field that day. Record the standard's "true value" (found on the certificate of analysis or bottle label) and lot number (also called analysis number) on the calibration sheet. Traceable® Certificate of Analysis for Conductivity Solution sheets (1 certificate for each lot number) should be retained and stored in a notebook.

4) Rinse probe with distilled or deionized water and wipe dry with a chem-wipe or a lint-free cloth.
5) Rinse probe with a small amount of conductivity standard (make sure some of the standard rinse goes into the oval-shaped hole on the side of the probe).
6) Insert probe into a vessel containing the standard such that the conductivity cell is completely submersed.
   Do not rest the probe on the bottom of the container (probe should be approximately ¼ inch from the bottom).
   Move the probe from side to side to dislodge any bubbles; wait for readings to stabilize.
7) Record the displayed value as the "Initial Meter Reading" in the "Conductivity Standard" section of the calibration sheet. This value must be ± 10% of the standard value.
8) If the displayed value is not within ±10% of the standard value, clean probe and repeat calibration check with a **FRESH** standard.

Calibration Confirmation:

9) Rinse probe with distilled or deionized water and wipe dry with a chem-wipe or a lint-free cloth. Confirm that the meter display is reading 0 (zero) µS before moving to the next step.
10) Repeat steps 5 thru 8 with a second standard that has a different specific conductance value. Record value in the "Calibration Check" section of the calibration sheet.

Terminal Conductivity Calibration Check (Post-Sampling Meter Check)

a. Repeat steps 1 thru 10, and record values on the calibration sheet.
b. For the "Dry Air" check, displayed value should be between –2 and 2 µS.
c. For "Conductivity Standard" and "Calibration Check", values should be within ±10% of the standard.

MAINTENANCE

Cleaning Conductivity Cell:

1) Dip the conductivity cell (oval-shaped hole on the side of the probe) in alcohol or a mild detergent and agitate for 2 to 3 minutes. Remove from cleaning solution.
2) Use a soft nylon brush to remove any contaminants from the inside of the electrode chamber.
3) Repeat steps 1 and 2 until the cell is clean.
4) Rinse cell with deionized water.
5) Dry cell thoroughly, and verify that the unit reads between -2 to 2 µS in dry air.

If the above cleaning procedure does not restore the meter, repeat steps 1-5 using 1:1 isopropyl alcohol and 1 N HCl. This more extensive cleaning is rarely required.

Conductivity Cell Check:

If having difficulty calibrating or readings are erratic, check the conductivity cell constant:

1) Turn meter "ON". The unit will go through a self-test procedure.
2) A value will be displayed, along with "CEL". The displayed value should be between 4.8 and 5.2.

If the displayed value is not within the specified range, clean the cell, and recalibrate the meter (see Meter Manual for calibration instructions; re-calibration is RARELY required).

pH

ACCUMET AP61

CALIBRATION

Two-point pH Calibration Required:

Clear Previous Slope Efficiency

1) Turn meter on.
2) Press "SETUP" to view the electrode efficiency (as percent slope) stored in the meter. In most cases, you do NOT want to accept this existing efficiency and should clear it.
3) Press "SETUP" again to access the clear buffers option.
4) "Clr" will be displayed on the unit. Press "ENTER" to clear the existing buffers and return to the Measure screen.

1st Calibration Point (start with 7 Buffer):

5) Open fill hole on probe.
6) Rinse sensors with distilled or deionized water and blot dry.
7) Rinse sensors with small amount of 7 buffer. Discard buffer rinse.
8) If the meter is not in the pH Mode, press "MODE" until the display indicates the pH mode.
9) Immerse the end of the probe into 7 buffer. Wait for reading to stabilize.
10) Record buffer temperature. Record displayed value as the "Initial Meter Reading" for Buffer # 1 on the calibration sheet.
11) Press "std" to access the Standardize Screen. The buffer group used by the meter will be displayed briefly, and the prompt "PRESS std TO STANDARDIZE" will flash.
12) Press "std" again to initiate standardization. The meter will automatically recognize the buffer and display the value on the screen.
13) Record the displayed value as "Calibrated Meter Reading" for Buffer #1 on the calibration sheet.

2nd Calibration Point (4 or 10 Buffer):

14) Repeat steps 5 thru 12 using a pH buffer similar to the anticipated pH of the samples to be measured.
15) Record values as instructed above for Buffer #2 on the calibration sheet.

Slope Efficiency Check:

16) When the meter accepts the second buffer, the unit will briefly display the efficiency (as the percent slope) of the electrode's performance.
17) Record displayed value as "Slope Efficiency" on the calibration sheet.

    The "Slope Efficiency" should be ≥ 95%.

    If the menu changes before the displayed value can be recorded, the slope efficiency can be accessed by pressing "SETUP".

Confirmation Buffer (7.0)

18) Rinse sensors with distilled or deionized water and blot dry.
19) Rinse sensors with small amount of 7 buffer. Discard buffer rinse.
20) Immerse the probe into 7 buffer again to confirm the calibration.
21) Record the displayed value as the "Meter Reading" under "Confirmation Buffer" on the calibration sheet
    Confirm that the "Meter Reading" value is within ± 0.1 of the buffer value (between 6.9 and 7.1).

Terminal pH Check (Post-Sampling Meter Check)

a. Repeat calibration steps 5 thru 10 (for 7 buffer) and record displayed value on the calibration sheet.
   This value should be within ±0.2 of 7 (for 7 buffer).
b. Repeat procedure for the other buffer (Buffer #2) that was used to calibrate meter. Record value on calibration sheet
   Value should be within ±0.2 of Buffer #2.

pH

ACCUMET AP61

MAINTENANCE

Electrolyte Level

Check the electrolyte level frequently. The electrolyte level should be within ¼ inch of the cap. Fill as needed.

Refilling Electrolyte:

1) Open the fill hole on the cap ring.
2) Hold probe such that sensors are facing downwards.
3) Insert the tip of the electrolyte-dispensing bottle into the fill hole and press firmly to make an airtight seal. Squeeze the dispensing bottle for approximately 30 seconds or until adequately filled.
4) Remove dispensing bottle from fill hole.

Cleaning pH Glass Electrode:

1) Wet a cotton swab with alcohol.
2) Gently swab the pH glass electrode.
3) Rinse electrode with deionized water

Cleaning the pH reference electrode:

If crystal residue forms on electrode junction or inside the electrolyte reservoir:

1) Empty filling solution from reservoir by shaking it out through the fill holes.
2) Rinse electrolyte reservoir repeatedly with distilled or deionized water until all crystals are dissolved. Warm tap water can be used as a preliminary step to quickly dissolve crystals.
3) Refill reservoir with the electrolyte (4M KCl saturated with AgCl).

Installing and Filling pH Reference Electrode:

1) Carefully remove new probe from packaging.
   Be careful when handling the probe; even a small scratch on the glass bulb can cause irreparable damage.
2) Rinse electrode and sensors with distilled or deionized water to remove crystal residue that may have formed on the surface during storage.
3) Open the fill hole on the cap ring.
4) Check the electrolyte level. If level is low, add electrolyte (4M KCl saturated with AgCl) as described above. Electrolyte solution is included with each new electrode.
5) Connect new probe to the display unit.
   Soak new probe in pH 4 buffer for 10 minutes prior to standardization.

Probe Storage:

When not in use, store the probe in pH 4 buffer and confirm that the fill hole is closed.
Never store probe in distilled or deionized water!

Appendix 3: DWR's YSI 6920 Multiparameter Guidance Sheet

DISSOLVED OXYGEN

YSI 6920 with ROX (OPTICAL D.O.) SENSOR

CALIBRATION

D.O. Calibration for YSI Meters with ROX (Optical D.O.) Sensor:
(% AIR CALIBRATION IN WATER-SATURATED AIR)

1) Remove calibration storage cup from sonde, and confirm that optical D.O. probe has been stored in moist environment. Place calibration cup on work surface with the uncapped end facing upward.
2) Use lens tissue to carefully dry all sensors. The temperature and optical D.O. sensors must be completely dry.
3) Pour a small amount of tap water into the calibration cup (just enough to completely cover the bottom of the calibration chamber and create a 100% humid environment). Temperature and optical D.O. sensors CANNOT be in contact with water during calibration.
4) Place probes (pointing downward) into calibration cup carefully so that no water droplets get on the temperature sensor or optical D.O. sensor.
5) Twist the calibration cup onto the sonde no more than one or two threads, so that the cup is vented to the atmosphere.
6) Wait approximately 15 minutes to guarantee thermal equilibration between the temperature and optical D.O. sensors. To observe readings during this time, place the sonde in **Run Mode**:
   650 Main Menu ⇨ Sonde run
7) Access the **D.O. Calibration Menu**:
   650 Main Menu ⇨ Sonde Menu ⇨ Calibrate ⇨ Optic-T Dissolved Oxy ⇨ ODOsat % ⇨ 1 point
   NOTE: Dissolved oxygen should always be calibrated using % saturation.
   Calibrations based on "mg/l" require a water sample with a known D.O. concentration (requires Winkler titration).
8) Enter Barometric Pressure in mmHg. Record "Barometric Pressure" and "Altitude" on calibration sheet.
   These values are available on the *Dissolved Oxygen Table* for your corresponding regional office. All calibrations should be performed in a controlled environment (field calibrations are not recommended).
9) Real-time values will be displayed for all active parameters. When readings are stable for 30 seconds, record the following values on the calibration sheet: "Temperature", "Initial % Saturation", and "Initial Meter Reading (mg/L)".
10) Use the *Dissolved Oxygen Table* for your location to find the "D.O. Table Value" (based on the temperature displayed on the meter), and record value on calibration sheet.
11) Press ↵ (Enter) to calibrate Dissolved Oxygen. "**Calibrated**" should be displayed at the top of the screen.
12) On the calibration sheet, record the displayed mg/L value as "Calibrated Meter Reading" and the % SAT value as the "Calibrated % Saturation" value.
    * **NOTE: The "D.O. Table Value" and the "Calibrated Meter Reading" value should be within ±0.5 mg/L of each other.**
13) Press ↵ (Enter) to return to the **D.O. Calibration Menu**. Press "**Esc**" (3 times) to return to the main menu.

Terminal Calibration Check (Post-Sampling Meter Check)

14) Repeat calibration steps 1 thru 6. Record "Temperature", "% Saturation", "Initial Meter Reading" from the **Run Mode**.
15) Record the barometric pressure and altitude where the terminal calibration check is being performed.
16) Repeat calibration step 10.
17) The post-sampling "D.O. Table Value" and the post-sampling "Initial Meter Reading" should be within ±0.5 mg/L of each other.

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DISSOLVED OXYGEN

YSI 6920 with ROX (OPTICAL D. O.) SENSOR

MAINTENANCE

Probe Storage:

The probe must be stored in a moist environment.

Store probe in Calibration/Storage Cup approximately half full of tap water. Do not use distilled water (this will negatively affect the pH probe).

During long-term storage, inspect at least once a month to ensure the probe is still in a moist environment.

Optical D.O. Membrane Re-hydration:

If left in ambient air for more than 2 hours, the optical D.O. membrane must be re-hydrated.

1) Pour approximately 400 mL of water into a 600 mL glass beaker (plastic containers should NOT be used). Use a thermostatted hotplate or an oven to heat the water to a consistent temperature of 50° C, ± 5° C.
2) Place the probe tip containing the optical D.O. membrane in warm water and leave it at the elevated temperature for approximately 24 hours. Cover vessel to minimize evaporation.
3) After re-hydration, store the probe in either water or water-saturated air before calibration and deployment.

Optical D.O. Sensor Cleaning:

Clean only with a lens tissue that has been moistened with water.

NEVER use alcohol or other organic solvents; organic solvents will ruin the membrane.

Wiper Operation:

The wiper can be used as-needed to wipe the sensor face during sampling.

1.) Use the display menus to activate the wiper:
`650 Main Menu` ⇨ `Sonde run` ⇨ `Clean Optics` (upper right corner of screen) ⇨ Press ↵ (Enter) to clean optics.
2.) After the wiper has finished rotating, wait 30 seconds before recording a measurement.

Changing the Wiper:

1.) Loosen setscrew until the wiper can be removed from the shaft.
2.) Place new wiper on the wiper shaft.
3.) Gently press the wiper against the face of the probe until the foam pad is compressed to roughly one half of the original thickness and then tighten the setscrew.
It is recommended that a business card be slid in between the wiper arm body and the probe face when installing the wiper. After installation, a gap about the thickness of a business card should be between the wiper arm body and the face of the probe
4.) Rotate the wiper to confirm that it "parks" correctly (180° from the ROX membrane):
`650 Main Menu` ⇨ `Sonde run` ⇨ `Clean Optics` (upper right corner of screen) ⇨ Press ↵ (Enter) to clean optics.

NEVER rotate the wiper manually. This will void the warranty.

Optical D.O. Membrane (sensor cap) Replacement:

Optical D.O. membrane should be replaced once a year or if damaged.

Detailed instructions are sent with the new membrane kit (YSI 6155).

When installing a new membrane, new calibration codes (included with each new membrane) must be entered.

SPECIFIC CONDUCTANCE

YSI 6920

CALIBRATION

THREE-STEP SPECIFIC CONDUCTIVITY PROCEDURE:

I. "DRY AIR" (ALWAYS ZERO):

The "Dry Air" step is a **check** for YSI meters.

1) Attach calibration cup to probe. Fill calibration cup half-full with deionized water and seal with lid. Shake probe to rinse. Repeat.
2) Remove calibration cup. Place cup on work surface with the uncapped end facing upward.
3) Use a cotton swab to dry the inside of the conductivity cells.
4) Record displayed value as "Initial Meter Reading" in the "Dry Air" section of the calibration sheet. The probe should read close to zero (± 2).
   If the reading is not within ± 2, follow cleaning procedure, and repeat calibration procedure.

II. CONDUCTIVITY STANDARD:

Calibrations should be performed using a **fresh, certified** conductivity standard that is similar to the conductivity of the samples to be collected that day. Record the standard's "true value" (found on the certificate of analysis or bottle label) and lot number (also called analysis number) on the calibration sheet. Traceable® Certificate of Analysis for Conductivity Solution sheets (one certificate for each lot number) should be retained and stored in a notebook.

5) Re-attach calibration cup. Fill cup half-full with deionized water. Seal cup with lid and shake probe to rinse. Repeat.
6) Rinse sensors with small amount of fresh conductivity standard. Discard rinse.
7) Remove calibration cup from sonde. Place cup on work surface with the uncapped end facing upward.
8) Pour conductivity standard into the calibration cup. Make sure there is enough standard to cover the entire conductivity cell when the probe is placed in the cup.
9) Place sonde into the calibration cup. Agitate sonde to remove air bubbles trapped in the conductivity cells. Air bubbles will give erroneously low readings.
10) Enter Run mode to view readings:
    `650 Main Menu ⇨ Sonde run`
11) Press **Esc** to go back to the `650 Main Menu`.
12) Access the `Calibrate menu` for Specific Conductance:
    `650 Main Menu ⇨ Sonde menu ⇨ Calibrate ⇨ Conductivity ⇨ SpCond`
13) Enter the True Value of the conductivity standard in **milliSiemens/cm**. Press ↵ (Enter).
14) Wait for readings to stabilize.
15) Record displayed value as "Initial Meter Reading" in the "Conductivity Standard" section of the calibration sheet.
16) Press ↵ (Enter) to calibrate meter. The message in the top center of the screen will switch to "`Calibrated`".
    Record displayed value as "Calibrated Meter Reading" on calibration sheet.
    Never accept an out-of-range calibration.
17) Press ↵ (Enter) to return to the `Calibrate menu`.

III. CALIBRATION CHECK:

18) Rinse with deionized water and wipe dry with a lens tissue or a lint-free cloth.
19) Confirm that the meter display is reading 0 (zero) µS before going to the next step.
20) Repeat steps 5-10 with a fresh conductivity standard of a value different from the one used in the previous calibration steps. Choose a standard that will give the best range of values for the anticipated samples to be collected.
21) Record SpCond value as "Initial Meter Reading" in the Calibration Check section on the calibration sheet. The value must be within 10% of the standard.

Terminal Calibration Check (Post-Sampling Meter Check)

a. Repeat calibration steps 1 thru 4, and record value in the "Dry Air" section on the calibration sheet.
   For the "Dry Air" check, displayed value should be between –2 and 2 µS.
b. Repeat calibration steps 5 thru 10. Record value in the "Conductivity Standard" section on the calibration sheet.
   "Conductivity Standard" value should be within ±10% of the standard.
c. Repeat step 18-21, and record value in the "Calibration Check" section on the calibration sheet.
   "Calibration Check" value should be within ±10% of the standard.

SPECIFIC CONDUCTANCE

YSI 6920

MAINTENANCE

✱ **Never accept an out-of-range calibration**!

Checking the Conductivity Cell Constant:

When troubleshooting the conductivity probe, first check the cell constant.

1) `650 Main Menu ⇨ Sonde menu ⇨ Advanced ⇨ Cal constants`
2) The value displayed next to "`Cond`" should be 5.0, ± 0.45.
   Numbers outside of this range indicate a problem in the calibration process or that a contaminated standard was used to calibrate the meter.
3) If conductivity cell constant is not within the acceptance range (between 4.55 and 5.45), clean the cell, and reset the calibration cell constant (see instructions below).

Cleaning Conductivity Sensor:

Conductivity cell should be rinsed with deionized water after field use.

Clean conductivity cell frequently. A clean cell is imperative for accurate readings.

1) Dip small cleaning brush (provided with new meters) into distilled or deionized water and insert brush into each hole 15-20 times. For a more thorough cleaning, use a mild dishwashing detergent with the brush.
2) Rinse sensor thoroughly with deionized water.
3) Perform the Dry Air Check described in Calibration Steps 1-4 to ensure probe reads close to zero in air.

Reset Calibration Cell Constant:

1) Reset the calibration cell constant by accessing the `Calibrate menu`:
   `650 Main Menu ⇨ Sonde menu ⇨ Calibrate ⇨ Conductivity ⇨ SpCond`
2) When prompted to "`Enter the SpCond (mS/cm)`", press and hold the **Enter** key (↵) and press the **Esc** key.
3) The menu will ask "**`Uncal?`**" Select Yes. Press the **Enter** key (↵)
4) Recalibrate the meter using fresh, certified conductivity standards.

pH — **YSI 6920**

CALIBRATION

Two-point pH Calibration Required (Three-point pH Calibration is Optional):

1ST CALIBRATION POINT (ALWAYS START WITH 7 BUFFER):

1) Rinse probes and calibration cup with distilled water.
2) Rinse probes and calibration cup with small amount of 7 pH buffer. Discard buffer rinse. Repeat.
3) Remove calibration cup from sonde. Place cup on work surface with uncapped end facing upward.
4) Fill calibration cup with enough **fresh** buffer to cover the pH glass bulb and temperature sensor.
5) Check temperature of pH buffer. Record value on calibration sheet.
   To view buffer temperature: `650 Main Menu ⇨ Sonde run`
6) Note: If the temperature at which you are calibrating is significantly different from 25° C, check the buffer bottle for the corrected pH value at the corresponding temperature (the corrected pH value will be used in step 8).
6) Access `Calibrate menu` for pH: `650 Main Menu ⇨ Sonde menu ⇨ Calibrate ⇨ ISE1 pH`
7) Choose either the `2-point` or `3-point calibration`.
8) The prompt "`Enter 1st pH`" will appear. Enter 7.0 (or, if applicable, the corrected pH value from step 5).
9) Real-time readings will be displayed. When readings have stabilized, record displayed pH value as "Initial Meter Reading" for Buffer # 1 on calibration sheet.
10) Press ↵ (Enter) to calibrate.
11) "`Calibrated`" will be displayed at the top of the screen. Record displayed pH value as "Calibrated Meter Reading" for Buffer #1.

2ND CALIBRATION POINT:

12) Remove calibration cup from sonde.
Note: "Calibrated" should still be displayed at the top of the screen. Remain on the same display screen as in Step 11 in order to see the real-time temperature reading for the 2nd buffer. Do not return to the calibration menu.
13) Rinse probes with distilled water.
14) Rinse probes and calibration cup with small amount of 2nd buffer (either 4 or 10 pH buffer).
15) Fill calibration cup with enough fresh buffer to cover the pH glass bulb and temperature sensor.
16) Real-time readings will be displayed. When readings have stabilized, record the temperature reading.
Note: If the temperature at which you are calibrating is significantly different from 25° C, check the buffer bottle for the corrected pH value at the corresponding temperature (the corrected pH value will be used in step 18).
17) Press ↵ (Enter) to return to the `Calibrate menu`. The prompt "Enter 2nd pH" will be displayed.
18) At the "`Enter 2nd pH`" prompt, enter value of 2nd buffer (or, if applicable, the corrected pH value from Step 16).
19) Real-time readings will be displayed. When readings have stabilized, record displayed pH value as "Initial Meter Reading" for Buffer # 2 on the calibration sheet.
20) Press ↵ (Enter) to calibrate.
21) "`Calibrated`" will be displayed at the top of the screen. Record displayed pH value as "Calibrated Meter Reading" for Buffer #2.

If you chose to do a "`3-point calibration`", repeat steps 12 through 21 using the 3rd buffer.

If only performing a 2-point calibration, press ↵ (Enter) and then **Esc** to return to the main menu.

CONFIRMATION BUFFER:

22) Rinse probes and calibration cup with distilled water.
23) Rinse probes and calibration cup with small amount of 7.0 pH buffer. Discard buffer rinse. Repeat.
24) Remove calibration cup from sonde. Place cup on work surface with the uncapped end facing upward.
25) Fill the calibration cup with enough **fresh** buffer to cover the pH glass bulb and temperature sensor.
26) Enter Run mode to view readings: `650 Main Menu ⇨ Sonde run`
27) Wait 1 to 3 minutes for pH readings to stabilize.
28) Record the displayed pH value as the "Meter Reading" under "Confirmation Buffer 7.0" on the calibration sheet
29) Confirm that the "Meter Reading" value is within ± 0.1 of the buffer value (between 6.9 and 7.1).

Terminal Check (Post-Sampling Meter Check)

a. Repeat steps 22 thru 29 (for 7 buffer); record displayed value on calibration sheet. Value should be within ±0.2 of 7.0.
b. Repeat steps 22 thru 29 for Buffer #2. Record value on calibration sheet. Value should be within ±0.2 of Buffer #2.

pH — **YSI 6920**

MAINTENANCE

Indicators that maintenance is needed:
Difficulty calibrating pH sensor, slow response, erratic readings, clogged or black reference junction, coated glass bulb.

Probe Storage:
Store probe in calibration/storage cup filled half-full with tap water (never use distilled water to store probe). If probe will not be used for several months, remove probe and store in pH 4 buffer or electrode storage solution.

Probe Lifespan:
The pH probe has a lifetime of approximately 18-24 months (in some cases, probes may last 3+ years).

When troubleshooting pH sensor problems, start by checking age of probe and replace as-needed:
Near the silver stainless steel connector of each probe is the imprint "YSI 6561" followed by 2 numbers and a letter. The 2 numbers and the letter indicate the year and month in which the probe was made. For instance, 07D means the probe was made in April, 2007. (i.e. A=Jan, B=Feb, etc.).

Troubleshooting with mV readings:
1) Activate pH mV readings in the `Report menu:`
   `650 Main Menu ⇨ Sonde menu ⇨ Report ⇨ pH mV`
   Note: pH mV is active when a black dot appears in the circle next to it. Press "Enter" to toggle between active and inactive.
2) Follow steps for pH calibration. During calibration, record pH mV values from the "Calibrated " screen for each buffer.
3) Evaluate the pH mV values:
   The span or "slope" between the pH 4 and pH 7 and between pH 7 and pH 10 should be approximately 165 to 180 mV.
   pH 7 should be 0 mV ± 50 mV. pH 4 should be 180 mV ± 50 mV. pH 10 should be -180 mV ± 50 mV.
   Example: If a probe reads +10 mV in pH 7 buffer, then the probe should also read between 175 and 190 mV in pH 4 buffer, and between -155 mV and -170mV in pH 10 buffer.
4) If the mV values fall outside the range of 160-180 mV, the probe should be replaced soon.
   Note: The probe will no longer calibrate when the span is outside of the range of 150-210 mV.

General pH Probe Cleaning:
Use deionized water and a soft lens cloth or a cotton swab to remove foreign material from the glass bulb. If good response is not restored, perform the following procedure:
1) GENTLY clean the glass bulb and white probe face by carefully rubbing a cotton swab soaked in mild dishwashing detergent. Apply little to no pressure, as the glass bulb is very thin and fragile!
2) Rinse probe thoroughly with deionized water.
3) Wipe probe with cotton swab that has been saturated with water. Rinse probe again.

Advanced pH Probe Cleaning and Restoration:
To remove more resistant deposits and biological growth, use HCl acid and bleach.
The need and frequency depend on the type of surface water being monitoring.
The probe must be removed from the sonde before advanced cleaning.
To perform an advanced cleaning, refer to Section 2.10.2 of the YSI 6-Series User Manual.

Reference Junction:
The reference junction is a small tab located between the edge of the white surface of the pH probe face and the gray raised area around the pH probe face.
When new, the junction will be an off-white color.
As it ages, the junction will become darker.
A black reference junction coupled with slow response and/or erratic readings indicates a more advanced cleaning may be needed.

Appendix 4: DWR's YSI Pro Plus Multiparameter Guidance Sheet

DISSOLVED OXYGEN

YSI PRO PLUS with POLARGRAPHIC SENSOR

CALIBRATION

D.O. Calibration for YSI Meters with Polargraphic Sensor:

All calibrations should be performed in a controlled environment. Field calibrations are not recommended.

I. BAROMETER CALIBRATION

Access the Barometer Calibration Menu:

1) Press **Cal** key, highlight **Barometer,** and press **Enter**.
2) Highlight **mmHg**, and press **Enter**.
3) Highlight **Calibration Value**, and press **Enter.** Input the "true" barometric pressure (mmHg). Highlight **<<<Enter>>>,** and press **Enter**. *True barometric pressure is listed on the Dissolved Oxygen Table for your corresponding regional office.*
4) Wait for readings to stabilize. Record displayed value as "Initial Reading" in the "Barometer Calibration" section of the calibration sheet.
5) Highlight **Accept Calibration** and press **Enter** to calibrate Barometric Pressure.
6) "`Calibrating Channel...`" and then "`Saving Configuration...`" will be displayed at bottom of calibration screen before returning to the main screen.
7) Record displayed value as "Calibrated Value" in the "Barometer Calibration" section of the calibration sheet.

II. (% AIR CALIBRATION IN WATER-SATURATED AIR)

8) Remove calibration storage cup from sonde. Confirm D.O. probe has been stored in moist environment. Place calibration cup on work surface with uncapped end facing upward.
9) Use lens tissue to carefully dry all sensors. Temperature and D.O. sensors must be completely dry.
10) Pour small amount of tap water into calibration cup (approximately 1/8" - just enough to completely cover the bottom of the calibration cup and create a 100% humid environment). Temperature and D.O. sensors CANNOT be in contact with water during calibration.
11) Carefully place probes (pointing downward) into calibration cup so that no water gets on the temperature or D.O. sensor.
12) Twist calibration cup onto sonde **no more than 1 or 2 threads**, so the cup is able to vent to the atmosphere.
13) Wait at least 15 minutes for D.O. sensor to stabilize.
14) Record the following values on calibration sheet: "Temperature", "Initial % Saturation", and "Initial Meter Reading (mg/L)".
15) Use the *Dissolved Oxygen Table* for your location to find the "D.O. Table Value" (based on the temperature displayed on the meter), and record value on calibration sheet.

Access the D.O. Calibration Menu:

16) Press **Cal** key, highlight **DO,** and press **Enter**.
17) Highlight **DO%**, and press **Enter**.
    NOTE: Dissolved oxygen should always be calibrated using % saturation.
    Calibrations based on "mg/l" require a water sample with a known D.O. concentration (requires Winkler titration).
18) In the "Dissolved Oxygen" section of the calibration sheet, record the "Barometric Pressure" displayed on the meter and the altitude for your location (provided on regional office *Dissolved Oxygen Tables*).
19) Highlight **Accept Calibration** and press **Enter** to calibrate Dissolved Oxygen.
    "`Calibrating Channel...`" and then "`Saving Configuration...`" will be displayed at bottom of calibration screen before returning to the main screen.
20) On the calibration sheet, record the displayed mg/L value as "Calibrated Meter Reading" and the DO% value as the "Calibrated % Saturation" value.

    *** NOTE: The "D.O. Table Value" and the "Calibrated Meter Reading" value should be within ±0.5 mg/L of each other.**

Terminal Calibration Check (Post-Sampling Meter Check)

NOTE: Barometric pressure is not checked or recalibrated post-sampling.

a. Repeat calibration steps 8 thru 13. Record "Temperature", "% Saturation", "Initial Meter Reading".
b. Record the barometric pressure and altitude for the location post-sampling checks are being performed.
c. Repeat calibration step 15.
d. The post-sampling "D.O. Table Value" and the post-sampling "Initial Meter Reading" should be within ±0.5 mg/L of each other.

DISSOLVED OXYGEN

YSI PRO PLUS with POLARGRAPHIC SENSOR

MAINTENANCE

Cracked Probe:

If meter readings are unusual and calibrating the meter does not correct the issue, check the condition of the D.O. probe – sometimes a crack can develop in the plastic along the side of the probe. Cracked probes should be replaced immediately.

Probe Storage:

Store probe in Calibration/Storage Cup with a small of tap water to create a 100% saturated air environment.

During storage, probes should not be submerged in water.

Do not use distilled water (this will damage the pH probe).

During long-term storage, inspect at least once a month to ensure the probe is still in a moist environment.

D.O. Membrane Replacement (YSI 5908- Yellow Teflon):

Replace electrode solution and membrane at least every 30 days during regular use or if: bubbles are visible under membrane; significant deposits of dried electrolyte are visible on membrane; calibration is impossible; readings are erratic or unstable; or membrane is damaged.

1) Remove and discard old membrane cap.
2) Rinse sensor tip with distilled or deionized water.
3) Prepare electrolyte solution (Na_2SO_4, KCl) according to the directions on the bottle (included in Membrane Cap Kit). Newly prepared solution must sit for 1 hour before using to prevent air bubbles under the membrane.

   When a new electrolyte solution is prepared, record preparation date (in permanent ink) on the side of the solution bottle. Discard electrolyte solutions 12 months after the recorded preparation date.
4) Fill new membrane cap half-full with electrolyte solution. Do not touch membrane surface. Tap side of cap lightly to release bubbles.
5) Screw membrane cap onto probe (small amount of electrolyte should overflow).
6) Re-attach probe sensor guard.

Cleaning Dirty, Tarnished Silver Anode and Gold Cathode:

SANDING AND CLEANING THE ELECTRODE ARE NOT PART OF THE ROUTINE MAINTENANCE AND SHOULD ONLY BE PERFORMED WHEN ABSOLUTELY NECESSARY! If performed too frequently, the electrode will be destroyed!

1) Remove membrane and soak probe overnight in 3% ammonium hydroxide (NH_4OH).
2) Rinse sensor tip with deionized water.
3) Use 400 or 600 grit wet/dry sandpaper to clean and polish the anode and cathode – no more than 3 to 4 twists of the sandpaper should be sufficient to remove any deposits or tarnish.
4) Rinse heavily with deionized water.
5) Install new membrane.
6) Turn meter "ON" and allow unit to stabilize for at least 30 minutes to 3 hours before calibrating.

May take several hours for the meter to stabilize.

SPECIFIC CONDUCTANCE

YSI PRO PLUS

CALIBRATION

THREE-STEP SPECIFIC CONDUCTANCE PROCEDURE:

I. "DRY AIR" (ALWAYS ZERO):

The "Dry Air" step is a **check** for YSI meters.

1) Attach calibration cup to probe. Fill cup half-full with deionized water and seal with lid. Shake probe to rinse.
2) Remove calibration cup. Place cup on work surface with the uncapped end facing upward.
3) Use a cotton swab to dry the inside of the conductivity cells.
4) Record displayed value as "Initial Meter Reading" in the "Dry Air" section of the calibration sheet. The probe should read close to zero (± 2).
   If the reading is not within ± 2, follow cleaning procedure, and repeat calibration procedure.

II. CONDUCTIVITY STANDARD:

Calibrations should be performed using a **fresh, certified** conductivity standard that is similar to the conductivity of the samples to be collected that day. Record the standard's "true value" (found on the certificate of analysis or bottle label) and lot number (also called analysis number) on the calibration sheet. Traceable® Certificate of Analysis for Conductivity Solution sheets (one certificate for each lot number) should be retained and stored in a notebook.

5) Re-attach calibration cup. Fill cup half-full with deionized water. Seal cup with lid and shake probe to rinse. Discard rinse water.
6) Rinse sensors with small amount of conductivity standard. Discard rinse.
7) Pour fresh conductivity standard (≥1000 µS/cm) into the calibration cup. Make sure there is enough standard to cover the entire conductivity cell and temperature sensor when the probe is placed in the cup.
8) Tap or agitate sonde to remove air bubbles trapped in the conductivity cells. Air bubbles will result in erroneously low readings.
9) Press **Cal** key, highlight **Conductivity**, and press **Enter**.
10) Highlight **Sp. Conductance** and press **Enter**.
11) Highlight **SPC- uS/cm** and press **Enter**.
12) Highlight **Calibration Value** and press **Enter**. Input the True Value of the conductivity standard in **microSiemens/cm (µs/cm)**. Highlight **<<<Enter>>>**, and press **Enter.**
13) Wait for readings to stabilize. Record displayed value as "Initial Meter Reading" in the "Conductivity Standard" section of the calibration sheet.
14) Highlight **Accept Calibration** and press **Enter** to calibrate meter.
    "`Calibrating Channel…`" and then "`Saving Configuration…`" will be displayed at bottom of calibration screen before returning to the main screen.
15) Record displayed value as "Calibrated Meter Reading" on calibration sheet.
    Never accept an out-of-range calibration (flagged by an error message on the meter).

III. CALIBRATION CHECK:

16) Rinse with deionized water and wipe dry with a lens tissue or a lint-free cloth.
17) Confirm that the meter display is reading 0 (zero) µS before going to the next step.
18) Repeat steps 5-8 with a conductivity standard of a value different from the one used in the previous calibration steps. Choose a standard that will give the best range of values for the anticipated samples to be collected.
19) Record SpCond value as "Initial Meter Reading" in the Calibration Check section on the calibration sheet. The value must be within 10% of the standard.

Terminal Calibration Check (Post-Sampling Meter Check)

a. Repeat calibration steps 1 thru 4, and record value in the "Dry Air" section on the calibration sheet.
   For the "Dry Air" check, displayed value should be between -2 and 2 µS.
b. Repeat calibration steps 5 thru 8. Record value in the "Conductivity Standard" section on the calibration sheet.
   "Conductivity Standard" value should be within ±10% of the standard.
c. Repeat steps 16-19, and record value in the "Calibration Check" section on the calibration sheet.
   "Calibration Check" value should be within ±10% of the standard.

SPECIFIC CONDUCTANCE

YSI PRO PLUS

MAINTENANCE

✱ **Never accept an out-of-range calibration**! (flagged by an error message on the meter display)

Checking the Conductivity Cell Constant:

When troubleshooting the conductivity probe, first check the cell constant.

1) Press **Folder** Key. Highlight **View GLP**, and press **Enter**. Scroll to most recent conductivity calibration to view the Cal Cell Constant.
2) The value displayed next to "`Cal Cell Constant`" should be 5.0, ± 0.45.
   Numbers outside of this range indicate a problem in the calibration process or that a contaminated standard was used to calibrate the meter.
3) If conductivity cell constant is not within the acceptance range (between 4.55 and 5.45), clean the cell, and reset the calibration cell constant (see instructions below).

Cleaning Conductivity Sensor:

Conductivity cell should be rinsed with deionized water after field use.

Clean conductivity cell frequently. A clean cell is imperative for accurate readings.

1) Dip small cleaning brush (provided with new meters) into distilled or deionized water and insert brush into each hole 15-20 times. For a more thorough cleaning, use a mild liquid or foam dishwashing detergent with the brush.
2) Rinse sensor thoroughly with deionized water.
3) Perform the Dry Air Check described in Calibration Steps 1-4 to ensure probe reads close to zero in air.

Reset Calibration Cell Constant:

Reset the calibration cell constant by accessing the Calibrate menu:

1) Press **Cal** Key, highlight **Restore Default Cal,** and press **Enter**. Highlight **Conductivity**, and press **Enter.**

2) The menu will ask **"Are you sure you want to remove the current user calibration parameters for this channel?"** Highlight **Yes**. Press the **Enter** key.

3) Recalibrate the meter using fresh, certified conductivity standards.

pH — **YSI PRO PLUS**

CALIBRATION

Two-point pH Calibration Required (Three-point pH Calibration is Optional):

1ST CALIBRATION POINT (ALWAYS START WITH 7 BUFFER):

1) Rinse probes and calibration cup with distilled water.
2) Rinse probes and calibration cup with small amount of 7 pH buffer. Discard buffer rinse.
3) Fill calibration cup with enough **fresh** 7 pH buffer to cover the pH glass bulb and temperature sensor.
4) Check temperature of pH buffer. Record value on calibration sheet.
   Note: If the temperature at which you are calibrating is significantly different from 25° C, check the buffer bottle for the corrected pH value at the corresponding temperature (the corrected pH value will be used in step 7).
5) Press **Cal** key. Highlight **ISE1 (pH)** and press **Enter**.
6) The prompt "`Ready for point 1`" will appear briefly at the bottom of the screen. Check Calibration Value. If value is correct, go to Step 7. If value is incorrect, highlight **Calibration Value** and press **Enter**. Input 7.0 (or, if applicable, the corrected pH value from step 4). Highlight **<<<Enter>>>**, and press **Enter.**
7) Actual-time readings will be displayed. When readings have stabilized, record displayed actual pH value as "Initial Meter Reading" for Buffer # 1 on calibration sheet.
8) Highlight **Accept Calibration**, and press **Enter** to calibrate.
9) "`Ready for Point 2`" will be displayed at the bottom of the screen very briefly. Record displayed pH calibration value as "Calibrated Meter Reading" for Buffer #1.

NOTE: IF YOU ACCIDENTALLY LEAVE THE PH CALIBRATION MENU BEFORE CALIBRATING YOUR 2ND POINT, YOU MUST START OVER BECAUSE THE 1ST CALIBRATION POINT WAS NOT COMPLETED. "Calibrate ISE1 (pH)" should still be displayed at the top of the screen. Remain on the same display screen as in Step 9 in order to see the actual-time temperature reading for the 2nd buffer.

2ND CALIBRATION POINT:

10) Rinse probes and calibration cup with distilled water.
11) Rinse probes and calibration cup with small amount of 2nd buffer (either 4 or 10 pH buffer). Discard buffer rinse.
12) Fill calibration cup with enough **fresh** buffer to cover the pH glass bulb and temperature sensor.
13) Actual-time readings will be displayed. When readings have stabilized, record the temperature reading.
    Note: If the temperature at which you are calibrating is significantly different from 25° C, check the buffer bottle for the corrected pH value at the corresponding temperature (the corrected pH value will be used in step 14).
14) Check Calibration Value. If value is correct, go to Step 15. If value is incorrect, highlight **Calibration Value** and press **Enter**. Input correct buffer value (or, if applicable, the corrected pH value from step 13). Highlight **<<<Enter>>>**, and press **Enter.**
15) Actual-time readings will be displayed. When readings have stabilized, record displayed actual pH value as "Initial Meter Reading" for Buffer # 2 on the calibration sheet.
16) Highlight **Accept Calibration** and press **Enter** to calibrate.
17) "`Ready for Point 3`" will be displayed briefly at the bottom of the screen. If only performing a 2-point calibration, press **Cal** Key to complete calibration process.
18) Record displayed pH value as "Calibrated Meter Reading" for Buffer #2.

If you chose to do a "`3-point calibration`", do NOT press **Cal** key in step 17 and repeat steps 10 through 17 using the 3rd buffer.

CONFIRMATION BUFFER: CONFIRMATION BUFFER STEP IS VERY CRITICAL FOR THE YSI PRO PLUS – DO NOT SKIP IT!

20) Rinse probes and calibration cup with distilled water.
21) Rinse probes and calibration cup with small amount of 7.0 pH buffer. Discard buffer rinse.
22) Fill the calibration cup with enough **fresh** buffer to cover the pH glass bulb and temperature sensor.
23) Wait 1 to 3 minutes for pH readings to stabilize.
24) Record the displayed pH value as the "Meter Reading" under "Confirmation Buffer 7.0" on the calibration sheet
25) Confirm that the "Meter Reading" value is within ± 0.1 of the buffer value (between 6.9 and 7.1).

Terminal Check (Post-Sampling Meter Check)

a. Repeat steps 20 thru 23 (for 7 buffer); record displayed value on calibration sheet. Value should be within ±0.2 of 7.0.
b. Repeat steps 20 thru 23 for Buffer #2. Record value on calibration sheet. Value should be within ±0.2 of Buffer #2.

pH — **YSI PRO PLUS**

MAINTENANCE

✱ **Never accept an out-of-range calibration**! (flagged by an error message on the meter display)

Indicators that maintenance is needed:
Difficulty calibrating pH sensor, slow response, erratic readings, clogged or black reference junction, coated glass bulb.

Probe Storage:
Do NOT allow the pH sensor to dry out! Sensors that have dried out may be permanently damaged!
Store probe in calibration/storage cup filled with 1/8" of tap water (never use distilled water to store probe).
If probe will not be used for several months, remove probe and store in pH 4 buffer. Seal the vacant port with a port plug.

Probe Lifespan:
The pH probe has a lifetime of approximately 12-24 months (in some cases, probes may last 3+ years).
When troubleshooting pH sensor problems, start by checking age of probe and replace as-needed:
On the side of each probe is the imprint "YSI 1001" followed by 2 numbers and a letter. The 2 numbers and the letter indicate the year and month in which the probe was made. For instance, 07D means the probe was made in April, 2007. (i.e. A=Jan, B=Feb, etc.).

Troubleshooting with mV readings:
1) Follow steps for pH calibration. During calibration, record pH mV values from the "Calibrated" screen for each buffer.
2) Evaluate the pH mV values:
   The span or "slope" between the pH 4 and pH 7 and between pH 7 and pH 10 should be approximately 165 to 180 mV.
   pH 7 should be 0 mV ± 50 mV. pH 4 should be 180 mV ± 50 mV. pH 10 should be -180 mV ± 50 mV.
   Example: If a probe reads +10 mV in pH 7 buffer, then the probe should also read between 175 and 190 mV in pH 4 buffer, and between -155 mV and -170mV in pH 10 buffer.
3) If the mV values fall outside the range of 160-180 mV, the probe should be replaced soon.
   Note: The probe will no longer calibrate when the span is outside of the range of 150-210 mV.

General pH Probe Cleaning:
Use deionized water and a soft lens cloth or a cotton swab to remove foreign material from the glass bulb. If good response is not restored, perform the following procedure:
1) GENTLY clean the glass bulb and white probe face by carefully rubbing a cotton swab soaked in mild dishwashing detergent. Apply little to no pressure, as the glass bulb is very thin and fragile!
2) Rinse probe thoroughly with deionized water.
3) Wipe probe with cotton swab that has been saturated with water. Rinse probe again.

Advanced pH Probe Cleaning and Restoration:
The need and frequency depend on the type of surface water being monitoring.
The probe must be removed from the sonde before advanced cleaning.
To remove more resistant deposits and biological growth, use HCl acid and bleach.
To perform an advanced cleaning, refer to the Care, Maintenance, and Storage section of the YSI Professional Plus User Manual.

Reference Junction:
The reference junction is a small tab located between the edge of the white surface of the pH probe face and the gray raised area around the pH probe face.
When new, the junction will be an off-white color.
As it ages, the junction will become darker.
A black reference junction coupled with slow response and/or erratic readings indicates a more advanced cleaning may be needed.

APPENDIX 5: Uncorrected Dissolved Oxygen Table

Corrected D.O. Tables for a specific location or DWR office are available upon request from the ESS

Sea Level *(Uncorrected D.O. Values)*

Dissolved Oxygen (D.O.) TABLE

Altitude at Sea Level = 0 feet **Barometric Pressure (BP) at Sea Level = 760 mm Hg**

Temp (°C)	0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	Temp (°C)
0	14.6	14.6	14.5	14.5	14.5	14.4	14.4	14.3	14.3	14.3	0
1	14.2	14.2	14.1	14.1	14.1	14.0	14.0	13.9	13.9	13.9	1
2	13.8	13.8	13.8	13.7	13.7	13.6	13.6	13.6	13.5	13.5	2
3	13.5	13.4	13.4	13.4	13.3	13.3	13.2	13.2	13.2	13.1	3
4	13.1	13.1	13.0	13.0	13.0	12.9	12.9	12.9	12.8	12.8	4
5	12.8	12.7	12.7	12.7	12.6	12.6	12.6	12.5	12.5	12.5	5
6	12.4	12.4	12.4	12.4	12.3	12.3	12.3	12.2	12.2	12.2	6
7	12.1	12.1	12.1	12.0	12.0	12.0	12.0	11.9	11.9	11.9	7
8	11.8	11.8	11.8	11.8	11.7	11.7	11.7	11.6	11.6	11.6	8
9	11.6	11.5	11.5	11.5	11.4	11.4	11.4	11.4	11.3	11.3	9
10	11.3	11.3	11.2	11.2	11.2	11.2	11.1	11.1	11.1	11.1	10
11	11.0	11.0	11.0	11.0	10.9	10.9	10.9	10.9	10.8	10.8	11
12	10.8	10.8	10.7	10.7	10.7	10.7	10.6	10.6	10.6	10.6	12
13	10.5	10.5	10.5	10.5	10.4	10.4	10.4	10.4	10.4	10.3	13
14	10.3	10.3	10.3	10.2	10.2	10.2	10.2	10.1	10.1	10.1	14
15	10.1	10.1	10.0	10.0	10.0	10.0	10.0	9.9	9.9	9.9	15
16	9.9	9.8	9.8	9.8	9.8	9.8	9.7	9.7	9.7	9.7	16
17	9.7	9.6	9.6	9.6	9.6	9.6	9.5	9.5	9.5	9.5	17
18	9.5	9.4	9.4	9.4	9.4	9.4	9.4	9.3	9.3	9.3	18
19	9.3	9.3	9.2	9.2	9.2	9.2	9.2	9.1	9.1	9.1	19
20	9.1	9.1	9.1	9.0	9.0	9.0	9.0	8.97	8.9	8.9	20
21	8.9	8.9	8.9	8.9	8.8	8.8	8.8	8.8	8.8	8.8	21
22	8.7	8.7	8.7	8.7	8.7	8.7	8.6	8.6	8.6	8.6	22
23	8.6	8.6	8.5	8.5	8.5	8.5	8.5	8.5	8.4	8.4	23
24	8.4	8.4	8.4	8.4	8.4	8.3	8.3	8.3	8.3	8.3	24
25	8.3	8.2	8.2	8.2	8.2	8.2	8.2	8.2	8.1	8.1	25
26	8.1	8.1	8.1	8.1	8.1	8.0	8.0	8.0	8.0	8.0	26
27	8.0	8.0	7.9	7.9	7.9	7.9	7.9	7.9	7.9	7.8	27
28	7.8	7.8	7.8	7.8	7.8	7.8	7.7	7.7	7.7	7.7	28
29	7.7	7.7	7.7	7.7	7.6	7.6	7.6	7.6	7.6	7.6	29
30	7.6	7.5	7.5	7.5	7.5	7.5	7.5	7.5	7.5	7.4	30
31	7.4	7.4	7.4	7.4	7.4	7.4	7.4	7.3	7.3	7.3	31
32	7.3	7.3	7.3	7.3	7.3	7.2	7.2	7.2	7.2	7.2	32
33	7.2	7.2	7.2	7.1	7.1	7.1	7.1	7.1	7.1	7.1	33
34	7.1	7.1	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0	34
35	6.9	6.9	6.9	6.9	6.9	6.9	6.9	6.9	6.9	6.8	35

* All D.O. values are in mg/L

Uncorrected Table **08/24/2007**

D.O. Correction Chart

Altitude (ft)	Barometric Pressure (mmHg)	Correction Factor	Altitude (ft)	Barometric Pressure (mmHg)	Correction Factor
100	757	0.996	2400	695	0.914
200	755	0.993	2500	692	0.910
300	752	0.989	2600	689	0.907
400	749	0.985	2700	686	0.903
500	746	0.981	2800	684	0.900
600	743	0.978	2900	682	0.897
700	740	0.974	3000	679	0.893
800	737	0.970	3100	676	0.890
900	735	0.967	3200	673	0.886
1000	732	0.963	3300	671	0.883
1100	729	0.959	3400	669	0.880
1200	727	0.956	3500	666	0.876
1300	724	0.952	3600	663	0.873
1400	721	0.949	3700	661	0.870
1500	718	0.945	3800	658	0.866
1600	715	0.941	3900	656	0.863
1700	713	0.938	4000	654	0.860
1800	710	0.934	4100	651	0.857
1900	708	0.931	4200	648	0.853
2000	705	0.927	4300	646	0.850
2100	702	0.924	4400	644	0.847
2200	699	0.920	4500	641	0.844
2300	697	0.917			

How to Correct D.O. Table Values:

Corrected D.O. Value = Value from Sea Level Table x Correction Factor

1) Use the temperature displayed on your meter and the "Sea Level Table" (on the back of this page) to find the *Uncorrected D.O. Value*.

2) Use your location's altitude and the "D.O. Correction Chart" (on this page) to find the corresponding *Correction Factor*.

3) Multiply the *Uncorrected D.O. Value* (from step 1) by the *Correction Factor* (from step 2) to get the *Corrected D.O. Value*.

4) The value calculated in step 3 (*Corrected D.O. Value*) and the value displayed on the meter should be within ± 0.5 mg/L of each other.

Corrected D.O. Tables for a specific location or DWR office are available upon request from the ESS QA Coordinator.

D.O. Correction Factors

08/24/2007

APPENDIX 6: SOP for Filtering in the Field

STANDARD OPERATING PROCEDURES FOR FIELD FILTERING USING THE VACUUM PUMP PROCEDURE.

Field Procedure:

1. Obtain filtering equipment including sterile 0.45 εm 47 mm diameter Millipore filters, glass fiber filters, nitrile gloves, forceps, and a supply of deionized (DI) water. An example of an appropriate filtering kit is Nalgene - filter holder with receiver 500 mL (Nalgene #300-4050).
2. After donning gloves, thoroughly rinse the field filtering equipment with deionized water on the day of sampling at the first sampling station.
3. Remove 0.45 um filter from package with clean forceps and place on the filter platform, gridded side up.
4. Inspect filter for proper placement-centered; no wrinkles, bends, cracks, holes, or gaps.
5. Reassemble apparatus.
6. Attach hand pump to outlet of bottom chamber with tubing.
7. If first sample of the day, do field blank for quality control first using DI water and following steps 8-16.
8. Pour required volume of sample water in the top chamber (example-volume for orthophosphorus and dissolved phosphorus is at least 200 mL for each).
9. Use hand pump to create vacuum.
10. Continue adding sample and pumping until required filtered volume (based on parameter) is obtained and top chamber is empty. **Note:** It may be necessary to change filters several times or use a glass fiber pre-filter (see turbid samples options below) to obtain enough filtrate.
11. Samples for all dissolved parameters can be filtered at once.
12. Before disassembling, make sure that no sample remains in the top chamber and no pressure in the bottom chamber: remove tubing, or press release on pump.
13. Disassemble apparatus.
14. Decant filtrate into sample bottles, preserve and handle as per laboratory guidance.
15. Remove filter with forceps and dispose of filter.
16. Rinse filtering apparatus with DI water. This rinse must be repeated before field filtering at any additional locations (i.e. between stations).
17. After last sample of the day is completed, do terminal field blank sample.

Turbid Samples Options:

When the filter becomes clogged:

Option 1: Change filters

1. Finish filtering any sample left in top chamber.
2. Ensure zero pressure in bottom chamber.
3. Disassemble apparatus.
4. Using forceps, remove clogged filter and replace with new filter. **Caution:** Donôt let residue on filter contact any part of the interior of the apparatus or tips of forceps.
5. Re-assemble apparatus and continue filtering.

Field Filtering SOP (Cont.)

Option 2: Pre-filter

1. The sample can be taken through a preliminary step using a filter with a larger pore size, such as a glass fiber filter.
2. This can be accomplished by placing the glass fiber filter on top of the 0.45 *u*m filter on the filter platform. A small amount of DI water can be squirted on top of the combined filters to prevent vapor lock. It may be necessary to change these filters as they get clogged to obtain enough filtrate but this procedure should minimize the number of times the filters must be changed.

Quality Control Procedures

1. The filtering apparatus and DI wash bottle should be regularly cleaned with phosphate-free detergent and completely rinsed with DI water, as is done with all other sampling equipment.
2. Initial and terminal quality control samples (blanks) of filtered deionized water must be taken for each dayõs sampling for each parameter and submitted to the laboratory.
3. Blanks must be filtered in the field: one at the beginning of the day before the first water sample is processed, and one at the end of the day after the last water sample is processed.
4. Sources of contamination include:
   -air/environment;
   -field staff;
   -sampling equipment and bottle;
   -filtration equipment (filter holder, filter, tubing);
   -DI water, and
   -chemical preservatives.
5. Station location on the lab sheet should indicate QC sample type.
6. Blanks should come back as non-detects.
7. If blanks show detectable levels of analytes:
   - results from associated samples must be flagged, and flags reported to data users.
   - perform rigorous data review to see if contamination concerns are severe enough to warrant discarding the data.
   - patterns of dirty blanks should be reviewed and a plan for contamination source identification, corrective actions, and re-evaluations should be developed.

APPENDIX 7: Flow Measurement Field Sheet

Flow Sheet

Location: _____

Station: _____

Date: _____ Time: _____

Staff: _____

	Distance (ft.)	Depth (ft.)	Velocity (fps)		Notes:
RB		------	---------		

****need a minimum of 18 points

***depths of 2.5 feet and less, the average velocity is measured at 0.6 of the depth from the water surface.

*** depths greater than 2.5 feet measure the velocity at 0.2 and 0.8 of the water depth (the average of these two velocities will later be recorded as a single value).

ATTACHMENT 2:

QUALITY ASSURANCE MANUAL FOR THE NC DWR LABORATORY SECTION

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Quality Assurance Manual
for the
North Carolina
Division of Water Quality
Laboratory Section

April 2004

Quality Assurance Manual for the North Carolina Division of Water Quality Laboratory Section

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Raleigh, NC 27607
(919) 733-3908

Asheville Region Laboratory
2090 US Highway 70
Swannanoa, NC 28778
(828) 296-4500

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Washington, NC 27889
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Prepared by
NC DWQ Central Laboratory

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Disclaimer

The mention of trade names or commercial products in this manual is for illustration purposes only and does not constitute endorsement or recommendation for use by the DWQ.

2.0 Table of Contents

3.0 Statement of Policy

It is the mission of the North Carolina Department of Environment and Natural Resources (NCDENR) to provide leadership, education and advocacy for the responsible stewardship of North Carolina's environment and natural resources. The Division of Water Quality (DWQ) Laboratory Section is committed to protecting North Carolina's environment and human health by providing the highest quality data obtainable with reasonable cost and effort and the best overall service in environmental testing. The Laboratory Section provides analytical and technical support to the divisions and programs within the Department of Environment and Natural Resources. To ensure that the results produced and reported meet the requirements of the data users and comply with state and federal regulations, a quality management system has been implemented that is clear, effective, well-communicated, and supported at all levels of the Division. The Quality Assurance Manual (QAM) details the quality assurance (QA) program in effect at the DWQ laboratories. The primary purpose of this document is to establish and maintain uniform operational and quality control procedures and to ensure data is of a known and documented quality.

A well conceived QA program provides a sound framework for the generation of laboratory data that is scientifically valid, representative and legally defensible. The validity and reliability of the data generated by the Laboratory Section are assured by adherence to rigorous quality assurance/quality control (QA/QC) protocols. The application of sound QA/QC principles, beginning with initial planning and continuing through all field and laboratory activities, including the final report, are designed to meet that goal. The fundamental elements of the Laboratory Section's QA program include Standard Operating Procedures (SOPs), quality control practices, performance testing samples, internal audits, external audits and an ethics policy.

This manual and the quality control procedures described within are not to be viewed as complete. Rather, they serve as a basic foundation on which to build a stronger, more viable Quality Assurance Management Plan (QAMP) within the Section. Other documents that may detail or affect the quality management program include the Chemical Hygiene Plan (CHP), quality guidance documents, memoranda, work instructions, standard operating procedures and periodic reports. These documents may further define or guide the implementation of quality standards within the Laboratory Section, but shall not conflict with the QAMP or diminish the effectiveness of the program. Adherence to the practices described in this manual is required of all employees. **All analysts are required to familiarize themselves with the sections of this manual that pertain to their operations and are encouraged to comment on its contents and make recommendations for more efficient procedures.**

Following is a list of documents used to develop the Laboratory Section's QAMP.

- Interim Draft EPA Requirements for Quality Management Plans, U.S. Environmental Protection Agency, EPA QA/R-2, July 1993 et seq.
- EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5 et seq.
- Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs, American Society for Quality Control, Energy and Environmental Quality Division, Environmental Issues Group, ANSI/ASQC E4-1994 (Formerly EQA-1), January 1994 et seq.
- Quality Management and Quality System Elements for Laboratories - Guidelines, American National Standard, American Society for Quality Control, ANSI/ASQC Q2-1991 et seq.
- International Standard ISO/IEC Guide 17025 - 1999 et seq.
- NELAC standards, Chapter 5, Quality Systems
- The North Carolina Administrative Code, 15A NCAC 2H .0800, governing Laboratory Certification.

3.1 Analytical Laboratory Services

The DWQ Laboratory Section is a technical support organization with the following functions:

- Provides analytical laboratory support to the Department of Environment and Natural Resources in the form of physical and chemical analyses of stream, wastewater, groundwater, soil, sediment and fish tissue samples.
- Provides consultation and assistance to Divisional personnel, state and local agencies, private laboratories and individuals in matters of analytical methodology and quality assurance.
- Operates a laboratory certification program to control the quality of state-required monitoring analysis.

The North Carolina Division of Water Quality's Laboratory Section provides chemical, physical and microbiological analyses of surface water, groundwater, sediment, fish tissue and spill samples from around the state for the Division's Water Quality and Groundwater Sections and the Underground Storage Tank Section of the Division of Waste Management. The Quality Assurance/Quality Control (QA/QC) Coordinator is responsible for establishing, implementing and coordinating a comprehensive QA/QC program for environmental sampling and analyses performed by the North Carolina Division of Water Quality Laboratory Section. The QA/QC Coordinator is dedicated to ensuring that environmental data operations are of a quality that meet or exceed requirements for informed decision making. This office is responsible for providing information, guidance and expertise in quality control and regulatory compliance issues to ensure the laboratories of the Laboratory Section adhere to standards that meet federal and state monitoring requirements allowing for appropriate decisions to be made to protect human health and the environment. Analytical results produced by the laboratories are utilized by a variety of state and federal agencies including the NC Division of Water Quality, NC Division of Solid Waste Management, NC Division of Marine Fisheries, Dept. of Health and Human Services, municipal governments, USEPA, and US Centers for Disease Control.

3.2 Ethics

All employees of the DWQ Laboratory Section are held to high professional ethical standards in the performance of their duties. All employees are required to read, understand and sign a 'Code of Ethics Statement' (see Figure 3-1) attesting to their commitment to honesty and integrity in discharging their public duties. A copy of this document is retained in the employee's Training Documentation File. Improper, unethical or illegal actions will be dealt with according to the published Administrative Directives of the State Personnel Manual (Section 7.0) which contains the policies, regulations and procedures of the Office of State Personnel that apply to employees covered by the State Personnel Act.

Unethical activities are defined as intentional falsification of records. Records may be personal credentials, resumes or educational transcripts, instrument logbooks, maintenance logbooks, raw data and data reports. *Scientific misconduct* is defined as intentionally not adhering to the prescribed method or Standard Operating Procedure. Falsifications in the environmental laboratory industry that the NC DWQ Laboratory Section will not tolerate include, but are not limited to:

- **Falsifying data** - This includes “dry labbing”, the process of making up/creating data without performing the procedure. This may also include intentionally representing another individual's work as one’s own or changing lab data results.

- **Improper peak integration** - Intentionally integrating data chromatograms so that the quality control samples meet QC criteria. This is also known as peak shaving or enhancing.

- **Improper clock setting** - Readjusting the computer clock so that it appears samples were analyzed within hold times. This is also known as time traveling.

- **Improper representation of quality control samples** - Misrepresenting analytical spikes as matrix (digested) spikes. Analyzing a blank or LCS without sending it through the preparatory procedure. Treating a QC sample differently than a client sample.

- **Improper calibration** - Manipulating the calibration or tune so that it meets QC criteria. Examples are deleting/discarding calibration points along a curve leaving less than the minimum number of calibration points required by a method or forging tuning data so that it appears to have met calibration criteria.

- **File substitution** - Substituting invalid calibration data with valid data from a different time so that the analysis appears to be successful.

- **Hiding or concealing a problem** - Concealing a known analytical or sample problem as well as concealing a known ethical problem.

Such actions are considered personal conduct violations under State disciplinary policy. Disciplinary action for ethics violations may include verbal and/or written reprimand, reassignment, or termination depending on the number of infractions observed, the severity of the infraction, or the impact it may cause to the environment and human health.

3.3 Confidentiality

All records and documents generated by the DWQ Laboratory Section, except those associated with active criminal investigations, are public records and may be subject to disclosure according to the guidelines and exceptions published in Chapter 132 of the North Carolina General Statutes.

Figure 3-1. Code of Ethics Statement Form

NC DWQ
Laboratory Section
Code of Ethics Statement

I, the undersigned, CERTIFY that:

I have an ethical and legal responsibility to produce data that is accurate and defensible. I must conduct myself at all times in an honest and ethical manner.

I have read and reviewed the most current Quality Assurance Manual and will adhere to it in the strictest manner. I continually strive to improve the quality and service of my work.

I will promptly notify my Supervisor or Branch Manager of any problem that may slow down or limit my work productivity. I will promptly and efficiently resolve the problem prior to generating reportable data.

I understand that *unethical activities* are defined as intentional falsification of records. Records may be personal credentials, resumes or educational transcripts, instrument logbooks, maintenance logbooks, raw data and data reports. *Scientific misconduct* is defined as intentionally not adhering to the prescribed method or Standard Operating Procedure. Falsifications in the environmental laboratory industry that the NC DWQ Laboratory Section will not tolerate include, but are not limited to:

- **Falsifying data** - This includes "dry labbing", the process of making up/creating data without performing the procedure. This may also include intentionally representing another individual's work as one's own or changing lab data results.
- **Improper peak integration** - Intentionally integrating data chromatograms so that the quality control samples meet QC criteria. This is also known as peak shaving or enhancing.
- **Improper clock setting** - Readjusting the computer clock so that it appears samples were analyzed within hold times. This is also known as time traveling.
- **Improper representation of quality control samples** - Misrepresenting analytical spikes as matrix (digested) spikes. Analyzing a blank or LCS without sending it through the preparatory procedure. Treating a QC sample differently than a client sample.
- **Improper calibration** - Manipulating the calibration or tune so that it meets QC criteria. Examples are deleting/discarding calibration points along a curve leaving less than the minimum number of calibration points required by a method or forging tuning data so that it appears to have met calibration criteria.
- **File substitution** - Substituting invalid calibration data with valid data from a different time so that the analysis appears to be successful.
- **Hiding or concealing a problem** - Concealing a known analytical or sample problem as well as concealing a known ethical problem.

I agree to inform my direct line supervisor of any accidental reporting of non-authentic data by myself in a timely manner and I agree to inform my direct line supervisor of any accidental or intentional reporting of non-authentic data by other employees.

I know this policy will be strictly enforced and the NC DWQ Laboratory Section will not tolerate any unethical activities or scientific misconduct. Consequences of violating this Code of Ethics may lead to repercussions ranging from a severe reprimand to immediate termination, and depending on the situation, possible criminal prosecution.

_____	_____	_____	_____/_____/_____
Employee Name	Signature	Initials	Date

4.0 Organization, Facilities and Equipment

The Laboratory Section is a section of the Division of Water Quality of the North Carolina Department of Environment and Natural Resources. The Laboratory Section is comprised of managers, chemists, technicians, and support personnel. The main laboratory (referred to as the *Central Laboratory*) is located in Raleigh, NC. Two satellite laboratories are strategically located in the eastern (Washington, NC) and western (Swannanoa, NC) regions of the state to provide assistance with time-sensitive tests. These are referred to as the *WARO Laboratory* and the *ARO Laboratory*, respectively.

The Central Laboratory is divided into two analytical branches: the Organic Chemistry Branch and the Microbiology & Inorganic Chemistry Branch. The Organic Chemistry Branch is subdivided into two analytical units: Volatile Organics and Semivolatile Organics/Pesticides. The Microbiology & Inorganic Chemistry Branch is subdivided into three sections: Bio/Chemistry Unit, Metals Unit and Regional Labs. The Bio/Chemistry Unit is further subdivided into three analytical units: Wet Chemistry, Nutrients and Microbiology. There is also a Support Branch, which provides assistance to all of the sections listed above. The Laboratory Certification Branch is responsible for certifying commercial, industrial, municipal and field laboratories engaged in wastewater analyses and monitoring for North Carolina facilities.

The Laboratory Section is headed by the Section Chief, who is responsible for both the technical and administrative direction of the Section and is committed to the Quality Assurance program described in this manual. The Section Chief is supported by the Branch Managers. A Supervisor is assigned to each analytical unit to oversee the daily operations of these units. The QA/QC Coordinator has the responsibility of establishing, implementing and coordinating all activities related to the quality assurance program. The QA/QC Coordinator manages the QA/QC program for the three laboratories, including working with lab management and staff to identify improvements to QA systems, and establishing policy for the Laboratory Section's QA program. The QA/QC Coordinator documents these objectives in the Quality Assurance Manual (QAM) which includes procedures for sample handling, method validation, statistical analyses, and data verification.

An organization chart of the Laboratory Section is provided in Figure 4-1.

4.1 Responsibilities of Key Positions

4.1.1 Section Chief (ESIV)

Directs the activities of the Laboratory Section. Responsibilities include providing direction to the various laboratory branches and units including laboratory operations, accounting, procurement, QA/QC, and customer service. General duties involve budgeting, decisions on equipment, development of policies, personnel issues, working with clients on various matters and signatory authority for all Certification actions. The Section Chief authorizes any significant changes to the Quality Assurance Manual, in writing.

4.1.2 Quality Assurance/Quality Control Coordinator (CIII)

Plans, implements and assesses the Laboratory Section QA program. Manages the laboratory's blind proficiency program. The QA/QC Coordinator manages the QA/QC program for the laboratories, including working with lab management and staff to identify improvements to QA systems, and establishing policy for the labs' QA program. The QA/QC Coordinator documents these objectives in a Quality Assurance Manual (QAM) which includes procedures for sample handling, method validation, statistical analyses, and data verification. The employee distributes controlled copies of the QAM to all affected personnel and provides training in its interpretation. The employee ensures all routinely used procedures that impact data quality are documented in standard operating procedures (SOP's) that are complete and have been reviewed and approved by both management and the staff responsible for implementing those procedures. The employee coordinates audits/reviews to assure adherence to the QAM and to identify deficiencies in the QA/QC systems. The employee subsequently makes appropriate recommendations for correction and improvement of QA/QC activities by means of written reports. The QA/QC Coordinator ensures adequate follow-through actions are implemented in response to audit/review findings. The QA/QC Coordinator also coordinates external audits and serves as the primary liaison with regulatory agencies to ensure the labs' compliance with all pertinent regulatory and accreditation requirements.

4.1.3 Organic Chemistry Branch Manager (CSIV)

Supervises the Volatile Organics and Semivolatile Organics/ Pesticides analytical units of the Central Laboratory. Responsible for technical conduct, evaluation and reporting of all analytical tasks associated with results generated on water, soil, tissue and waste samples submitted for organic analyses. Ensures that only approved procedures are documented and followed, that all data are recorded and verified and that all deviations from approved procedures are documented. Ensures compliance with quality control objectives and laboratory quality assurance in the organic subsection. Assists Unit Supervisors in correcting problems revealed by QA audits and in bringing out-of-control methods back to within established protocol. Certifies analytical reports for release to clients. Performs work performance reviews of Unit Supervisors.

4.1.4 Microbiology and Inorganic Chemistry Branch Manager (CSIV)

Supervises the Bio/Chemistry section and Metals analytical unit of the Central Laboratory and the Regional laboratories. Responsible for technical conduct, evaluation and reporting of all analytical tasks associated with results generated on water, soil, tissue and waste samples submitted for inorganic analyses including trace metal content, minerals, nutrients, and microbiological determinations. Ensures that only approved procedures are documented and followed, that all data are recorded and verified and that all deviations from approved procedures are documented. Ensures compliance with quality control objectives and laboratory quality assurance in the Microbiology and Inorganic Chemistry Branch. Assists Unit Supervisors in correcting problems revealed by QA audits and in bringing out-of-control methods back to within established protocol. Certifies analytical reports for release to clients. Performs work performance reviews of Unit Supervisors and Regional Office personnel.

4.1.5 Bio/Chemistry Unit Supervisor (CSII)

Oversees the daily operation of the Wet Chemistry, Nutrients, and Microbiology analytical units of the Bio/Chemistry Unit. Responsible for training of staff, monitoring daily work plans for routine analytical work to ensure that sample holding time requirements and turnaround commitments are met; resolving analytical and instrumental problems; maintaining protocols to meet QA/QC objectives of the laboratory; supervising analysts and technicians in their duties; ensuring subordinates are following proper laboratory safety and waste management procedures; and implementing new or modified analytical procedures and instruments. Validates all analytical reports. Provides significant amount of customer support and consulting. Performs work performance reviews of unit personnel.

4.1.6 Metals Unit Supervisor (CIII)

Oversees the daily operation of the Metals Unit. Responsible for results generated for metals analyses of water, soil, tissue and waste samples submitted to the laboratory. Ensures compliance with quality control objectives and laboratory quality assurance in the Metals Unit. Responsible for training of staff, developing daily work plan for routine analytical work to ensure that sample holding time requirements and turnaround commitments are met; resolving analytical and instrumental problems; maintaining protocols to meet QA/QC objectives of the laboratory; supervising analysts and technicians in their duties; ensuring subordinates are following proper laboratory safety and waste management procedures; and implementing new or modified analytical procedures and instruments. Validates all analytical reports. Provides significant amount of customer support and consulting. Performs work performance reviews of unit personnel.

4.1.7 Volatiles Unit Supervisor (CIII)

Oversees the daily operation of the Volatile Organic Unit. Responsible for training of staff, developing daily work plan for routine analytical work to ensure that sample holding time requirements and turnaround commitments are met; resolving analytical and instrumental problems; maintaining protocols to meet QA/QC objectives of the laboratory; supervising analysts and technicians in their duties; ensuring subordinates are following proper laboratory safety and waste management procedures; and implementing new or modified analytical procedures and instruments. Validates all analytical reports. Provides significant amount of customer support and consulting. Performs work performance reviews of unit personnel.

4.1.8 Semivolatile Organic/Pesticides Unit Supervisor (CIII)

Oversees the daily operation of the Semivolatile Organic/Pesticides Unit, which is responsible for the analysis of semivolatile organics, pesticides and acid herbicides by GC and GC/MS techniques. Responsible for training of staff, developing daily work plan for routine analytical work to ensure that sample holding time requirements and turnaround commitments are met; resolving analytical and instrumental problems; maintaining protocols to meet QA/QC objectives of the laboratory; supervising analysts and technicians in their duties; ensuring subordinates are following proper laboratory safety and waste management procedures; and implementing new or modified analytical procedures and instruments. Validates all analytical reports. Provides significant amount of customer support and consulting. Performs work performance reviews of unit personnel.

4.1.9 Chemists/Technicians

These positions involve sample preparation and routine microbiological, chemical and physical analyses of environmental samples including maintenance and troubleshooting of assigned instrumentation. They must adhere to the daily schedule provided by the Supervisor for sample priorities and utilize SOP's for assigned tasks. Perform a variety of routine analyses or preparation procedures to determine and evaluate chemical and physical properties of laboratory samples. Verify proper preservation of samples. Carry out detailed preparation and analysis steps according to published analytical methods and standard operating procedures. Report and review data, and handles routine maintenance of instrumentation. Work under direct supervision of the Unit Supervisor or Branch Manager and performs any additional tasks that are assigned. Comply with all policies established in the QA manual and Chemical Hygiene Plan. Perform routine analytical techniques and sample preparation procedures with well-defined standards and SOP's, such as organic extractions, metals digestion, or wet chemistry. May have a role in customer support and consultation.

4.1.10 Support Unit Staff

Serve as contact persons to clients at point of sample receipt. Conduct sample receiving procedures including unloading coolers, organizing samples, comparing samples to chain-of-custody documentation, taking sample temperatures, and labeling samples. Perform routine tasks such as shipping, bottle preparation, acting as liaison with the state and private courier services, and performing sample disposal. Reference materials regarding hold times, containers, and preservatives. Prepare receipt non-conformance reports and manage sample distribution. Responsibilities may also include (but are not limited to) glassware cleaning. Report to the Section Chief. Perform additional tasks as requested. Comply with all policies in the QA manual and Chemical Hygiene Plan.

4.1.11 Processing Assistant (PAIII)

Enters sample information into the computerized DWQ Sample Tracking and Reporting (DWQ STAR) system. Verifies and notifies Unit Supervisors of samples that have not been reported within target turnaround times. Generates final reports and after they have been checked for completeness, sends them to the appropriate client.

4.1.12 Processing Assistant (PAIV)

Serves as primary receptionist for the Central Chemistry lab. Checks data entry into DWQ STAR and reviews final reports for completeness. Serves as backup to Processing Assistant III position. Manages data filing and data archiving for the Laboratory Section. Receives and distributes correspondence by mail or facsimile. Provides secretarial assistance to the Laboratory Section in the form of typing forms, records, correspondence, memoranda, reports, minutes of meetings, scientific or technical material and numerical data from rough draft or corrected copy. Oversees maintenance on office equipment.

4.2 Personnel Orientation and Training

All activities performed by the Laboratory Section shall be accomplished by qualified personnel. Each individual engaged in the conduct of, or responsible for the supervision of, sample handling and analysis shall have education, training, and experience, or a combination thereof, to enable that individual to perform the assigned

functions. Each operating unit shall have job descriptions for all positions. These job descriptions shall specify the minimum qualifications for education, experience, knowledge and skills that are necessary to perform at a satisfactory level. All staff will be encouraged to perform at a level exceeding satisfactory.

4.2.1 Orientation

Each new permanent employee receives a three part orientation including 1) a human resources orientation, 2) a safety orientation and 3) a supervisory orientation. Temporary employees receive all but the human resources orientation.

4.2.1.1 Human Resources Orientation

The human resources orientation provides information on departmental policies, procedures and benefits. New employees also participate in a 6-hour course entitled *Introduction to Organizational Excellence*. This program provides information about the Agency's mission, vision and values; organizational structure; DENR's Quality Program; the expectations of public service, and provides an opportunity for employees to learn how their work contributes to the Agency's mission.

4.2.1.2 Safety Orientation

Each new employee will take part in a two-tiered safety orientation process that will include a Division orientation with the Division Safety Officer and a Laboratory Section orientation with the employee's Supervisor. The Division safety orientation will include Hazard Communication and Chemical Hygiene Plan (CHP) review consistent with the requirements of OSHA's Hazard Communication Standard (29 CFR 1910.1200) and the Occupational Exposure to Hazardous Chemicals in Laboratories Standard (29 CFR 1910.1450). This process is documented on the *CHP Orientation Training* form (see Figure 4-2) and filed in the employee's Training Documentation File and the Division Training files.

The Laboratory Section safety orientation is an in-depth examination of the Laboratory Section's Chemical Hygiene Plan. On employment with the Laboratory Section, new personnel will receive a copy of the Chemical Hygiene Plan. The employee is required to sign a statement (see *Certification of Unit Training* form in Figure 4-3) indicating that orientation information was made available, that they have viewed the CHP, and understand the information contained in that document. The Unit Supervisor will allow adequate time, before beginning work, to read the document and clarify any areas that are not understood. This general safety orientation will, at a minimum, include:

- Use of chemicals and equipment in the laboratory, the hazards associated with those chemicals and equipment, and appropriate chemical waste disposal procedures.
- Accident/Incident prevention and reporting procedures.
- Laboratory fire safety and evacuation plans.
- A tour of the Laboratory facility.
- Personal protective equipment.

4.2.1.3 Supervisory Orientation

During the supervisory orientation, the new employee's Branch Manager or Unit Supervisor provides the employee with a basic understanding of the role of the laboratory within the Division of Water Quality and the basic elements of that individual's position within the laboratory. Personnel issues such as timesheets, workplans and the application process are reviewed. The employee is required to sign a statement (see *Employee Orientation Checklist* in Figure 4-4) indicating that the checked items were discussed and that the employee understands and agrees to abide by those policies and procedures.

Orientations for new employees should be scheduled within the first two weeks of employment, where possible, to allow new employees time to select their benefits and become acquainted with administrative and safety polices prior to beginning analytical duties.

4.2.2 Training

4.2.2.1 Safety and Chemical Hygiene Training

Employees will be apprised of the hazards present in the workplace upon initial assignment to the analytical unit or whenever new chemicals or processes are introduced into the work area. Unit Supervisors will be responsible for unit-specific chemical hygiene training for new employees. All unit safety training is documented on a *New Employee Safety Orientation and Training* form (see Figure 4-5). A copy is kept in the employee's Training Documentation File and a copy forwarded to the Division Safety Officer for inclusion in the Division Training files.

At a minimum, employees are to be trained in the following areas:

- The contents of the laboratory Chemical Hygiene Plan (CHP) and how it applies to the analytical unit to which the employee is assigned.
- The location and general contents of the unit Material Safety Data Sheet (MSDS) file. This training can be handled on a hazard class basis for normal chemicals; however, particularly hazardous chemicals must be covered in detail to ensure employees are aware of the chemical's hazardous properties.
- The current Permissible Exposure Limits (PELs) for exposure to chemicals in the analytical unit.
- The detection of leaks or releases of chemicals in the unit and specific cleanup procedures to be used.
- The personal protective equipment (PPE) required to be used in the analytical unit.
- Proper disposal protocol for chemical and sample waste.

Additional safety training courses will be made available from time to time. These courses may be mandatory or optional, depending on the topic. Employees are required to attend all mandatory training and are encouraged to take part in any optional training. Optional training may include such training as First Aid or CPR training. Mandatory and optional training will also be documented and filed in the employee's Training Documentation File.

Any time substantial changes are made to the CHP, all Laboratory Section employees will receive update training in the changes made to the plan and the process will be documented.

4.2.2.2 Analytical Training

The analytical training of a new employee concentrates on his/her scientific background and work experience to provide the employee with a level of competence so that the individual will be able to function within the defined responsibilities of his/her position as soon as possible. Training is a process used to assist laboratory personnel in their professional development. Training is usually conducted "on-the-job", teaming a qualified analyst with one in training.

Unit Supervisors shall be responsible for providing documentation of training and proficiency for each employee under their supervision. The employee's Training Documentation File indicates what procedures (SOPs) a chemist/technician is capable of performing either independently or only with supervision. The file shall include, at a minimum, the following:

- Job description
- Performance Management Work Plan

- Resume
- Code of Ethics Statement form
- Orientation forms
- Certificates of coursework completion
- Training forms and associated IDOCs and MDLs
- Performance testing results
- Audit reports and corrective action responses
- Emergency contact information

The Unit Supervisor is responsible for keeping a Training Documentation File for each person under his or her supervision that is up-to-date and current.

New laboratory personnel are trained in basic lab techniques, safety and chemical hygiene, chemistry theory of the test procedures employed, quality control procedures, the DWQ STAR LIMS, record keeping and the operating principles and regulations governing the methods employed by the Laboratory Section. A designated chemist/technician and/or the Unit Supervisor closely supervise every new employee until he/she exhibits proficiency in accepted laboratory techniques. This process includes reading specific SOP's and other associated references. Once a chemist/technician demonstrates a technological aptitude within the framework of the Quality Assurance program, he/she will perform an Initial Demonstration of Capability (IDOC) study and a Method Detection Limit study (MDL), if applicable. This training process is documented (see SOP/Method Training Summary form - Figure 4-6, IDOC form - Figure 4-7, and MDL form - Figure 4-8) for each chemist and each method and is retained in the employee's Training Documentation File. Upon completion of analytical or QA/QC training, the Supervisor or Branch Manager should certify that the person is qualified to independently perform the procedures.

Additional training techniques utilized may include:

- Lectures
- Programmed learning
- Conferences and seminars
- Short courses
- Specialized training by instrument manufacturers
- Participation in check-sample or proficiency sample programs

All laboratory personnel are required to review and update (as necessary) all Standard Operating Procedures (SOPs) annually that pertain to the work they perform within the laboratory. Any updates to SOPs must have the approval of the Supervisor, Branch Manager and QA Coordinator and must conform to the policies of the laboratory. It is the responsibility of the Supervisor to ensure that documentation demonstrating that their employees have read, understand and are using the latest version of SOPs is current and on file. The latest official versions of SOPs are only located on the Laboratory Section Intranet site (http://www.esb.enr.state.nc.us/lab/qa/sop.htm) and are accessible to all analysts and technicians throughout the Laboratory Section.

As an initial and continuing demonstration of proficiency, laboratory analysts are required to successfully analyze annually (at least once per calendar year) either 1) a blind sample, 2) a blind PT sample, 3) at least four consecutive laboratory quality control samples, 4) an authentic sample that has been analyzed by another trained analyst or 5) another acceptable demonstration of capability. Results of initial and continuing proficiency are maintained by laboratory supervisors.

Employees are encouraged to participate in advanced training courses, seminars, and professional organizations and meetings as opportunities and funding become available. Additionally, meetings may be held to discuss procedures, work schedules and problems requiring immediate attention.

At the discretion of the analyst's Supervisor or Branch Manager, an analyst may demonstrate proficiency in a test method without going through the formal training process. A *Statement of Capability* form (Figure 4-9) may be used to document the process of "grandfathering" analysts currently performing a procedure or method of analysis. This decision will be based on the analyst's experience, participation in training workshops, acceptable PT results, or an IDOC study. The completed form will be maintained in the analyst's Training Documentation File.

4.3 Facilities

The Central Laboratory building was completed and occupied in 1991. The single-story facility includes a full service analytical laboratory operation with all supporting equipment and space. The total facility consists of approximately 18,000 square feet. This includes 3 organic laboratories, 4 inorganic laboratories, a shipping/receiving area, storage areas and office space for staff. Operation and maintenance of the facility is the responsibility of the Facility Management Division of the Department of Administration. The facility is equipped with centralized water purification and HVAC systems. A floor plan of the Central Laboratory is presented in Figure 4-10.

The Asheville Region Laboratory is housed in the Asheville Regional Office in Swannanoa, NC. The total laboratory area consists of approximately 1007 square feet with approximately 61 linear feet of bench space. This includes a main laboratory, a bacteria lab, a small storage area and office space. A floor plan of the ARO Laboratory is presented in Figure 4-11.

The Washington Region Laboratory is housed in the Washington Regional Office in Washington, NC. The total laboratory area consists of approximately 1225 square feet with approximately 79 linear feet of bench space. This consists of a main laboratory and office space. A floorplan of the WARO Laboratory is presented in Figure 4-12.

4.3.1 Environment

Laboratory accommodations, test areas, energy sources, lighting, heating and ventilation must be adequate to facilitate proper performance of tests. The environment in which these activities are undertaken shall not invalidate the results or adversely affect the required accuracy of measurement. The laboratory shall provide for the effective monitoring, control and recording of environmental conditions as appropriate. Such environmental conditions may include biological sterility, humidity, and temperature. In instances where monitoring or control of any of the above-mentioned items is specified in a test method or by regulation, the laboratory shall meet and document adherence to those laboratory facility requirements.

4.3.2 Work Areas

There shall be effective separation between neighboring areas when the activities therein are incompatible (e.g., volatile organic chemicals handling and analytical areas). Access to and use of all areas affecting the quality of these activities shall be defined and controlled. Adequate measures will be taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality or staff safety.

4.3.3 Building Security

Persons not in the employ of the Laboratory Section are considered to be visitors to each site. Each visitor to the laboratory must sign in and out in a visitor's logbook and must be escorted by staff while in the laboratory. The building is always locked and keys are distributed to all permanent employees. The main

entrance and the Receiving Room (G-098) doors are equipped with an electric lock that can be released by lab personnel. Under special circumstances, sample storage coolers may be locked as well and assigned custodians will control access to each.

The regional laboratories shall store Chain-of-Custody samples in secure or locked areas.

4.4 Equipment

4.4.1 Inventory

The laboratories are equipped with a diversity of analytical equipment including gas chromatographs, gas chromatograph/mass spectrometers, atomic absorption spectrometers, inductively coupled plasma-atomic emission spectrometers, ion chromatograph, flow injection analyzers, fluorometer, UV-VIS spectrophotometers and ancillary analytical equipment and software essential to a quality environmental laboratory. The equipment and software used for testing, calibration and sampling shall be capable of achieving the accuracy required and shall comply with specifications relevant to the environmental tests and/or calibrations concerned. Instrument serial numbers or assigned ID numbers are recorded on all appropriate laboratory data.

Before being placed into service, equipment shall be calibrated or checked to establish that it meets the laboratory's specification requirements and complies with the relevant standard specifications. Similar restrictions apply to devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include, but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices (including thermometers and thermistors), thermal sample preparation devices and volumetric dispensing devices (e.g., Eppendorf® or automatic dilutor/dispensing devices) if quantitative results are dependent on their accuracy, as in standard preparation and dispensing. The major equipment housed in each laboratory is detailed in Tables 4-1: Central Laboratory, 4-2: Asheville Regional Laboratory and 4-3: Washington Regional Laboratory.

4.4.2 Maintenance/Service

Proper maintenance of laboratory instrumentation is a key ingredient to both the longevity of the useful life of the instrument and providing reliable analyses. Maintenance and service requires an alert analytical staff that recognizes the need for equipment maintenance coupled with support services provided either by in-house staff or by vendor technicians.

4.4.2.1 All staff members have the responsibility for insuring that primary maintenance is carried out on instrumentation. The primary elements of the equipment maintenance program include:

- All major equipment receives a daily check for such things as cooling fan operation, pump operation, indicator readings, mechanical checks, clean air filters, clean tubing, clean cells, etc.
- Routine preventive maintenance on all major equipment is performed as needed;
- Records are kept in maintenance logs for all repairs;
- Instrument utilization records are maintained in the form of analysis logs or instrument run logs;
- A conservative inventory of critical spare parts is maintained for high-use instrumentation;
- Vendor-produced operation and maintenance manuals (where available) are maintained for all laboratory instrumentation.

4.4.2.2 Daily maintenance responsibilities are generally delegated to the chemists/technicians. This measure improves overall lab productivity by minimizing instrument downtime. Other benefits include job knowledge enhancement, maintenance cost reduction and less frequent out-of-control situations. In a situation where the analyst is unable to rectify a problem with the instrument, the Supervisor or Branch Manager steps in to help prior to calling the manufacturer's service representative.

Some instruments are under service contract with the manufacturer and in most cases include preventative maintenance checks by their service technicians. Most service contracts are written with 48-72 hour response times to service calls. All maintenance is documented in the maintenance logbooks to be used as a source of information in solving future instrument problems.

Many consumable parts are kept in stock. Examples may include, pump tubing for FIA systems and spare columns for GC techniques. In many cases, vendors are able to provide for overnight shipment of parts that do not require manufacturer's installation.

4.4.3 Equipment Redundancy

Where feasible, redundant equipment and instruments are maintained. In case one instrument goes down, another instrument can be used (e.g., 2 gas chromatographs or a backup dissolved oxygen meter) to meet hold times and/or client due dates. In some cases, samples may be routed to one of the other three labs of the Laboratory Section if they have the capability and if the samples will meet the published hold times.

Figure 4-1. North Carolina Division of Water Quality Laboratory Section Organization Chart

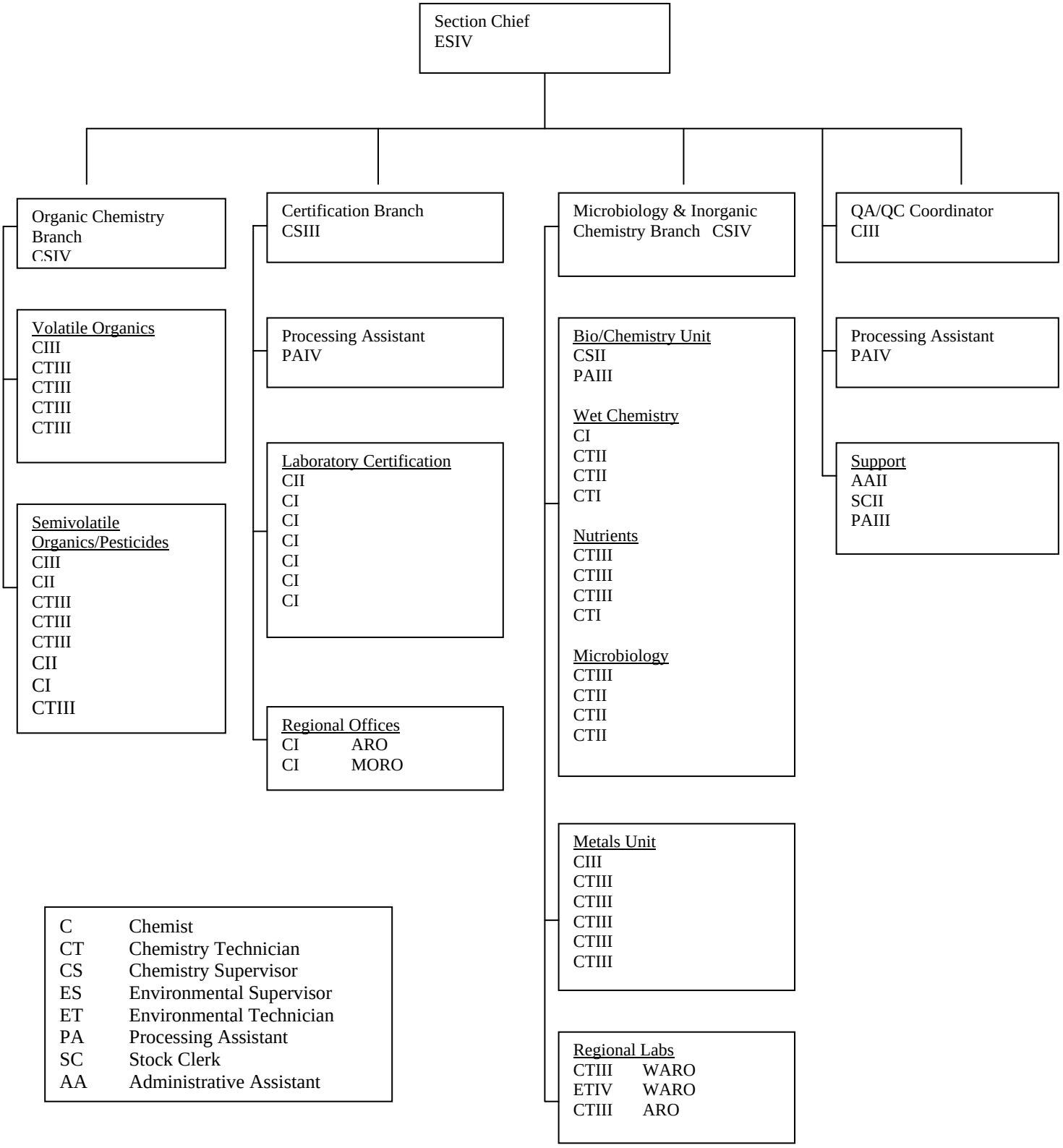


Figure 4-2. CHP Orientation Training Form

CHP Orientation Training

Date of Training:	
Name of Employee:	
Trainers (and initials):	Steve Kaasa

☐ **Hazard Communication Review (29 CFR 1910.1200)**
Hazard Classes of Chemicals used in the Laboraotry
Material Safety Data Sheets
Labeling
Storage and Handling Chemicals

☐ **CHP Review (29 CFR 1910.1450)**
Emergency Actions and Notification
Fire Prevention Guidelines
Housekeeping Rules, clothing and Personal Items

☐ **Personal Protective Equipment**

☐ **Evacuation Routes**

☐ **General Laboratory Hazards**
Recognizing work area hazards
Electrical Hazards
Compressed Gases
Vacuum
Radioactive Hazards
Noise Exposure
Fume Hood Use

☐ **Chemicals Used in the Laboratory**
Extremely Hazardous and Toxic Materials
Transporting Chemicals
Chemical Waste Disposal
Biological Waste Disposal

Figure 4-3. Certification of Unit Training Form

Certification of Unit Training

By my signature below, I acknowledge that I

was instructed by the below named individual on the hazards

present in _____ and the proper
location of areas (e.g., room number)

laboratory procedures to follow when working there as outlined

in the Chemical Hygiene Plan and any other operational plans for my work area.

I understand these hazards and accept them as a necessary part of my work.

I will follow the proper procedures in my work in this area at all times.

_____ _____
Employee Signature Date

_____ _____
Supervisor Signature Date

Figure 4-4. Employee Orientation Checklist

Employee Orientation Checklist

During the supervisory orientation, the employee is provided with a basic understanding of the elements of that individual's position within the laboratory, and is familiarized with the work area and general policies and procedures.

- ☐ Job description (review within 30 days of hire)
- ☐ Performance Management Work Plan
- ☐ Work Schedule
- ☐ Rules regarding lunch and break period
- ☐ Timesheets (for a detailed explanation, consult Kimberly Moses at 733-7015 ext. 212)
- ☐ Absences/Tardiness/Leave/Compensatory Time/Adverse Weather
- ☐ Telephone Operation
- ☐ Use of State Property (e.g., internet, telephone, computer, fax, copier, vehicle use, etc.)
- ☐ Mail Procedures
- ☐ Location and storage of personal belongings
- ☐ Travel Procedures (for detailed explanation, consult Tony Bass, DWQ Budget at 733-7015 ext. 220)
- ☐ Organization Chart
- ☐ Access to Policy Manuals (internet links are provided in New Employee Notebook)
- ☐ Impact of job/work on the Department, the public and co-workers
- ☐ Work Area
- ☐ Location of restrooms, break rooms and smoking areas
- ☐ Pay day/direct deposit
- ☐ Employee Health programs
- ☐ Drug and alcohol abuse policy and programs
- ☐ Employee suggestions/open door policy
- ☐ Parking/building security
- ☐ Tour of the laboratory and facilities/introduction to co-workers and key personnel
- ☐ Obtain key to building/work area
- ☐ Obtain personal protective equipment (e.g., labcoat, safety glasses/goggles/prescription, etc.)
- ☐ Schedule baseline physical (contact Mary Wiggins at Duke 8-919-286-5569, contract #EW03028)

By signing, the employee verifies that he/she has received, read and understands the information checked above.

Supervisor signature _____ Date _____

Employee signature _____ Date _____

Figure 4-5. New Employee Safety Orientation and Training Form

New Employee Safety Orientation and Training

Date of Orientation:	
Name of Employee:	
Orientation Instructor:	

Safety Plan Review

- ☐ Emergency Actions and Notification
- ☐ Reporting Laboratory Accidents
- ☐ Fire Prevention Guidelines
- ☐ Housekeeping Rules, Clothing and Personal Items
- ☐ Recognizing work area hazards

Personal Protective Equipment

- ☐ Location
- ☐ Instructions for Use (including cleaning, repairing and replacing)

Evacuation Plan

- ☐ When/where to Evacuate
- ☐ Alarm System (including tests and fire drills)

General Laboratory Hazards

- ☐ Hazardous Equipment
- ☐ Non-hazardous Equipment
- ☐ Electrical Hazards
- ☐ Compressed Gases
- ☐ Vacuums
- ☐ Noise Exposure
- ☐ Fume Hood Use
  - ☐ When one should use the Fume Hood
  - ☐ Operation of the Fume Hood

Chemicals used in the Laboratory

- ☐ Inventory of Chemicals used in the Laboratory
- ☐ Hazard Classes of Chemicals used in the Laboratory
  - ☐ Material Safety Data Sheets
  - ☐ Storage
  - ☐ Extremely Hazardous and Toxic Materials
- ☐ Transporting Chemicals
- ☐ Hazardous Waste Disposal
- ☐ _____
- ☐ _____

By signing, the employee verifies that he/she has received, read and understands the information checked above.

_____ _____ _____ _____

Signature of Employee Date Signature of Instructor Date

Figure 4-6. SOP/Method Training Summary Form

SOP/METHOD TRAINING SUMMARY

Analytical unit/region		Instrument(s)	
Trainee		Technique	
SOP(s) reviewed		Date of completion	
Reference method(s)		Matrix	
Parameter			

I. METHOD/PARAMETER

- ☐ Reference Method/SOP
- ☐ Basic Method/Instrument Theory
- ☐ Safety Precautions
- ☐ Waste Handling
- ☐ Regulatory Standards
- ☐ Routine Maintenance
- ☐ Interferences
- ☐ Extraction/Preparation

II. QUALITY CONTROL

- ☐ Calibration curve, Initial Calibration Verification, and Continuing Calibration Verification
- ☐ Precision/Accuracy
- ☐ MDL study
- ☐ Review of COC procedures
- ☐ Documentation (sequences, maintenance, logbooks, benchsheets, observations, modifications, standards/reagent preparation, etc.)
- ☐ QC Requirements (MS/MSD, QCS, blanks, duplicates, surrogates, internal standards, interference checks, etc.)
- ☐ Miscellaneous QC (retention time window studies, IDL, etc.)
- ☐ Non-Conformance and Corrective Action
- ☐ Documentation (SCUR/SAR)

III. DATA HANDLING AND REPORTING

- ☐ Review Equations and Calculations (concentrations, dry/wet weight ratios, etc.)
- ☐ Data Entry (DWQ STAR LIMS)
- ☐ Significant Figures
- ☐ Reporting Dilutions
- ☐ Reporting Data with Qualifier Codes

IV. GENERAL TRAINING

Describe what was discussed. General training topics might include sample receiving, aseptic technique, shipping, supporting equipment use (e.g., pH meter), training course attendance, etc. Attach additional pages if necessary.

V. RESULTS OF START-UP QC

IDOC Results	Acceptable:	Y / N / NA	Attach a copy of the IDOC study summary.
MDL Study (when applicable)	Completed:	Y / N / NA	Attach a copy of the MDL study summary.

VI. SIGNATURE AUTHORIZATION

By signing, the trainee verifies that he/she has received, read and understands the SOP(s), reference method and any other related materials required to effectively perform the subject analysis or general procedure. Signature of the supervisor, branch manager and QA/QC Coordinator verifies that the analyst has met the minimum requirements of demonstration of capability to perform the subject analysis or procedure. If additional training is required, this form should not be signed.

Trainee Signature:		Date:	
Supervisor Signature:		Date:	
Branch Manager Signature		Date:	
QA/QC Coordinator Signature:		Date:	

Figure 4-7. IDOC Summary Form

Initial Demonstration of Capability (IDOC)
Certification Statement

Laboratory Name:		**Analytical Method:**	
Analyst Name:		**SOP#:**	
Date:		**Instrument:**	
Sample Prep. Method:		**Column:**	
SOP#:		**Detector:**	
Matrix:		**Cleanup/Modification:**	

Analyte	Spike conc.	Units	1	2	3	4	Mean Recovery %	Mean X	Acceptance Range[1]	Standard Deviation s	Acceptance Criteria[1]	% RSD[2]	P/F

P = pass F = fail

[1]Cite reference**. _____.**

[2]% RSD = % relative standard deviation = (s/X) 100

Comments:

_____

We, the undersigned, CERTIFY that:
The analysts identified above, using the cited test method(s), which is in use at this facility for the analyses of samples have met the Initial Demonstration of Capability. The test method(s) was performed by the analyst(s) identified on this certification. A copy of the test method(s) and the laboratory-specific SOPs are available for all personnel on-site. The data associated with the demonstration of capability are true, accurate, complete and self-explanatory. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized assessors.

_____ _____ _____
Chemist's/Technician's Name Signature

_____ _____ _____
Branch Manager's Name Signature Date

_____ _____ _____
Supervisor's Name

_____ _____ _____
Quality Assurance Officer's Name Signature
Date

Figure 4-8. MDL Summary Form

Method Detection Limit (MDL) Study

Laboratory Name:		**Analytical Method:**	
Analyst(s) Name(s):		**SOP#:**	
Date:		**Instrument:**	
Sample Prep. Method:		**Column:**	
SOP#:		**Detector:**	
Matrix:		**Cleanup/Modification:**	

Analyte	Spike conc.	Units	1	2	3	4	5	6	7	Mean Recovery %	Average Recovery X	Standard Deviations	MDL	PQL

MDL = t (n-1, 1-a = 0.99) (s)
t = Student's t values appropriate for 99% confidence level. Table of Students' t values can be found in 40 CFR Part 136, Appendix C.
PQL = 3 to 5 times the calculated MDL.

Comments: _____

_____ _____ _____
Chemist's/Technician's Name

_____ _____ _____
Branch Manager's Name Signature Signature

_____ _____ _____
Supervisor's Name Signature

_____ _____ _____
Quality Assurance Officer's Name Signature

Date Date

Figure 4-9. Statement of Capability Form

STATEMENT OF CAPABILITY

(used to "grandfather" in current analysts)

_____ has been performing the following analyses:
(analyst's name)

Method / Parameter	SOP #	Start Date	End or Current Date

The analyst is deemed proficient in the performance of the analyses listed above because (check all that apply):

- ❑ Analyst's experience. Comment: _____

_____

_____

- ❑ The analyst has demonstrated the use and understanding of the SOP and referenced methods.
- ❑ Acceptable results on past PT samples. Attach examples.
- ❑ Acceptable IDOC on four QCS replicates. Attach IDOC study summary.

APPROVED BY:

Supervisor: _____ Date: _____

Branch Manager: _____ Date: _____

QA/QC Coordinator: _____ Date: _____

Figure 4-10. Central Laboratory Floorplan.

Central Laboratory
4405 Reedy Creek Road
Raleigh, NC 27607

(not to scale)

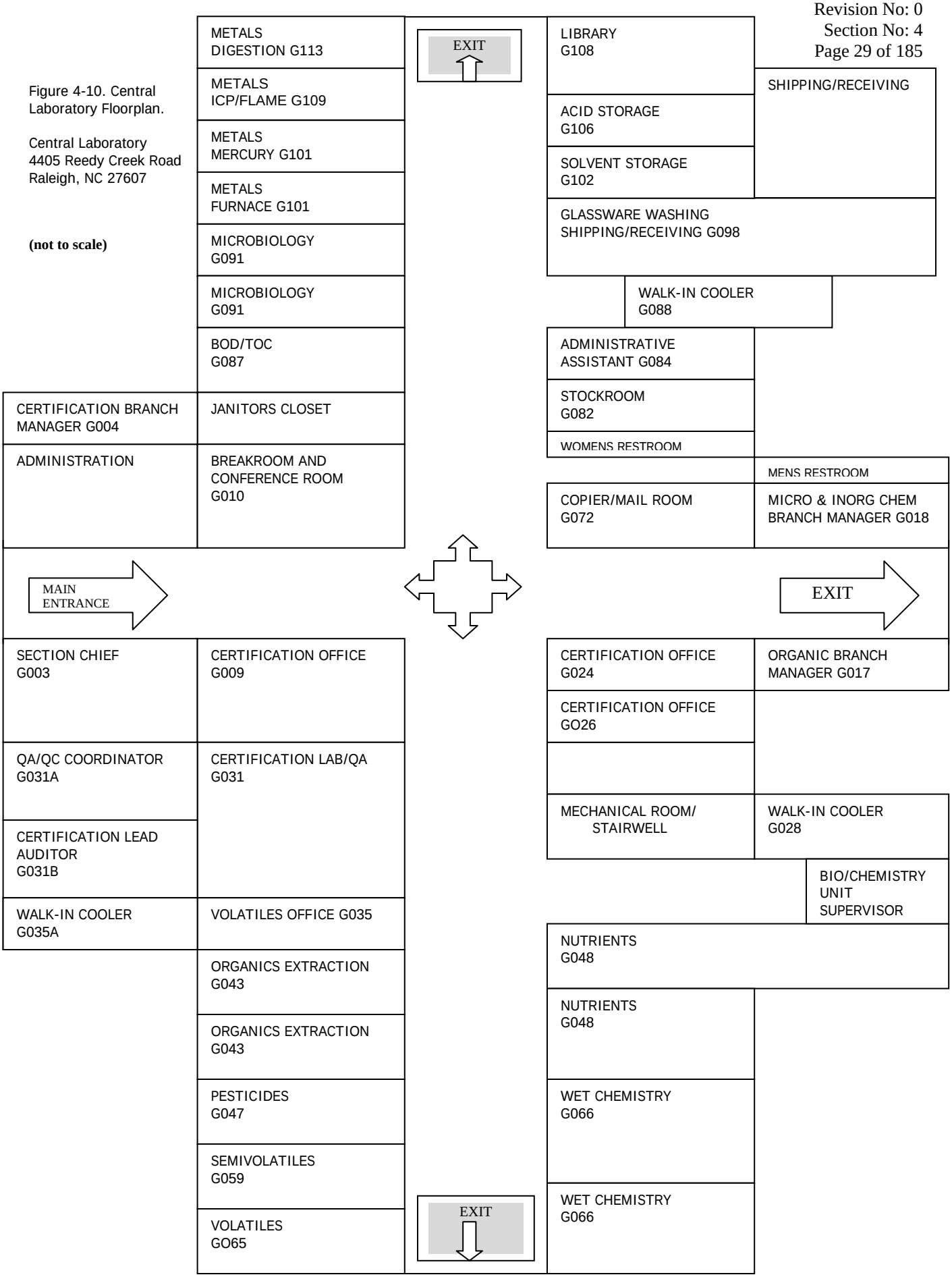


Figure 4-11. Asheville Regional Laboratory Floorplan.

Asheville Laboratory
2090 US Highway 70
Swannanoa, NC 28778

(not to scale)

Lab area is shaded

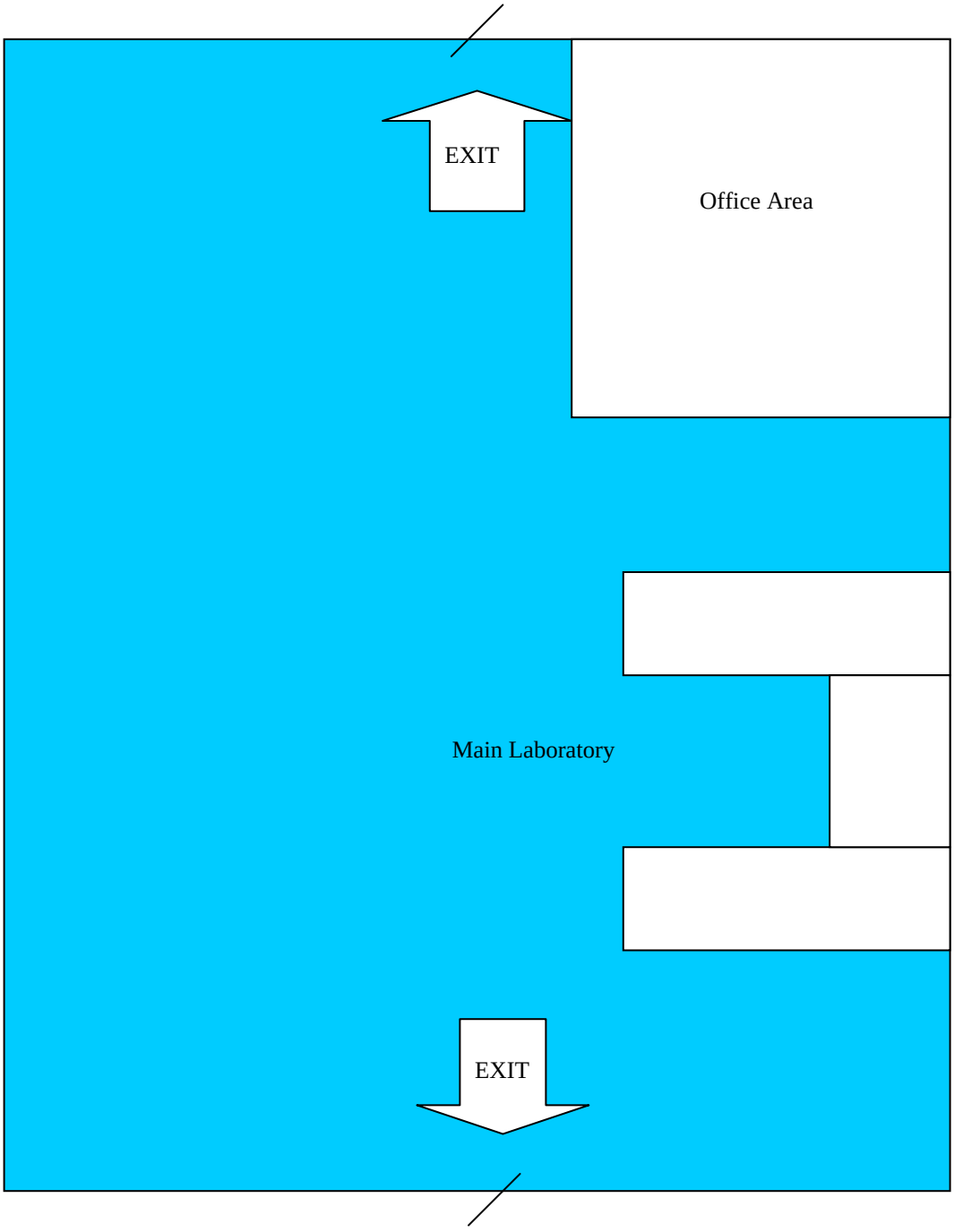


Figure 4-12. Washington Regional Laboratory Floorplan.

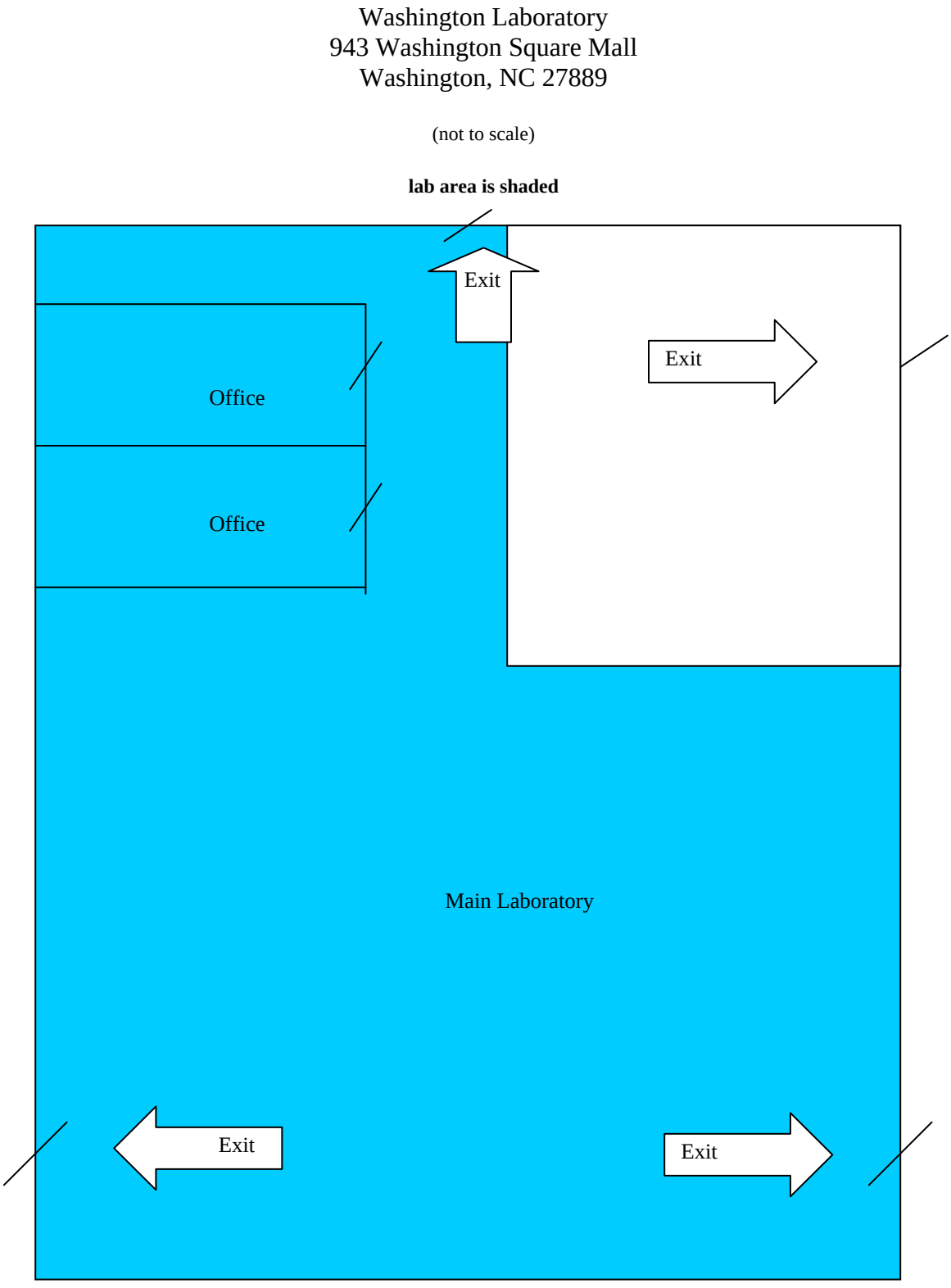


Table 4-1. Central Laboratory Major Equipment List

VOA Equipment	Manufacturer	Model	Serial/ID No.	Year placed in service
GC- ELCD/PID (A)	Hewlett-Packard	5890	2750A16571	1988
Purge and Trap Concentrator	OI Analytical	4460A REV-B	3572-8-286	1988
P&T Autosampler	OI Analytical	MPM16	1988-8-02	1988
GC-ELCD/PID (B)	Hewlett-Packard	5890	2843A19424	1988
Purge and Trap Concentrator	OI Analytical	4460A REV-B	3702-8-294	1988
P&T Autosampler	OI Analytical	MPM16	3793-8-010	1988
GC- PID/FID (C)	Tremetrics	9000	930235	1993
Purge and Trap Concentrator	Tekmar	LSC 2000	92226001	1993
P&T Autosampler	Tekmar	ALS 2016	93041006	1993
GC- ELCD/PID (D)	Hewlett-Packard	5890 Series II Plus	3336A58501	1995
Purge and Trap Concentrator	OI Analytical	4560	3438460079	1995
P&T Autosampler	OI Analytical	MPM16	11203-1-287	1995
GC- ELCD/PID (E)	Finnigan	9001	GC100294	1998
Purge and Trap Concentrator	Tekmar	3000	95132006	1995
P&T Autosampler	Tekmar	ALS 2016	95131001	1995
GC/MS	Agilent	HP5973/6890	US93123009	1999
Purge and Trap Concentrator	Tekmar	3100	00285007	2000
P&T Autosampler	Tekmar	ALS 2016	00304001	2000
XA Computer	Hewlett-Packard	D6720T	US94554773	1999
HP Chemstation software	Hewlett-Packard	G1701BA.02	BN2138C084	1999
Dell Optiplex Computer	Dell	GX240	HJ7HJ11	2002
Total Chrom Software	Perkin-Elmer	TCWS6.2 N515-0511	0502-UP WS0003	2002
GC/Computer Interface	PE Nelson	970A	3290270061	1992
GC/Computer Interface	PE Nelson	970A	3051270236	1992
GC/Computer Linkbox	PE Nelson	610	3047110062	1992
8 Port Terminal Server	Lantronix	ETS8P	8313312	2002
Top Loading Balance	Denver Inst. Co.	XL-500	44656	
Refrigerator	Kenmore			
Refrigerator	Kenmore	5		
SVOA Equipment	**Manufacturer**	**Model**	**Serial/ID No.**	**Year placed in service**
GC/FID	Hewlett Packard	5890B	2908A21482	1989
GC/FID	Shimadzu	GC17A	C10693101072	1995
GC/MS GC	Agilent	6890	US00038661	2000
GC/MS MS	Agilent	5973N	US03960480	2000
GC/MS GC	Agilent	6890	US00034057	2000
GC/MS MS	Agilent	5973N	US03360393	2000
Analytical Balance	Denver	XE-100A	120001249	1993
Muffle Furnace	Lindberg	51894	899200	1990
Wrist Shaker	Labline	3589-1	11940022	1995
Sonifier	Branson	450	88803021174A	2003
Sonicator	Heat Systems	W-385	G-8179	1987
Freezer	Baxter	U2005XA15	5N6229690	
Freezer	VWR			2003
Freezer	Baxter	U2005XA15	5N6229690	
Freezer	Whirlpool	EEV-203FW0	593413628	

SVOA Equipment, *cont'd.*	**Manufacturer**	**Model**	**Serial/ID No.**	**Year placed in service**
Top Loading Balance	Denver Instrument Co.	100A	12 001249	1993
Oven	Lindberg		899200	1990
Pesticides Equipment	**Manufacturer**	**Model**	**Serial/ID No.**	**Year placed in service**
GC/FPD/ECD	Hewlett Packard	5890	2843A20188	1989
GC/NPD/NPD	Tremetrics	9000	920/85	1992
GC/ECD/ECD	Hewlett Packard	5890	2843A20187	1989
GC/ECD/ECD	Tracor	540	871168	1988
Tecator Soxtec	Foss	2050	307880005	2001
Automated Sample Processing System (GPC)	O-I Analytical	AP1000	9254AV	2000
Freezer	VWR	U2020FA14	W08G-350948-WG	1998
Oven	Lindberg/Blue M	G01330A	P27F-295803-PF	1996
Freezer	Labline	FV21M2WLFA	1015392492	1982
GC/FPD/ECD	Tracor	540	861423	1986
GC/ECD/ECD	Tracor	540	84057	1984
Metals Equipment	**Manufacturer**	**Model**	**Serial/ID No.**	**Year placed in service**
ICP/MS	PerkinElmer	6100	949907	2000
ICP	PerkinElmer	3000XL	069N4121902	1995
AutoSampler	PerkinElmer	AS91	3130	1995
Furnace	PerkinElmer	AAnalyst 800	800S2060103	2002
FIMS	PerkinElmer	FIAS 400	4324	1996
AutoSampler	PerkinElmer	AS90	1305	1991
HotBlock digestion system	Environmental Express	SC100	145CECD310	1999
HotBlock digestion system	Environmental Express	SC100	145CEC0293	1999
Water cooler/recirculator	ISOTemp	1013S	198107070	1998
Argon manifold/ 2 tank hookup	Western	LCHP-7-2	23708	2000
Analytical balance	Mettler	AG204	1121490982	2003
Flow injection/ICPMS	PerkinElmer	FIAS 400	5340	2000
HEPA filtered hood	Environmental Express	SC801	NA	2003
HEPA filtered hood	Environmental Express	SC801	NA	2003
HotBlock digestion system	Environmental Express	SC154	2002CEC1219	2003
Water bath	Blue M	MW-1140A	14957	1986
Water bath	Blue M	MW-1140A	N5-1554	1987
Top loading balance	Fisher	2400-DR	2431	1984
Top loading balance	Denver	XL1800	53725	1994
Hot plate	Barbstead/ Thermodyne	RC2240	41193123888	1994
Hot plate	Barbstead/ Thermodyne	RC2240	411940896575	1995
Water Deionizer	Barbstead/ Thermodyne	D8961	896010867669	2001
Freezer	Kenmore	9203381	VSA1684516	
Nutrients Equipment	**Manufacturer**	**Model**	**Serial/ID No.**	**Year placed in service**
FIA system with Ammonia	Lachat	QuickChem 8000 series	160878	2001

Nitrogen /Nitrate + Nitrite Nitrogen Manifold				
Nutrients Equipment, *cont'd.*	**Manufacturer**	**Model**	**Serial/ID No.**	**Year placed in service**
FIA system with TKN Manifold	Lachat	QuickChem 8000 series	160952	2001
FIA system with TP Manifold	Lachat	QuickChem 8000 series	161030	2001
Ion Chromatograph	Lachat	QuickChem 8000 series	160959	2002
Block Digestor	Lachat	BD-46		2001
Block Digestor	Lachat	BD-46		2001
Autoclave	Market Forge	Sterilmatic	166592	2004
PH meter	Fisher Scientific	Accumet Model 15	141979	1996
Centrifuge	Centrific	225	156622	2001
Refrigerator	Kenmore	7674310	E70741344	1980
Top loading balance	Denver Inst. Co.		A42051118	2001
Wet Chemistry Equipment	**Manufacturer**	**Model**	**Serial/ID No.**	**Year placed in service**
Conductivity Meter	YSI	3100	03B1296	2003
Drill Press, 8" benchtop	Westward	4TM69		2001
UV-Vis Spectrophotometer	Shimadzu	UV1601	A10753983866	2001
UV-Vis Spectrophotometer	Milton Roy	1201	0327833A	1987
Centrifuge	Beckman Coulter	Allegra 6	ALS03A31	2003
UV-Vis Spectrophotometer	Thermo Electron Corp.	Aquamate	114510	2003
Freezer, Explosion-proof	VWR	U2020XA14	025N-622974-ON	2003
Fluorometer	Turner-Designs	10-AU-005-CE	6670R0XX	2003
Fluorometer	Turner-Designs	10-AU-005	5379 FRXX	
Ionanalyzer	Orion	920A	002380	
Analytical Balance	Sartorius	A120S	37100120	1998
Analytical Balance	Mettler	AE200	N61981	1993
Top Load Balance	Ohaus	TS4000	6707	1996
Waterbath	Fisher Scientific	Isotemp 128	107N004-	2002
COD reactor	Hach	45600-02	020300023015	2002
Microbiology Equipment	**Manufacturer**	**Model**	**Serial/ID No.**	**Year placed in service**
Top Loading Balance	Denver Instrument Co.	XP-3000		
Balance	Denver Instrument Co.	A-200DS	13024400	1993
Quantitray Sampler Sealing System	IDEXX	2X	16096000	2002
Stereo Microscope	Fisher Scientific	Stereo Master	16100000	2002
Stereo Microscope	Fisher Scientific	Stereo Master	16100100	2002
Steri Microscope	Reichert-Jung		13029000	1993
Organic Carbon Analyzer	Tekmar Dohrman	Apollo 9000	15668800	2001
Turbidimeter	HF Scientific	Micro 100	16095400	2002
DO meter	YSI	5100	16195000	2003
ECON Incubator	Precision Scientific	5EM	16098300	2002
Incubator	Curtin Matheson Scientific	Equatherm	10869100	1987
Incubator	Precision Scientific	6LM	13323600	1994
Autoclave	Market Forge	Sterilmatic	16098400	2002
Dry Air Oven	Blue M	Lindberg	14096000	1996
Refrigerator	Kenmore			

Water Bath	Precision	253	14192600	1996
Water Bath	Blue M	Magni World	00579900	
Water Bath	Blue M	Magni World	00150000	
Water Bath	Blue M	Magni World	00149900	
Microbiology Equipment, *cont'd*.	**Manufacturer**	**Model**	**Serial/ID No.**	**Year placed in service**
Incubator	Sure-Temp	3478-2-Y	DI89216902	1991
Support Equipment	**Manufacturer**	**Model**	**Serial/ID No.**	**Year placed in service**
Autoclave	AMSCO	3201	010539106	1991
Dishwasher	AMSCO	470		1991
Dryer	AMSCO	475		1991
Walk-in cooler (rm. G035-A)	Sure-Temp	3478-2-W	DX89216901	1991
Walk-in cooler (rm. G028)	Sure-Temp	3478-2-W	DX89216902	1991
Walk-in cooler (rm. G088)	Sure-Temp	3478-2-W	DX89216904	1991

Table 4-2. Asheville Regional Laboratory Major Equipment List

Equipment	Manufacturer	Model	Serial/ID No.	Year placed in service
Muffle Furnace	Hotpack	7074	51462	1976
Oven	Blue M	OV-490A-2	JT8798	1976
Oven	Blue M	OV-12A	KAA 8586	1976
Turbidimeter	HF Scientific	Micro 100	201092	2003
DO Meter	YSI	50B	96B46283	1996
pH Meter	Orion	920A	002249	1992
Waterbath	Blue M	MW-1130A-1	MOS-16278	1976
Waterbath	Blue M	MW-1120A-1	M5-5216	1976
Autoclave	Market Forge	STM-E	012105	1976
Analytical Balance	Mettler	AE200	1113052122	1994
Incubator	VWR	2020	1102102	2003
DI water system	Pure Water Solutions		5469	2003
Compound Microscope	Spencer		AROM1	1976

Table 4-3. Washington Regional Laboratory Major Equipment List

Equipment	Manufacturer	Model	Serial/ID No.	Year placed in service
Turbidimeter	Monitek	126-01I-3C	T035	1994
Turbidimeter	HFScientific	Micro 100	211225	2002
Balance	Mettler	H-30	618407	~1975
Balance	Sartorius	MC1, Analytic AC 120 S	20401645	1993
Autoclave	Market Forge	STM-E	14998	~1975
PH meter	Symphony	SB301	0001982	2001
PH meter	Corning	10	D4507	~1975
BOD incubator	Precision	815	699071301	1999
BOD incubator	Shel Lab		0101103	2003
DO Meter	YSI	5000	03C1280	2003
DO Meter	YSI		L8006363	~1998
DO Meter	YSI		91K033108	~1998
Incubator	Blue M	MW-1130A-1	M5-1034	~1975
Incubator	Blue M	MW-1120A-1	M5-5213	~1975
Microscope	American Optical			~1975
Still	Barnstead		8207417	~1975
Oven	Blue M	OV 490A-2	OV3 8829	~1975
Oven	Blue M	OV-12 A		~1975
Oven	Shel Lab		0400103	2003
Oven	Despatch	LEB-1-20	96318	~1975
Muffle Furnace	Lindberg Blue		T29J422375UJ	1998
Incubator	GCA	FREAS 815	19-AF-3	1999

~ Indicates approximate date placed in service.

5.0 QA Targets for Precision, Accuracy and MDLs/PQLs

The DWQ Laboratory Section quality assurance objectives are described in terms of precision, accuracy, representativeness, and comparability. Criteria for data quality indicators such as matrix spikes, laboratory control samples and duplicate sample precision are specified in this section.

5.1 Quality Assurance Objectives

5.1.1 Precision

The laboratory objective for precision is to meet the precision demonstrated for the analytical methods on similar samples and to meet data for the analyses published by the US EPA. Precision is defined as the degree of reproducibility of repetitive measurements under a given set of analytical conditions (exclusive of field sampling variability). It is the degree of mutual agreement among independent measurements as the result of repeated application of the same process under similar conditions. Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike duplicate samples.

5.1.2 Accuracy

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the analytical methods on similar samples and to meet the recovery data published by the US EPA. Accuracy is defined as the degree of bias in a measurement system. It is a statistical measurement of correctness and includes components of random error (variability due to imprecision) and systematic error. It reflects the error associated with a measurement. A measurement is accurate when the value reported does not differ from the true value or known concentration of the spike or standard. Analytical accuracy is typically measured by determining the percent recovery of known target analytes that are spiked into a field sample (i.e., a surrogate or matrix spike) or reagent water (i.e., laboratory control sample or QC check sample). Surrogate compound recovery is reported and is used to assess method performance for each sample analyzed for volatile and semivolatile organic compounds. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

5.1.3 Representativeness

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

5.1.4 Comparability

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness and reporting limits statistics are similar to these quality indicators generated:

- By other laboratories for similar samples, and
- Data generated by the Laboratory Section over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects, by comparison of periodically generated statements of accuracy, precision and reporting limits with those of other laboratories and by the degree to which approval from the US EPA or other pertinent regulatory agencies is obtained for any procedure for which significant modifications have been made.

5.2 QA Targets

Analytes, preparative and analytical methods, matrices, accuracy and precision targets, MDLs and PQLs for analyses performed by the Laboratory Section are presented in Table 5.1. Unless otherwise noted, the limits in these tables are laboratory generated. Some acceptability limits are derived from USEPA methods when they are provided. Where USEPA limits are not provided, the Laboratory Section has adopted interim limits or developed limits from general laboratory practice or evaluation of data from similar matrices. Acceptability of QC will be determined as compared to these tables. Data may be accepted where QC falls outside these limits if probable cause can be attributed to the matrix and laboratory control samples show that the method is in control. Deviations are to be fully documented in the final report. In instances where a LCS limit is not available, a limit of 70-130% recovery is acceptable until in-house limits can be generated. In some cases, wider default limits may be set with the Quality Assurance/Quality Control Coordinator and Section Chief's approval. In the absence of in-house or method-defined limits, the following guidelines may be used to determine interim limits for matrix spike and matrix spike/matrix spike duplicates:

MS	60-140%
MS/MSD	20% RPD

Some criteria may need to be wider based on prior knowledge of the compound (e.g., phenols in EPA 8270).

5.3 Statistically Derived Limits

Statistically derived precision and accuracy limits are required by selected methods and programs. The Laboratory Section will routinely utilize statistically derived limits (based upon laboratory derived data) to evaluate method performance and determine when corrective action may be appropriate. The laboratory may periodically update the limits as stated in this manual. The analysts are instructed to use the current limits posted in the laboratory (dated and approved by the Quality Assurance Officer) and entered into a master log. The Quality Assurance Officer maintains an archive of all limits used within the laboratory. These updated limits may be equal to or tighter than the limits displayed in this QAM. If limits need to be adjusted outside of the limits in this QAM, the QAM will be revised to reflect these changes.

Where EPA acceptability criteria does not exist for a given method being utilized for the first time, the laboratories will establish control limits derived from a minimum of four data points. Until verified by a statistically significant data population, a reasonable interim value will be assigned and the control limits will be considered as advisory limits only and will not automatically initiate a corrective action if they are not met.

5.4 Method Detection Limits

Method Detection Limits (MDLs) are set such that the risk of reporting a false positive is less than 1%. MDLs are determined using the method specified in the Federal Register, 40 CFR Part 136 Appendix B. MDLs are based on the latest MDL study available at the time this document was published and may be superseded by the results from new studies. MDLs are updated annually or any time there is a significant change in laboratory operations.

5.5 Practical Quantitation Limits

Practical Quantitation Limits (PQLs) are set at 3 to 5 times the calculated MDL unless otherwise noted. Because PQL level checks are required, ease of preparation of commercial analytical mixes may dictate to some extent the reported PQL. Generally the PQL is not set at less than 3 times the MDL. However, in some instances, systematic bias (i.e., analyte background in reagents, etc.) necessitates that the reported MDL be elevated to levels that are readily quantifiable. In those instances, the PQL may be set at a level less than three times the reported MDL. Published PQLs may be set higher than experimentally determined PQLs to 1) avoid observed positive interferences from matrix effects or common reagent contaminants, or 2) for reporting convenience (i.e., to group common compounds with similar but slightly different experimentally determined PQLs).

Values between the MDL and PQL can be reported as required by a client; however, these values are always reported with a qualifier code (N3). Additionally, non-detected analytes are always reported as less than the PQL.

5.6 QA Targets Table

Note that MDLs and PQLs for soil/sediment matrices are based on method-specific sample dry weights. Detection limits may vary from that published, due to moisture content, dilution effects, interferences, special reporting requirements, etc.

The QA targets for all inorganic analyses are within the range of 80 - 120 % for accuracy [except for metals in solid samples, which have been set based on method defined limits (75-125 %)] and < 20% RPD for precision, unless laboratory-generated data indicate that tighter control limits can be routinely maintained. This convention was adopted due to the fact that targets set according to historical data are usually less stringent. The organic QA targets are statutory in nature; Warning and control limits for organic analyses are initially set for groups of compounds based on preliminary method validation data. When additional data is available, the QA targets may be reconsidered. All QA targets are routinely re-evaluated at least annually (and updated, if necessary) against laboratory generated data to insure targets continue to reflect realistic, methodologically achievable goals.

Each table in this section is formatted in the same way and the following conventions apply to all of them.

Matrices are denoted as follows:

W: surface, ground and waste water
S: soil, sediment, solid
T: tissue

Table 5.2 lists the keys to the clean up method in Table 5.1.

Acronyms used in the method citations are:

- **EPA** refers to methods published in *Methods for Chemical Analysis of Water and Wastes*, EPA 600/4-79-020, March 1983, 40 CFR Part 136, Appendices A-D and *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*, SW-846 (3rd Edition) as amended by Updates I, II, IIA and III.

- **SM##** refers to methods published in *Standard Methods for the Examination of Water and Wastewater*, APHA. Each citation is followed by a two-digit number, which refers specifically to the edition of Standard Methods being cited. For example, **SM18** refers to the 18th Edition (1992), **SM19** refers to the 19th Edition (1995) and **SM20** refers to the 20th Edition (1998).

- **ASTM** refers to methods published in the *Annual Book of ASTM standards*, Vols. 11.01 and 11.02, 1999.

- **HACH** refers to methods published in *Hach Water Analysis Handbook*, 3rd Edition, Hach Company Loveland, CO, 1997.

- **QuikChem** refers to methods published by Lachat Instruments, Milwaukee, WI.

- **USGS** refers to *Methods for Analysis of Inorganic Substances in Water and Fluvial Sediments*, U.S. Department of the Interior, Techniques of Water-Resource Investigation of the U.S. Geological Survey, Denver, CO, Revised 1989.

- Modified methods are designated with an "M" after the method number.

Table 5.1
QA Targets for Accuracy, Precision and MDLs/PQLs

Metals

Analyte	Prep Method[1]	Analysis Method[2]	Matrix	Spike[3] Recovery Range (%)	QCS[4] Accuracy Range (%)	Precision % RPD	MDL	PQL	Clean-Up Code
	EPA 200.2M/ SM18 3030C	EPA 200.7 / 200.8	W	70 – 130	90-110	≤20	3.10 / 5.78 µg/L	50 µg/L	A
Aluminum	EPA 200.2M	EPA 200.7	S	70 - 130	90-110	≤20	NA[5]	1.0 mg/kg	A
	EPA 200.3	EPA 200.8	T	70 – 130	90-110	≤20	NA[5]	0.10 mg/kg	A
	EPA 200.2M / SM18 3030C	EPA 200.8	W	70 – 130	90-110	≤20	0.470 µg/L	10 µg/L	A
Antimony	EPA 200.2M	EPA 200.8	S	70 – 130	90-110	≤20	NA[5]	0.20 mg/kg	A
	EPA 200.3	EPA 200.8	T	70 - 130	90-110	≤20	NA[5]	0.10 mg/kg	A
	EPA 200.2 M/ SM18 3030C	EPA 200.8 / 200.9	W	70 – 130	90-110	≤20	0.381 / 0.405 µg/L	10 µg/L	A
Arsenic	EPA 200.2M	EPA 200.8	S	70 – 130	90-110	≤20	NA[5]	0.20 mg/kg	A
	EPA 200.3	EPA 200.8	T	70 – 130	90-110	≤20	NA[5]	0.10 mg/kg	A
	EPA 200.2M/ SM18 3030C	EPA 200.7	W	70 – 130	90-110	≤20	0.344 µg/L	10 µg/L	A
Barium	EPA 200.2M	EPA 200.8 / 200.7	S	70 – 130	90-110	≤20	NA[5]	0.20 mg/kg	A
	EPA 200.2M / SM18 3030C	EPA 200.8 / 200.7	W	70 – 130	90-110	≤20	0.539 / 0.351 µg/L	10 µg/L	A
Beryllium	EPA 200.2M	EPA 200.8	S	70 – 130	90-110	≤20	NA[5]	0.20 mg/kg	A
	EPA 200.3	EPA 200.8	T	70 – 130	90-110	≤20	NA[5]	0.10 mg/kg	A
	EPA 200.2M / SM18 3030C	EPA 200.7	W	70 - 130	90-110	≤20	0.012 mg/L	0.10 mg/kg	A
Calcium	EPA 200.2M	EPA 200.7	S	70 – 130	90-110	≤20	NA[5]	2.0 mg/kg	A
	EPA 200.2M / SM18 3030C	EPA 200.8 / 200.9	W	70 – 130	90-110	≤20	0.115 / 0.209µg/L	2.0 µg/L	A
Cadmium	EPA 200.2M	EPA 200.8	S	70 – 130	90-110	≤20	NA[5]	0.20 mg/kg	A
	EPA 200.3	EPA 200.8	T	70 – 130	90-110	≤20	NA[5]	0.10 mg/kg	A
	EPA 200.2 M/ SM18 3030C	EPA 200.8 / 200.7	W	70 – 130	90-110	≤20	0.243 / 0.534 µg/L	25 µg/L	A
Chromium	EPA 200.2M	EPA 200.8	S	70 – 130	90-110	≤20	NA[5]	0.20 mg/kg	A
	EPA 200.3	EPA 200.8	T	70 – 130	90-110	≤20	NA[5]	0.10 mg/kg	A
	EPA 200.2M / SM18 3030C	EPA 200.8 / 200.7	W	70 – 130	90-110	≤20	0.058 / 0.623 µg/L	50 µg/L	A
Cobalt	EPA 200.2M	EPA 200.8	S	70 - 130	90-110	≤20	NA[5]	1.0 mg/kg	A
	EPA 200.3	EPA 200.8	T	70 – 130	90-110	≤20	NA[5]	0.10 mg/kg	A
	EPA 200.2M / SM18 3030C	EPA 200.8 / 200.9	W	70 – 130	90-110	≤20	0.897 / 0.661 µg/L	2.0 µg/L	A
Copper	EPA 200.2M	EPA 200.8	S	70 – 130	90-110	≤20	NA[5]	0.20 mg/kg	A
	EPA 200.3	EPA 200.8	T	70 – 130	90-110	≤20	NA[5]	0.10 mg/kg	A
	EPA 200.2M / SM18 3030C	EPA 200.7	W	70 – 130	90-110	≤20	6.19 µg/L	50 µg/L	A
Iron	EPA 200.2M	EPA 200.7	S	70 – 130	90-110	≤20	NA[5]	1.0 mg/kg	A
	EPA 200.2M / SM18 3030C	EPA 200.8 / 200.9	W	70 – 130	90-110	≤20	0.091 / 0.737 µg/L	10 µg/L	A
Lead	EPA 200.2M	EPA 200.8	S	70 – 130	90-110	≤20	NA[5]	0.20 mg/kg	A
	EPA 200.3	EPA 200.8	T	70 - 130	90-110	≤20	NA[5]	0.10 mg/kg	A
	EPA 200.2M / SM18 3030C	EPA 200.7	W	70 – 130	90-110	≤20	0.585 µg/L	25 µg/L	A
Lithium	EPA 200.2M	EPA 200.7	S	70 – 130	90-110	≤20	NA[5]	0.20 mg/kg	A
	EPA 200.2 M/ SM18 3030C	EPA 200.7	W	70 – 130	90-110	≤20	0.006 mg/L	0.10 mg/kg	A
Magnesium	EPA 200.2M	EPA 200.7	S	70 – 130	90-110	≤20	NA[5]	2.0 mg/kg	A

Table 5.1
QA Targets for Accuracy, Precision and MDLs/PQLs

Metals

Analyte	Prep Method[1]	Analysis Method[2]	Matrix	Spike[3] Recovery Range (%)	QCS[4] Accuracy Range (%)	Precision % RPD	MDL	PQL	Clean-Up Code
	EPA 200.2M / SM18 3030C	EPA 200.7 / 200.8	W	70 – 130	90-110	≤20	0.506 / 0.219 µg/L	10 µg/L	A
Manganese	EPA 200.2M	EPA 200.8	S	70 – 130	90-110	≤20	NA[5]	0.20 mg/kg	A
	EPA 200.3	EPA 200.8	T	70 – 130	90-110	≤20	NA[5]	0.10 mg/kg	A
	EPA 245.1M	EPA 245.1	W	70 – 130	90-110	≤20	0.035 µg/L	0.20 µg/L	A
Mercury	EPA 245.5M	EPA 245.5	S	70 - 130	90-110	≤20	0.004 mg/kg	0.02 mg/kg	A
	EPA 245.6M	EPA 245.6	T	70 – 130	90-110	≤20	0.006 mg/kg	0.02 mg/kg	A
	EPA 200.2M / SM18 3030C	EPA 200.8 / 200.9	W	70 – 130	90-110	≤20	0.243 / 2.10 µg/L	10 µg/L	A
Nickel	EPA 200.2M	EPA 200.8	S	70 – 130	90-110	≤20	NA[5]	0.20 mg/kg	A
	EPA 200.3	EPA 200.8	T	70 – 130	90-110	≤20	NA[5]	0.10 mg/kg	A
	EPA 200.2M / SM18 3030C	EPA 200.7	W	70 – 130	90-110	≤20	0.006 mg/L	0.10 mg/kg	A
Potassium	EPA 200.2M	EPA 200.7	S	70 – 130	90-110	≤20	NA[5]	2.0 mg/kg	A
	EPA 200.2 M/ SM18 3030C	EPA 200.8 / 200.9	W	70 – 130	90-110	≤20	0.179 / 1.298 µg/L	5.0 µg/L	A
Selenium	EPA 200.2M	EPA 200.8	S	70 - 130	90-110	≤20	NA[5]	0.20 mg/kg	A
	EPA 200.3	EPA 200.8	T	70 – 130	90-110	≤20	NA[5]	0.10 mg/kg	A
	EPA 200.2M / SM18 3030C	EPA 200.8 / 200.9	W	70 – 130	90-110	≤20	0.277 / 0.967µg/L	5.0 µg/L	A
Silver	EPA 200.2M	EPA 200.8	S	70 – 130	90-110	≤20	NA[5]	0.20 mg/kg	A
	EPA 200.3	EPA 200.8	T	70 – 130	90-110	≤20	NA[5]	0.10 mg/kg	A
	EPA 200.2M / SM18 3030C	EPA 200.7	W	70 – 130	90-110	≤20	0.039 mg/L	0.10 mg/kg	A
Sodium	EPA 200.2M	EPA 200.7	S	70 – 130	90-110	≤20	NA[5]	2.0 mg/kg	A
	EPA 200.2 M/ SM18 3030C	EPA 200.8	W	70 – 130	90-110	≤20	0.067 µg/L	10 µg/L	A
Thallium	EPA 200.2M	EPA 200.8	S	70 - 130	90-110	≤20	NA[5]	0.20 mg/kg	A
	EPA 200.3	EPA 200.8	T	70 – 130	90-110	≤20	NA[5]	0.10 mg/kg	A
	EPA 200.2 M/ SM18 3030C	EPA 200.7 / 200.8	W	70 – 130	90-110	≤20	0.685 / 1.80 µg/L	25 µg/L	A
Vanadium	EPA 200.2M	EPA 200.8	S	70 – 130	90-110	≤20	NA[5]	0.20 mg/kg	A
	EPA 200.3	EPA 200.8	T	70 – 130	90-110	≤20	NA[5]	0.10 mg/kg	A
	EPA 200.2M / SM18 3030C	EPA 200.7 / 200.8	W	70 – 130	90-110	≤20	1.69 / 2.69 µg/L	10 µg/L	A
Zinc	EPA 200.2M	EPA 200.8	S	70 – 130	90-110	≤20	NA[5]	0.20 mg/kg	A
	EPA 200.3	EPA 200.8	T	70 – 130	90-110	≤20	NA[5]	0.10 mg/kg	A

[1]SM 3030C must be used to prepare aqueous samples taken from groundwater monitoring wells.

[2]Where two methods are listed, the first one is preferred for analysis.

[3]EPA Method 200.7, Section 9.4.3, Revision 4.4 May 1994. EPA Method 200.8, Section 9.4.3, Revision 5.4 May 1994. EPA Method 200.9, Section 9.4.3, Revision 2.2 May 1994.

[4]The QCS (Quality Control Sample) must be from a different source than calibration standards and have a "Certificate of Analysis" document from the vendor. The accuracy range listed is for QCS containing concentrations at the midrange of calibration curve. QCS with concentration at the lower end of the calibration curve will use "Acceptance Limits based on US EPA WS and WP Interlaboratory Study" listed on the "Certificate of Analysis" sheet or calculated control limits.

[5]NA = not available. MDL values have not been determined for sediment "S" and tissue "T", except for Hg.

* Limits determined from historical data

Table 5.1
QA Targets for Accuracy, Precision and MDLs/PQLs

Nutrients

Analyte	Prep Method	Analysis Method	Matrix	Spike[1] Accuracy Range (%)	LCS[2] Accuracy Range (%)	Precision % RPD	MDL (ppm)	PQL (ppm)	Clean-Up Code
Ammonia Nitrogen as N	N/A	EPA 350.1 (QUIK CHEM 10-107-06-1-J)	W	90-110	90-110	≤10	0.005	0.01	A
Total Kjeldahl Nitrogen as N	N/A	EPA 351.2 (QUIK CHEM 10-107-06-2-H)	W	80-120 [3]	90-110	≤20 [3]	0.04	0.20	A
Nitrate + Nitrite Nitrogen as N	N/A	EPA 353.2 (QUIK CHEM 10-107-04-1-C)	W	90-110	90-110	≤10	0.004	0.01	A
Phosphorous, Total as P	N/A	EPA 365.1 (QUIK CHEM 10-115-01-1-EF)	W	90-110	90-110	≤10	0.01	0.02	A
Phosphorous, Dissolved as P	N/A	EPA 365.1 (QUIK CHEM 10-115-01-1-EF)	W	90-110	90-110	≤10	0.01	0.02	A
Orthophosphate as P	N/A	EPA 365.1 (QUIK CHEM 10-115-01-1-A)	W	90-110	90-110	≤10	0.01	0.02	A

N/A = not applicable (the preparatory steps for TKN and Phosphorous are incorporated in the Lachat methods.

[1] Quik Chem Lachat Methods

[2] Quik Chem Lachat Methods

[3] SM18 interim until in-house limits are derived.

Table 5.1
QA Targets for Accuracy, Precision and MDLs/PQLs

Microbiology

Analyte	Prep Method	Analysis Method	Matrix	Spike Accuracy Range (%)	LCS Accuracy Range (%)	Precision % RPD	MDL (ppm or as noted)	PQL (ppm or as noted)	Clean-Up Code
BOD_5	N/A	SM20 5210 B	W	N/A	198 ± 30.5[1]	<20	N/A	2.0	A
Coliform, MF fecal	N/A	SM 189222 D	W	N/A	N/A	<20	N/A	1 cfu/100 ml	A
Coliform, MF total	N/A	SM18 9222 B	W	N/A	N/A	<20	N/A	1 cfu/100 ml	A
Coliform, MPN fecal	N/A	SM 189221 B	W	N/A	N/A	<20	N/A	2 MPN/100 ml	A
Coliform, MPN total	N/A	SM18 9221 B	W	N/A	N/A	<20	N/A	2 MPN/100 ml	A
Coliform, fecal strep	N/A	SM18 9230 C	W	N/A	N/A	<20	N/A	1 cfu/100 ml	A
TOC	N/A	SM18 5310 B	W	80-120	90-110	<20	0.1244	5 mg/L	A
Total residue	N/A	SM18 2540 B	W	N/A	90-110	<20*	N/A	10	A
Total volatile residue	N/A	EPA 160.4	W	N/A	90-110	<20*	N/A	10	A
Total fixed residue	N/A	EPA 160.4	W	N/A	90-110	<20*	N/A	10	A
Total Suspended Residue	N/A	SM18 2540 D	W	N/A	90-110	<20*	N/A	2	A
Total Volatile Suspended Residue	N/A	EPA 160.4	W	N/A	90-110	<20*	N/A	2	A
Total Fixed Suspended Residue	N/A	EPA 160.4	W	N/A	90-110	<20*	N/A	2	A
Turbidity	N/A	SM20 2130 B	W	N/A	90-110	<20	N/A	1 NTU	A

N/A = not applicable
* For concentrations > 50 mg/L
[1] GGA Standard

Table 5.1
QA Targets for Accuracy, Precision and MDLs/PQLs

Wet Chemistry

Analyte	Prep Method	Analysis Method	Matrix	Spike Accuracy Range (%)	LCS Accuracy Range (%)	Precision % RPD	MDL	PQL	Clean-Up Code
Acidity to pH 4.5	N/A	SM18 2310 B	W	N/A	mfg	≤20		1 mg/L	A
Acidity to pH 8.3	N/A	SM18 2310 B	W	N/A	mfg	≤20		1 mg/L	A
Alkalinity to pH 8.3, total as $CaCO_3$	N/A	SM18 2320 B	W	N/A	mfg	≤20		1 mg/L	A
Alkalinity to pH 4.5, total as $CaCO_3$	N/A	SM18 2320 B	W	N/A	mfg	≤20		1 mg/L	A
Boron	N/A	SM18 4500-B B	W	80-120	mfg	≤20	0.06 ug/L	0.25 ppb	A
Chloride	N/A	EPA 325.3	W	80-120	mfg	≤20	1.68	5 mg/L	A
Chlorophyll *a* (uncorrected)	N/A	EPA 445.0	W	N/A	95-105	≤20	N/A	1 ppb [1]	A
COD	N/A	SM18 5520 D M	W	80-120	mfg	≤20	10.64	20 mg/L	A
Color, ADMI	N/A	SM18 2120 E	W	N/A	80-120	≤20	1.2	25 mg/L	A
Color, True	N/A	SM18 2120 C	W	N/A	80-120	≤20	1.58	5 c.u.	A
Cyanide	SM18 4500-CN C	SM18 4500-CN E	W	80-120	mfg	≤20	0.01	0.02 mg/L	A
Fluoride	SM18 4500-F^- B	SM18 4500-F^- C	W	80-120	mfg	≤20	0.18	0.50 mg/L	A
Formaldehyde	N/A	APHA, 1972 Method 111	W	80-120	80-120	≤20	0.04	0.2 mg/L	A
Grease & Oil	N/A	EPA 1664	W	*	*	*	*	*	A
Hexavalent Chromium	N/A	SM18 3500-Cr D	W	80-120	90-110	≤20	12.45	50 ppb	A
MBAS	N/A	SM18 5540 C	W	80-120	mfg	≤20	0.03	0.1 mg/L	A
Phenol	N/A	EPA 420.1	W	80-120	mfg	≤20	2.94	10 ppb	A
Silica	N/A	USGS 1-1700-85	W	80-120	mfg	≤20	1.8	5 mg/L	A
Specific Conductance	N/A	SM18 2510 B	W	N/A	mfg	≤20	0.31	14.9 umhos/cm	A

Table 5.1
QA Targets for Accuracy, Precision and MDLs/PQLs

Wet Chemistry

Analyte	Prep Method	Analysis Method	Matrix	Spike Accuracy Range (%)	LCS Accuracy Range (%)	Precision % RPD	MDL	PQL	Clean-Up Code
Sulfate	N/A	EPA 375.4	W	80-120	mfg	≤20	0.42	5	A
Sulfide	N/A	SM18 4500-S_2 D	W	80-120	mfg	≤20	0.01	0.1	A
Tannin and Lignin	N/A	SM18 5550	W	80-120	mfg	≤20	*	*	A
Total Dissolved Residue	N/A	EPA 160.1	W	80-120	mfg	≤20	N/A	10	A
* Under method development.									
N/A = not applicable									
1 This is an estimated detection limit (EDL) - the minimum concentration of an analyte that yields a fluorescence 3X the fluorescence of blank filters which have been extracted according to the referenced method.									
mfg = Oustide quality control standards are purchased and the manufacturer's published limits are used.									

Table 5.1
QA Targets for Accuracy, Precision and MDLs/PQLs

Volatile Organics

Analyte	Prep Method	Analysis Method	Matrix	Spike Accuracy Range (%)	LCS Accuracy Range (%)	Precision % RPD	MDL (ppb)	PQL (ppb)	Clean-Up Code
1,1-Dichloroethene	EPA 5030/601	EPA 8021/601	W	60-150	60-150	≤20	0.03	0.30	A
	EPA 5030	EPA 8021	S					9	A
	EPA 5030/624	EPA 8260/624	W				0.23	1	A
Methylene Chloride	EPA 5030/601	EPA 8021/601	W	59-141	59-141	≤20	0.73	10	A
	EPA 5030	EPA 8021	S					30	A
	EPA 5030/624	EPA 8260/624	W				0.56	10	A
Trans-1,2-Dichloroethene	EPA 5030/601	EPA 8021/601	W				0.02	0.25	A
	EPA 5030	EPA 8021	S					7.5	A
	EPA 5030/624	EPA 8260/624	W				0.20	1	A
1,1-Dichloroethane	EPA 5030/601	EPA 8021/601	W				0.03	0.25	A
	EPA 5030	EPA 8021	S					7.5	A
	EPA 5030/624	EPA 8260/624	W				0.20	1	A
2,2-Dichloropropane	EPA 5030/601	EPA 8021/601	W				0.04	0.30	A
	EPA 5030	EPA 8021	S					9	A
	EPA 5030/624	EPA 8260/624	W				0.37	1	A
cis-1,2-Dichloroethene	EPA 5030/601	EPA 8021/601	W				0.02	0.25	A
	EPA 5030	EPA 8021	S					7.5	A
	EPA 5030/624	EPA 8260/624	W				0.16	1	A
Chloroform	EPA 5030/601	EPA 8021/601	W	67-133	67-133	≤20	0.03	0.25	A
	EPA 5030	EPA 8021	S					7.5	A
	EPA 5030/624	EPA 8260/624	W				0.24	1	A
Bromochloromethane	EPA 5030/601	EPA 8021/601	W				0.03	0.30	A
	EPA 5030	EPA 8021	S					9	A
	EPA 5030/624	EPA 8260/624	W				0.31	1	A
1,1,1-Trichloroethane	EPA 5030/601	EPA 8021/601	W	60-141	60-141	≤20	0.03	0.25	A
	EPA 5030	EPA 8021	S					7.5	A
	EPA 5030/624	EPA 8260/624	W				0.18	1	A
1,1-Dichloropropene	EPA 5030/601	EPA 8021/601	W				0.03	0.30	A
	EPA 5030	EPA 8021	S					9	A
	EPA 5030/624	EPA 8260/624	W				0.15	1	A
Carbon Tetrachloride	EPA 5030/601	EPA 8021/601	W	64-137	64-137	≤20	0.04	0.30	A
	EPA 5030	EPA 8021	S					9	A
	EPA 5030/624	EPA 8260/624	W				0.12	1	A
1,2-Dichloroethane	EPA 5030/601	EPA 8021/601	W	71-133	71-133	≤20	0.03	0.25	A
	EPA 5030	EPA 8021	S					7.5	A
	EPA 5030/624	EPA 8260/624	W				0.25	1	A
Trichloroethene	EPA 5030/601	EPA 8021/601	W	61-136	61-136	≤20	0.03	0.25	A
	EPA 5030	EPA 8021	S					7.5	A

Table 5.1
QA Targets for Accuracy, Precision and MDLs/PQLs

Volatile Organics

Analyte	Prep Method	Analysis Method	Matrix	Spike Accuracy Range (%)	LCS Accuracy Range (%)	Precision % RPD	MDL (ppb)	PQL (ppb)	Clean-Up Code
	EPA 5030/624	EPA 8260/624	W				0.19	1	A
1,2-Dichloropropane	EPA 5030/601	EPA 8021/601	W				0.03	0.25	A
	EPA 5030	EPA 8021	S					7.5	A
	EPA 5030/624	EPA 8260/624	W				0.25	1	A
Bromodichloro-methane	EPA 5030/601	EPA 8021/601	W	71-128	71-128	≤20	0.05	0.30	A
	EPA 5030	EPA 8021	S					9	A
	EPA 5030/624	EPA 8260/624	W				0.26	1	A
Dibromomethane	EPA 5030/601	EPA 8021/601	W				0.03	0.25	A
	EPA 5030	EPA 8021	S					7.5	A
	EPA 5030/624	EPA 8260/624	W				0.45	1	A
cis-1,3-Dichloropropene	EPA 5030/601	EPA 8021/601	W				0.03	0.30	A
	EPA 5030	EPA 8021	S					9	A
	EPA 5030/624	EPA 8260/624	W				0.19	1	A
trans-1,3-Dichloropropene	EPA 5030/601	EPA 8021/601	W				0.02	0.25	A
	EPA 5030	EPA 8021	S					7.5	A
	EPA 5030/624	EPA 8260/624	W				0.18	1	A
1,1,2-Trichloroethane	EPA 5030/601	EPA 8021/601	W	66-139	66-139	≤20	0.03	0.25	A
	EPA 5030	EPA 8021	S					7.5	A
	EPA 5030/624	EPA 8260/624	W				0.45	1	A
Tetrachloroethene	EPA 5030/601	EPA 8021/601	W	62-136	62-136	≤20	0.03	0.25	A
	EPA 5030	EPA 8021	S					7.5	A
	EPA 5030/624	EPA 8260/624	W				0.08	1	A
1,3-Dichloropropane	EPA 5030/601	EPA 8021/601	W				0.03	0.25	A
	EPA 5030	EPA 8021	S					7.5	A
	EPA 5030/624	EPA 8260/624	W				0.29	1	A
Dibromochloro-methane	EPA 5030/601	EPA 8021/601	W				0.03	0.30	A
	EPA 5030	EPA 8021	S					7.5	A
	EPA 5030/624	EPA 8260/624	W				0.29	1	A
1,2-Dibromoethane	EPA 5030/601	EPA 8021/601	W				0.05	0.30	A
	EPA 5030	EPA 8021	S					9	A
	EPA 5030/624	EPA 8260/624	W				0.21	1	A
Chlorobenzene	EPA 5030/601	EPA 8021/601	W	71-127	71-127	≤20	0.03	0.25	A
	EPA 5030	EPA 8021	S					9	A
	EPA 5030/624	EPA 8260/624	W				0.15	1	A
1,1,1,2-Tetrachloroethane	EPA 5030/601	EPA 8021/601	W				0.02	0.25	A
	EPA 5030	EPA 8021	S					7.5	A
	EPA 5030/624	EPA 8260/624	W				0.15	1	A
Bromoform	EPA 5030/601	EPA 8021/601	W	59-139	59-139	≤20	0.04	0.30	A

Table 5.1
QA Targets for Accuracy, Precision and MDLs/PQLs

Volatile Organics

Analyte	Prep Method	Analysis Method	Matrix	Spike Accuracy Range (%)	LCS Accuracy Range (%)	Precision % RPD	MDL (ppb)	PQL (ppb)	Clean-Up Code
	EPA 5030	EPA 8021	S					9	A
	EPA 5030/624	EPA 8260/624	W				0.36	1	A
1,1,2,2-Tetrachloroethane	EPA 5030/601	EPA 8021/601	W				0.03	0.30	A
	EPA 5030	EPA 8021	S					9	A
	EPA 5030/624	EPA 8260/624	W				1.52	5	A
1,2,3-Trichloropropane	EPA 5030/601	EPA 8021/601	W				0.05	0.30	A
	EPA 5030	EPA 8021	S					9	A
	EPA 5030/624	EPA 8260/624	W				0.54	1	A
Bromobenzene	EPA 5030/601/602	EPA 8021/601	W				0.05	0.30	A
	EPA 5030	EPA 8021	S					9	A
	EPA 5030/624	EPA 8260/624	W				0.33	1	A
2-Chlorotoluene	EPA 5030/601/602	EPA 8021/601	W				0.03	0.25	A
	EPA 5030	EPA 8021	S					7.5	A
	EPA 5030/624	EPA 8260/624	W				0.18	1	A
4-Chlorotoluene	EPA 5030/601/602	EPA 8021/601	W				0.06	0.30	A
	EPA 5030	EPA 8021	S					9	A
	EPA 5030/624	EPA 8260/624	W				0.18	1	A
1,3-Dichlorobenzene	EPA 5030/601/602	EPA 8021/601	W	66-127	66-127	≤20	0.02	0.25	A
	EPA 5030	EPA 8021	S					7.5	A
	EPA 5030/624	EPA 8260/624	W				0.22	1	A
1,4-Dichlorobenzene	EPA 5030/601/602	EPA 8021/601	W				0.03	0.25	A
	EPA 5030	EPA 8021	S					7.5	A
	EPA 5030/624	EPA 8260/624	W				0.16	1	A
1,2-Dichlorobenzene	EPA 5030/601/602	EPA 8021/601	W	72-124	72-124	≤20	0.06	0.30	A
	EPA 5030	EPA 8021	S					9	A
	EPA 5030/624	EPA 8260/624	W				0.23	1	A
1,2-Dibromo-3-Chloropropane	EPA 5030/601	EPA 8021/601	W				0.08	0.75	A
	EPA 5030	EPA 8021	S					23	A
	EPA 5030/624	EPA 8260/624	W				1.39	5	A
1,2,4-Trichlorobenzene	EPA 5030/601	EPA 8021/601	W				0.04	0.30	A
	EPA 5030	EPA 8021	S					9	A
	EPA 5030/624	EPA 8260/624	W				0.20	1	A
Hexachlorobutadiene	EPA 5030/601	EPA 8021/601	W				0.04	0.30	A
	EPA 5030	EPA 8021	S					9	A
	EPA 5030/624	EPA 8260/624	W				0.25	1	A
1,2,3-Trichlorobenzene	EPA 5030/601	EPA 8021/601	W				0.07	0.75	A
	EPA 5030	EPA 8021	S					23	A
	EPA 5030/624	EPA 8260/624	W				0.29	1	A

Table 5.1
QA Targets for Accuracy, Precision and MDLs/PQLs

Volatile Organics

Analyte	Prep Method	Analysis Method	Matrix	Spike Accuracy Range (%)	LCS Accuracy Range (%)	Precision % RPD	MDL (ppb)	PQL (ppb)	Clean-Up Code
Methyl-tert-butyl ether	EPA 5030/602	EPA 8021/602	W				0.53	5.0	A
	EPA 5030	EPA 8021	S					150	A
	EPA 5030/624	EPA 8260/624	W				1.72	5.0	A
Benzene	EPA 5030/602	EPA 8021/602	W	65-142	65-142	≤20	0.05	1.0	A
	EPA 5030	EPA 8021	S					30	A
	EPA 5030/624	EPA 8260/624	W				0.16	1.0	A
Toluene	EPA 5030/601/602	EPA 8021/602	W	72-126	72-126	≤20	0.09	1.0	A
	EPA 5030	EPA 8021	S					30	A
	EPA 5030/624	EPA 8260/624	W				0.17	1.0	A
Ethyl benzene	EPA 5030/602	EPA 8021/602	W	68-127	68-127	≤20	0.13	1.0	A
	EPA 5030	EPA 8021	S					30	A
	EPA 5030/624	EPA 8260/624	W				0.10	1.0	A
m,p-Xylenes	EPA 5030/602	EPA 8021/602	W				0.17	2.0	A
	EPA 5030	EPA 8021	S					60	A
	EPA 5030/624	EPA 8260/624	W				0.23	2.0	A
o-Xylene	EPA 5030/602	EPA 8021/602	W				0.08	1.0	A
	EPA 5030	EPA 8021	S					30	A
	EPA 5030/624	EPA 8260/624	W				0.11	1.0	A
Styrene	EPA 5030/602	EPA 8021/602	W				0.08	1.0	A
	EPA 5030	EPA 8021	S					30	A
	EPA 5030/624	EPA 8260/624	W				0.14	1.0	A
Isopropylbenzene	EPA 5030/602	EPA 8021/602	W				0.09	1.0	A
	EPA 5030	EPA 8021	S					30	A
	EPA 5030/624	EPA 8260/624	W				0.11	1.0	A
n-Propylbenzene	EPA 5030/602	EPA 8021/602	W				0.08	1.0	A
	EPA 5030	EPA 8021	S					30	A
	EPA 5030/624	EPA 8260/624	W				0.13	1.0	A
1,3,5-Trimethylbenzene	EPA 5030/602	EPA 8021/602	W				0.10	1.0	A
	EPA 5030	EPA 8021	S					30	A
	EPA 5030/624	EPA 8260/624	W				0.19	1.0	A
tert-Butylbenzene	EPA 5030/602	EPA 8021/602	W				0.11	1.0	A
	EPA 5030	EPA 8021	S					30	A
	EPA 5030/624	EPA 8260/624	W				0.16	1.0	A
1,2,4-Trimethylbenzene	EPA 5030/602	EPA 8021/602	W				0.09	1.0	A
	EPA 5030	EPA 8021	S					30	A
	EPA 5030/624	EPA 8260/624	W				0.18	1.0	A
sec-Butylbenzene	EPA 5030/602	EPA 8021/602	W				0.10	1.0	A
	EPA 5030	EPA 8021	S					30	A

Table 5.1
QA Targets for Accuracy, Precision and MDLs/PQLs

Volatile Organics

Analyte	Prep Method	Analysis Method	Matrix	Spike Accuracy Range (%)	LCS Accuracy Range (%)	Precision % RPD	MDL (ppb)	PQL (ppb)	Clean-Up Code
	EPA 5030/624	EPA 8260/624	W				0.12	1.0	A
p-isopropyltoluene	EPA 5030/602	EPA 8021/602	W				0.11	1.0	A
	EPA 5030	EPA 8021	S					30	A
	EPA 5030/624	EPA 8260/624	W				0.16	1.0	A
n-Butylbenzene	EPA 5030/602	EPA 8021/602	W				0.10	1.0	A
	EPA 5030	EPA 8021	S					30	A
	EPA 5030/624	EPA 8260/624	W				0.14	1.0	A
Naphthalene	EPA 5030/602	EPA 8021/602	W				0.10	1.0	A
	EPA 5030	EPA 8021	S					30	A
	EPA 5030/624	EPA 8260/624	W				025	2.0	A
								PPM	
TPH-GRO	EPA 5030	CA LUFT	W				0.02	0.2	A
	EPA 5030	CA LUFT	S					6	A

Table 5.1
QA Targets for Accuracy, Precision and MDLs/PQLs

Semivolatile Organics

Analyte	Prep Method	Analysis Method	Matrix	Spike Accuracy Range (%)	LCS Accuracy Range (%)	Precision % RPD	MDL (ppb)	PQL (ppb)	Clean-Up Code
ANILINE	EPA 625/3510	EPA 625/8270	W				4	10	A
	EPA 3550	EPA 8270	S				260	660	A
PHENOL	EPA 625/3510	EPA 625/8270	W	15 - 55	15 - 55	≤22	4	10	A
	EPA 3550	EPA 8270	S	26 - 90	26 - 90	≤35	260	660	A
BIS(2-CHLOROETHYL) ETHER	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
2-CHLOROPHENOL	EPA 625/3510	EPA 625/8270	W	49 – 93	49 – 93	≤16	2	10	A
	EPA 3550	EPA 8270	S	25 – 102	25 – 102	≤50	130	660	A
1,3-DICHLOROBENZENE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
1,4-DICHLOROBENZENE	EPA 625/3510	EPA 625/8270	W	41 - 92	41 - 92	≤22	2	10	A
	EPA 3550	EPA 8270	S	28 - 104	28 - 104	≤27	130	660	A
BENZYL ALCOHOL	EPA 625/3510	EPA 625/8270	W				4	20	A
	EPA 3550	EPA 8270	S				260	1300	A
1,2-DICHLOROBENZENE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
2-METHYL PHENOL	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
BIS(2-CHLOROISOPROPYL) ETHER	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
4-METHYL PHENOL	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
N-NITROSO-DI-N-PROPYLAMINE	EPA 625/3510	EPA 625/8270	W	55 - 107	55 - 107	≤17	2	10	A
	EPA 3550	EPA 8270	S	41 - 126	41 - 126	≤38	130	660	A
HEXACHLORO-ETHANE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
NITROBENZENE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
ISOPHORONE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
2-NITRO PHENOL	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
2,4-DIMETHYL PHENOL	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				200	660	A
BENZOIC ACID	EPA 625/3510	EPA 625/8270	W				10	50	A
	EPA 3550	EPA 8270	S				660	3300	A
BIS(2-CHLOROETHOXY) METHANE	EPA 625/3510	EPA 625/8270	W				2	10	A

Table 5.1
QA Targets for Accuracy, Precision and MDLs/PQLs

Semivolatile Organics

Analyte	Prep Method	Analysis Method	Matrix	Spike Accuracy Range (%)	LCS Accuracy Range (%)	Precision % RPD	MDL (ppb)	PQL (ppb)	Clean-Up Code
	EPA 3550	EPA 8270	S				130	660	A
2,4-DICHLORO PHENOL	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
1,2,4-TRICHLORO-BENZENE	EPA 625/3510	EPA 625/8270	W	43 - 94	43 - 94	≤21	2	10	A
	EPA 3550	EPA 8270	S	38 – 107	38 - 107	≤23	130	660	A
NAPHTHALENE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
4-CHLOROANILINE	EPA 625/3510	EPA 625/8270	W				4	20	A
	EPA 3550	EPA 8270	S				330	1300	A
HEXACHLORO-BUTADIENE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
4-CHLORO-3-METHYL PHENOL	EPA 625/3510	EPA 625/8270	W	48 - 104	48 - 104	≤15	2	10	A
	EPA 3550	EPA 8270	S	26 - 103	26 -103	≤33	130	660	A
2-METHYL NAPHTHALENE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
HEXACHLORO-CYCLOPENTADIENE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
2,4,6-TRICHLORO PHENOL	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
2,4,5-TRICHLORO PHENOL	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
2-CHLORO NAPHTHALENE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
2-NITROANILINE	EPA 625/3510	EPA 625/8270	W				10	50	A
	EPA 3550	EPA 8270	S				660	3300	A
DIMETHYL PHTHALATE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
ACENAPHTHYLENE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
2,6-DINITROTOLUENE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
3-NITROANILINE	EPA 625/3510	EPA 625/8270	W				10	50	A
	EPA 3550	EPA 8270	S				660	3300	A
ACENAPHTHENE	EPA 625/3510	EPA 625/8270	W	47 - 102	47 - 102	≤20	2	10	A
	EPA 3550	EPA 8270	S	31 - 137	31 - 137	≤19	130	660	A
2,4-DINITRO PHENOL	EPA 625/3510	EPA 625/8270	W				10	50	A
	EPA 3550	EPA 8270	S				660	3300	A
4-NITRO PHENOL	EPA 625/3510	EPA 625/8270	W	14 - 69	14 - 69	≤33	10	50	A

Table 5.1
QA Targets for Accuracy, Precision and MDLs/PQLs

Semivolatile Organics

Analyte	Prep Method	Analysis Method	Matrix	Spike Accuracy Range (%)	LCS Accuracy Range (%)	Precision % RPD	MDL (ppb)	PQL (ppb)	Clean-Up Code
	EPA 3550	EPA 8270	S	11 - 114	11 - 114	≤50	660	3300	A
DIBENZOFURAN	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
2,4-DINITROTOLUENE	EPA 625/3510	EPA 625/8270	W	41 - 103	41 - 103	≤17	2	10	A
	EPA 3550	EPA 8270	S	28 - 89	28 - 89	≤47	130	660	A
DIETHYL PHTHALATE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
4-CHLOROPHENYL PHENYL ETHER	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
FLUORENE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
4-NITROANILINE	EPA 625/3510	EPA 625/8270	W				10	50	A
	EPA 3550	EPA 8270	S				660	3300	A
4,6-DINITRO-2-METHYL PHENOL	EPA 625/3510	EPA 625/8270	W				10	50	A
	EPA 3550	EPA 8270	S				660	3300	A
N-NITROSODI-PHENYLAMINE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
4-BROMOPHENYL PHENYL ETHER	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
HEXACHLORO-BENZENE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
PENTACHLORO- PHENOL	EPA 625/3510	EPA 625/8270	W	46 - 122	46 - 122	≤18	10	50	A
	EPA 3550	EPA 8270	S	17 - 109	17 - 109	≤47	660	3300	A
PHENANTHRENE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
ANTHRACENE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
DI-N-BUTYL PHTHALATE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
FLUORANTHENE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
PYRENE	EPA 625/3510	EPA 625/8270	W	47 - 100	47 - 100	≤27	2	10	A
	EPA 3550	EPA 8270	S	35 - 142	35 - 142	≤36	130	660	A
BUTYLBENZYL PHTHALATE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
3,3'-DICHLORO-BENZIDINE	EPA 625/3510	EPA 625/8270	W				5	20	A
	EPA 3550	EPA 8270	S				260	1300	A
BENZO(A)-ANTHRACENE	EPA 625/3510	EPA 625/8270	W				2	10	A

Table 5.1
QA Targets for Accuracy, Precision and MDLs/PQLs

Semivolatile Organics

Analyte	Prep Method	Analysis Method	Matrix	Spike Accuracy Range (%)	LCS Accuracy Range (%)	Precision % RPD	MDL (ppb)	PQL (ppb)	Clean-Up Code
	EPA 3550	EPA 8270	S				130	660	A
CHRYSENE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
BIS(2-ETHYLHEXYL) PHTHALATE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
DI-N-OCTYL PHTHALATE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
BENZO(B)-FLUORANTHENE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
BENZO(K)-FLUORANTHENE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
BENZO(A)PYRENE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
INDENO(1,2,3-CD)-PYRENE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
DIBENZO(A,H)-ANTHRACENE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
BENZO(G,H,I)-PERYLENE	EPA 625/3510	EPA 625/8270	W				2	10	A
	EPA 3550	EPA 8270	S				130	660	A
TPH -DRO	EPA 3500	CA LUFT	W	12 - 127	12 - 127	$\leq$45	0.05 mg/L	0.5 mg/L	A
	EPA 3550	CA LUFT	S	36 - 119	55 - 110	$\leq$45	2 mg/Kg	10 mg/Kg	A

Table 5.1
QA Targets for Accuracy, Precision and MDLs/PQLs

Acid Herbicides (ECD)

Analyte	Prep Method	Analysis Method	Matrix	Spike Accuracy Range (%)	LCS Accuracy Range (%)	Precision % RPD	MDL (ppb)	PQL (ppb)	Clean-Up Code
ACIFLUORFEN (BLAZER)	EPA515.1, 8151A	EPA 8151A, 8000B	W				.04	0.1	A
	EPA 515.1, 8151A	EPA 8151A, 8000B	S				.71	3.3	A
BENTAZON	EPA 515.1, 8151A	EPA 8151A, 8000B	W				.05	0.3	A
	EPA 515.1, 8151A	EPA 8151A, 8000B	S				1.5	13	A
CHLORAMBEN	EPA 515.1, 8151A	EPA 8151A, 8000B	W				.02	0.1	A
	EPA 515.1, 8151A	EPA 8151A, 8000B	S				.94	3.3	A
2,4-D	EPA 515.1, 8151A	EPA 8151A, 8000B	W	30-142	30-142	≤35	.09	0.4	A
	EPA 515.1, 8151A	EPA 8151A, 8000B	S	39-146	39-146	≤30	2.56	6.7	A
2,4-DB	EPA 515.1, 8151A	EPA 8151A, 8000B	W				.23	0.7	A
	EPA 515.1, 8151A	EPA 8151A, 8000B	S				3.9	27	A
DCPA (ACID METABOLITES)	EPA 515.1, 8151A	EPA 8151A, 8000B	W				.12	0.4	A
	EPA 515.1, 8151A	EPA 8151A, 8000B	S				NE	NE	A
DICAMBA	EPA 515.1, 8151A	EPA 8151A, 8000B	W				.07	0.2	A
	EPA 515.1, 8151A	EPA 8151A, 8000B	S				1.2	3.3	A
3,5 DICHLOROBENZOIC ACID	EPA 515.1, 8151A	EPA 8151A, 8000B	W				.1	0.5	A
	EPA 515.1, 8151A	EPA 8151A, 8000B	S				1.16	3.3	A
DICHLORPROP	EPA 515.1, 8151A	EPA 8151A, 8000B	W				.11	0.5	A
	EPA 515.1, 8151A	EPA 8151A, 8000B	S				6.38	20	A
DINOSEB	EPA 515.1, 8151A	EPA 8151A, 8000B	W				.08	0.2	A
	EPA 515.1, 8151A	EPA 8151A, 8000B	S				2.02	6.7	A
5-HYDROXYDICAMBA	EPA 515.1, 8151A	EPA 8151A, 8000B	W				NA	NA	A
	EPA 515.1, 8151A	EPA 8151A, 8000B	S				NA	NA	A
4-NITROPHENOL	EPA 515.1, 8151A	EPA 8151A, 8000B	W				.21	0.6	A
	EPA 515.1, 8151A	EPA 8151A, 8000B	S				NE	13	A
PENTACHLORO-PHENOL (PCP)	EPA 515.1, 8151A	EPA 8151A, 8000B	W				.05	0.1	A
	EPA 515.1, 8151A	EPA 8151A, 8000B	S				.7	3.3	A
PICLORAM	EPA 515.1, 8151A	EPA 8151A, 8000B	W				.1	0.3	A
	EPA 515.1, 8151A	EPA 8151A, 8000B	S				.86	6.7	A
2,4,5- T	EPA 515.1, 8151A	EPA 8151A, 8000B	W	30-131	30-131	≤26	.04	0.1	A
	EPA 515.1, 8151A	EPA 8151A, 8000B	S	23-143	23-143	≤30	.87	3.3	A
2,4,5-TP (SILVEX)	EPA 515.1, 8151A	EPA 8151A, 8000B	W	41-117	41-117	≤21	.04	0.1	A
	EPA 515.1, 8151A	EPA 8151A, 8000B	S	10-134	10-134	≤30			A

Table 5.1
QA Targets for Accuracy, Precision and MDLs/PQLs

Chlorinated Pesticides/PCBs (ECD)

Analyte	Prep Method	Analysis Method	Matrix	Spike Accuracy Range (%)	LCS Accuracy Range (%)	Precision % RPD	MDL (ppb)	PQL (ppb)	Clean-Up Code
ALACHLOR	EPA 3510C	EPA 8081A	W					0.15	A
	EPA 3550	EPA 8081A	S					5.0	A
ALDRIN	EPA 3510C	EPA 8081A	W	47-113	47-113	≤21	.01	.025	A
	EPA 3550	EPA 8081A	S	44-135	44-135	≤43		0.83	A
ATRAZINE	EPA 3510C	EPA 8081A	W					3.0	A
	EPA 3550	EPA 8081A	S					100	A
BHC-ALPHA	EPA 3510C	EPA 8081A	W				.01	.025	A
	EPA 3550	EPA 8081A	S					0.83	A
BHC-BETA	EPA 3510C	EPA 8081A	W				.01	.025	A
	EPA 3550	EPA 8081A	S					0.83	A
BHC-DELTA	EPA 3510C	EPA 8081A	W				.01	.025	A
	EPA 3550	EPA 8081A	S					0.83	A
BHC-GAMMA (LINDANE)	EPA 3510C	EPA 8081A	W	42-126	42-126	≤21	.01	.025	A
	EPA 3550	EPA 8081A	S	47-118	47-118	≤50		0.83	A
CHLORDANE, TECHNICAL	EPA 3510C	EPA 8081A	W					0.50	A
	EPA 3550	EPA 8081A	S					17	A
CHLORDANE-ALPHA	EPA 3510C	EPA 8081A	W					0.020	A
	EPA 3550	EPA 8081A	S					0.50	A
CHLORDANE-GAMMA	EPA 3510C	EPA 8081A	W					0.020	A
	EPA 3550	EPA 8081A	S					0.50	A
CHLORDENE	EPA 3510C	EPA 8081A	W					0.025	A
	EPA 3550	EPA 8081A	S					0.83	A
CHLORNEB	EPA 3510C	EPA 8081A	W					0.20	A
	EPA 3550	EPA 8081A	S					6.7	A
CHLOROBENZILATE	EPA 3510C	EPA 8081A	W					0.60	A
	EPA 3550	EPA 8081A	S					20	A
CHLORPYRIFOS	EPA 3510C	EPA 8081A	W					0.050	A
	EPA 3550	EPA 8081A	S					1.7	A
CHLOROTHALONIL	EPA 3510C	EPA 8081A	W					0.025	A
	EPA 3550	EPA 8081A	S					0.83	A
DCPA	EPA 3510C	EPA 8081A	W					0.025	A
	EPA 3550	EPA 8081A	S					0.83	A
DDD, OP	EPA 3510C	EPA 8081A	W					0.050	A
	EPA 3550	EPA 8081A	S					1.7	A
DDD, PP	EPA 3510C	EPA 8081A	W				.01	0.025	A
	EPA 3550	EPA 8081A	S					0.83	A
DDE, OP	EPA 3510C	EPA 8081A	W					0.040	A

Table 5.1
QA Targets for Accuracy, Precision and MDLs/PQLs

Chlorinated Pesticides/PCBs (ECD)

Analyte	Prep Method	Analysis Method	Matrix	Spike Accuracy Range (%)	LCS Accuracy Range (%)	Precision % RPD	MDL (ppb)	PQL (ppb)	Clean-Up Code
	EPA 3550	EPA 8081A	S					1.3	A
DDE, PP	EPA 3510C	EPA 8081A	W				.03	0.025	A
	EPA 3550	EPA 8081A	S					0.83	A
DDT, OP	EPA 3510C	EPA 8081A	W					0.030	A
	EPA 3550	EPA 8081A	S					1.0	A
DDT, PP	EPA 3510C	EPA 8081A	W	35-137	35.137	≤24	.03	0.025	A
	EPA 3550	EPA 8081A	S	29-185	29-185	≤50		0.83	A
DIELDRIN	EPA 3510C	EPA 8081A	W	27-149	27-149	≤39	.03	0.025	A
	EPA 3550	EPA 8081A	S	54-127	54-127	≤48		0.83	A
ENDOSULFAN I	EPA 3510C	EPA 8081A	W				.01	0.025	A
	EPA 3550	EPA 8081A	S					0.83	A
ENDOSULFAN II	EPA 3510C	EPA 8081A	W				.01	0.025	A
	EPA 3550	EPA 8081A	S					0.83	A
ENDOSULFAN SULFATE	EPA 3510C	EPA 8081A	W				.03	0.025	A
	EPA 3550	EPA 8081A	S					0.83	A
ENDRIN	EPA 3510C	EPA 8081A	W	47-117	47-117	≤23	.01	0.025	A
	EPA 3550	EPA 8081A	S	44-120	44-120	≤45		0.83	A
ENDRIN ANDEHYDE	EPA 3510C	EPA 8081A	W				.01	0.025	A
	EPA 3550	EPA 8081A	S					0.83	A
ENDRIN KETONE	EPA 3510C	EPA 8081A	W					0.030	A
	EPA 3550	EPA 8081A	S					1.0	A
ETHAZOLE	EPA 3510C	EPA 8081A	W					0.060	A
	EPA 3550	EPA 8081A	S					2.0	A
HEPTACHLOR	EPA 3510C	EPA 8081A	W	41-122	41-122	≤25	.01	0.025	A
	EPA 3550	EPA 8081A	S	37-130	37-130	≤31		0.83	A
HEPTACHLOR EPOXIDE	EPA 3510C	EPA 8081A	W				.01	0.025	A
	EPA 3550	EPA 8081A	S					0.83	A
HEXACHLORO-BENZENE	EPA 3510C	EPA 8081A	W					0.015	A
	EPA 3550	EPA 8081A	S					0.50	A
MALATHION	EPA 3510C	EPA 8081A	W					0.20	A
	EPA 3550	EPA 8081A	S					6.7	A
METHOXYCHLOR, PP	EPA 3510C	EPA 8081A	W					0.10	A
	EPA 3550	EPA 8081A	S					3.3	A
MIREX	EPA 3510C	EPA 8081A	W					0.030	A
	EPA 3550	EPA 8081A	S					1.0	A
TRANS-NONACHLOR	EPA 3510C	EPA 8081A	W					0.020	A
	EPA 3550	EPA 8081A	S					0.50	A
OXYCHLORDANE	EPA 3510C	EPA 8081A	W					0.050	A

Table 5.1
QA Targets for Accuracy, Precision and MDLs/PQLs

Chlorinated Pesticides/PCBs (ECD)

Analyte	Prep Method	Analysis Method	Matrix	Spike Accuracy Range (%)	LCS Accuracy Range (%)	Precision % RPD	MDL (ppb)	PQL (ppb)	Clean-Up Code
	EPA 3550	EPA 8081A	S					1.70	A
MIXED-PERMETHRIN	EPA 3510C	EPA 8081A	W					1.20	A
	EPA 3550	EPA 8081A	S					40	A
PROPACHLOR	EPA 3510C	EPA 8081A	W					0.30	A
	EPA 3550	EPA 8081A	S					10.0	A
TECNAZENE	EPA 3510C	EPA 8081A	W					0.010	A
	EPA 3550	EPA 8081A	S					0.33	A
TRIFLURALIN	EPA 3510C	EPA 8081A	W					0.035	A
	EPA 3550	EPA 8081A	S					1.2	A
AROCHLOR 1016	EPA 3510C	EPA 8082	W	50-150	50-150	≤20		1.0	A
	EPA 3550	EPA 8082	S	50-150	50-150	≤30		33	A
AROCHLOR 1021	EPA 3510C	EPA 8082	W					1.0	A
	EPA 3550	EPA 8082	S					33	A
AROCHLOR 1032	EPA 3510C	EPA 8082	W					1.0	A
	EPA 3550	EPA 8082	S					33	A
AROCHLOR 1042	EPA 3510C	EPA 8082	W					1.0	A
	EPA 3550	EPA 8082	S					33	A
AROCHLOR 1048	EPA 3510C	EPA 8082	W					1.0	A
	EPA 3550	EPA 8082	S					33	A
AROCHLOR 1054	EPA 3510C	EPA 8082	W					1.0	A
	EPA 3550	EPA 8082	S					33	A
AROCHLOR 1260	EPA 3510C	EPA 8082	W	50-150	50-150	≤20		1.0	A
	EPA 3550	EPA 8082	S	50-150	50-150	≤30		33	A
AROCHLOR 1262	EPA 3510C	EPA 8082	W					1.0	A
	EPA 3550	EPA 8082	S					33	A
TOXAPHENE	EPA 3510C	EPA 8081A	W					3.0	A
	EPA 3550	EPA 8081A	S					100	A

Table 5.1
QA Targets for Accuracy, Precision and MDL's/PQL's

Nitrogen Pesticides (NPD)

Analyte	Prep Method	Analysis Method	Matrix	Spike Accuracy Range (%)	LCS Accuracy Range (%)	Precision % RPD	MDL (ppb)	PQL (ppb)	Clean-Up Code
ALACHLOR	EPA 3510C	EPA 619	W				1.6	15	A
	EPA 3550	EPA 619	S					500	A
AMETRYN	EPA 3510C	EPA 619	W				1.1	4.5	A
	EPA 3550	EPA 619	S					150	A
ATRAZINE	EPA 3510C	EPA 619	W				1.1	4.5	A
	EPA 3550	EPA 619	S					150	A
BROMACIL	EPA 3510C	EPA 619	W				4.6	15	A
	EPA 3550	EPA 619	S					500	A
BUTACHLOR	EPA 3510C	EPA 619	W					15	A
	EPA 3550	EPA 619	S					500	A
BUTYLATE	EPA 3510C	EPA 619	W				0.56*	4.5	A
	EPA 3550	EPA 619	S					150	A
CARBOXIN	EPA 3510C	EPA 619	W				3.9	15	A
	EPA 3550	EPA 619	S					500	A
CHLORPROPHAM	EPA 3510C	EPA 619	W				2.6	15	A
	EPA 3550	EPA 619	S					500	A
CHLORPYRIFOS	EPA 3510C	EPA 619	W				0.56	1.5	A
	EPA 3550	EPA 619	S					50	A
CYANAZINE	EPA 3510C	EPA 619	W					15	A
	EPA 3550	EPA 619	S					500	A
CYCLOATE	EPA 3510C	EPA 619	W				.96	4.5	A
	EPA 3550	EPA 619	S					150	A
DIAZINON	EPA 3510C	EPA 619	W				11*	15	A
	EPA 3550	EPA 619	S					500	A
DIPHENAMID	EPA 3510C	EPA 619	W				1.8	15	A
	EPA 3550	EPA 619	S					500	A
EPTC (EPTAM)	EPA 3510C	EPA 619	W				0.45	4.5	A
	EPA 3550	EPA 619	S					150	A
FENAMIPHOS	EPA 3510C	EPA 619	W				4.0	15	A
	EPA 3550	EPA 619	S					500	A
HEXAZINONE	EPA 3510C	EPA 619	W				7.3*	15	A
	EPA 3550	EPA 619	S					500	A
METOLACHLOR	EPA 3510C	EPA 619	W					15	A
	EPA 3550	EPA 619	S					500	A
METRIBUZIN	EPA 3510C	EPA 619	W				2.1	15	A
	EPA 3550	EPA 619	S					500	A
MGK 264	EPA 3510C	EPA 619	W					30	A

Table 5.1
QA Targets for Accuracy, Precision and MDL's/PQL's

Nitrogen Pesticides (NPD)

Analyte	Prep Method	Analysis Method	Matrix	Spike Accuracy Range (%)	LCS Accuracy Range (%)	Precision % RPD	MDL (ppb)	PQL (ppb)	Clean-Up Code
	EPA 3550	EPA 619	S					1000	A
MOLINATE	EPA 3510C	EPA 619	W				0.40*	4.5	A
	EPA 3550	EPA 619	S					150	A
NAPROPAMIDE	EPA 3510C	EPA 619	W					15	A
	EPA 3550	EPA 619	S					500	A
NORFLURAZON	EPA 3510C	EPA 619	W				2.3	15	A
	EPA 3550	EPA 619	S					500	A
PEBULATE	EPA 3510C	EPA 619	W				0.54	4.5	A
	EPA 3550	EPA 619	S					150	A
PROMETON	EPA 3510C	EPA 619	W				0.62	4.5	A
	EPA 3550	EPA 619	S					150	A
PROMETRYN	EPA 3510C	EPA 619	W				1.5	4.5	A
	EPA 3550	EPA 619	S					150	A
PRONAMIDE	EPA 3510C	EPA 619	W					15	A
	EPA 3550	EPA 619	S					500	A
PROPAZINE	EPA 3510C	EPA 619	W				0.56	4.5	A
	EPA 3550	EPA 619	S					150	A
SIMAZINE	EPA 3510C	EPA 619	W	63-143	63-143	19	0.96	4.5	A
	EPA 3550	EPA 619	S	50-126	50-126	30		150	A
SIMETRYN	EPA 3510C	EPA 619	W				0.72	4.5	A
	EPA 3550	EPA 619	S					150	A
TEBUTHIURON	EPA 3510C	EPA 619	W				4.0	15	A
	EPA 3550	EPA 619	S					500	A
TERBACIL	EPA 3510C	EPA 619	W				10	30	A
	EPA 3550	EPA 619	S					1000	A
TERBUFOS	EPA 3510C	EPA 619	W					15	A
	EPA 3550	EPA 619	S					500	A
TERBUTRYN	EPA 3510C	EPA 619	W				1.1	4.5	A
	EPA 3550	EPA 619	S					150	A
VERNOLATE	EPA 3510C	EPA 619	W	65-141	65-141	20	1.1*	4.5	A
	EPA 3550	EPA 619	S	68-124	68-124	30		150	A
*Interim values.									

Table 5.1
QA Targets for Accuracy, Precision and MDLs/PQLs

Organophosphorous Pesticides (FPD)

Analyte	Prep Method	Analysis Method	Matrix	Spike Accuracy Range (%)	LCS Accuracy Range (%)	Precision % RPD	MDL (ppb)	PQL (ppb)	Clean-Up Code
CARBOPHENOTHION	EPA 3510C	EPA 8141A	W				.30	.80	A
	EPA 3550	EPA 8141A	S				2.9	27	A
CHLORPYRIFOS	EPA 3510C	EPA 8141A	W				.16	.40	A
	EPA 3550	EPA 8141A	S				2.0	13	A
DEF	EPA 3510C	EPA 8141A	W				.33	.40	A
	EPA 3550	EPA 8141A	S				3.0	13	A
DEMETON	EPA 3510C	EPA 8141A	W				NE	.80	A
	EPA 3550	EPA 8141A	S				NE	27	A
DIAZINON	EPA 3510C	EPA 8141A	W	51-124	51-124	≤25	.25	.40	A
	EPA 3550	EPA 8141A	S	34-153	34-153	≤30	1.8	13	A
DICHLORVOS	EPA 3510C	EPA 8141A	W				.85	2.1	A
	EPA 3550	EPA 8141A	S				7.1	13	A
DIMETHOATE	EPA 3510C	EPA 8141A	W				.24	.40	A
	EPA 3550	EPA 8141A	S				2.0	13	A
DISULFOTON	EPA 3510C	EPA 8141A	W				NE	.80	A
	EPA 3550	EPA 8141A	S				NE	27	A
DISULFOTON SULFONE	EPA 3510C	EPA 8141A	W				NE	1.0	A
	EPA 3550	EPA 8141A	S				NE	33	A
DISULFOTON SULFOXIDE	EPA 3510C	EPA 8141A	W				NE	NE	A
	EPA 3550	EPA 8141A	S				NE	NE	A
EPN	EPA 3510C	EPA 8141A	W	42-133	42-133	≤38	.22	.40	A
	EPA 3550	EPA 8141A	S	72-117	72-117	≤30	1.8	13	A
ETHION	EPA 3510C	EPA 8141A	W				.17	.40	A
	EPA 3550	EPA 8141A	S				2.5	13	A
ETHOPROP	EPA 3510C	EPA 8141A	W				.29	.40	A
	EPA 3550	EPA 8141A	S				1.2	13	A
FENTHION	EPA 3510C	EPA 8141A	W	18-140	18-140	≤30	.40	.40	A
	EPA 3550	EPA 8141A	S	61-110	61-110	≤30	1.1	13	A
FENSULFOTHION	EPA 3510C	EPA 8141A	W				2.0	2.2	A
	EPA 3550	EPA 8141A	S				5.5	16	A
FOLEX (MERPHOS, TRIBUFOS)	EPA 3510C	EPA 8141A	W				NA	NA	A
	EPA 3550	EPA 8141A	S				NA	NA	A
MEVINPHOS	EPA 3510C	EPA 8141A	W				.42	.40	A
	EPA 3550	EPA 8141A	S				3.7	13	A
MONOCROTOPHOS	EPA 3510C	EPA 8141A	W				NE	1.0	A
	EPA 3550	EPA 8141A	S				NE	33	A

Table 5.1
QA Targets for Accuracy, Precision and MDLs/PQLs

Organophosphorous Pesticides (FPD)

Analyte	Prep Method	Analysis Method	Matrix	Spike Accuracy Range (%)	LCS Accuracy Range (%)	Precision % RPD	MDL (ppb)	PQL (ppb)	Clean-Up Code
NALED	EPA 3510C	EPA 8141A	W				1.02	2.7	A
	EPA 3550	EPA 8141A	S				11.4	NE	A
ETHYL PARATHION	EPA 3510C	EPA 8141A	W				.28	.40	A
	EPA 3550	EPA 8141A	S				2.9	13	A
METHYL PARATHION	EPA 3510C	EPA 8141A	W				.16	.40	A
	EPA 3550	EPA 8141A	S				1.5	13	A
PHORATE	EPA 3510C	EPA 8141A	W				NE	.40	A
	EPA 3550	EPA 8141A	S				NE	13	A
RONNEL	EPA 3510C	EPA 8141A	W				.17	.40	A
	EPA 3550	EPA 8141A	S				1.6	13	A
SULFOTEPP	EPA 3510C	EPA 8141A	W				NE	.40	A
	EPA 3550	EPA 8141A	S				NE	13	A
TERBUFOS	EPA 3510C	EPA 8141A	W				NE	.40	A
	EPA 3550	EPA 8141A	S				NE	13	A

Table 5.2 Key to Clean-up Procedures

Code	Definition
A	Not applicable
B	Clean up included in preparation or analysis method
C	EPA 3660A (Sulfur clean-up)
D	EPA 3620 (Florisil clean-up)
E	EPA 3665 (Sulfuric Acid clean-up)

6.0 Sampling Procedures

The DWQ Laboratory Section does not provide field-sampling services. The laboratory's responsibility in the sample collection process lies in supplying the sampler with proper containers and preservatives. The Laboratory Section welcomes consultation with collectors for whatever assistance can be provided.

6.1 Sampling Containers

6.1.1 Sampling Container Sources

The Laboratory Section offers pre-cleaned sampling containers for use by laboratory field sampling personnel. Some sampling containers are purchased from reputable manufacturers and are certified as cleaned according to EPA specifications (*Specifications and Guidance for Contaminant-Free Sample Containers*, OSWER Directive #9240.0-05A, December, 1992). When commercial pre-cleaned containers are not available, the procedures outlined in Table 8-1 are followed.

The sources for all bottles are:

(a) Carolina Food (Shelburne Plastics) - variety of inorganic analyses
(b) Eagle Picher - VOA
(c) Nalge® - BOD, Coliform, Chlorophyll, Cyanide
(d) Qorpak® - Total Phenol
(e) QEC® - Pesticides, SVOA, Oil & Grease (sometimes I-chem) sediments

6.1.2 Types of Bottles:

The types of bottles utilized are:

(a) 500 mL disposable plastic bottles
(b) 1000 mL, 250 mL plastic bottles
(c) 4000 mL amber glass bottles with Teflon-lined caps
(d) 125 mL, 250 mL, 1000 mL glass jar with Teflon-lined caps
(e) 40 mL clear and amber VOC vial with Teflon/silicon septum
(f) 1000 mL brown plastic bottles
(g) 1000 mL wide-mouth glass bottles with Teflon-lined caps

6.2 Preservatives

Upon request, preservatives are provided to field sampling personnel in bottles or in sealed pre-scored ampoules. In some cases, preservatives supplied directly from a private vendor are drop-shipped to the regional offices. The sodium thiosulfate preservative for coliform samples is supplied by the Central Laboratory support personnel in pre-cleaned, sterilized sampling containers. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are, at a minimum:

- Nitric Acid - ACS grade or equivalent
- Sodium Bisulfate - ACS grade or equivalent
- Sodium Hydroxide - ACS grade or equivalent
- Sulfuric Acid - ACS grade or equivalent
- Sodium Thiosulfate - ACS grade or equivalent
- Ascorbic Acid - ACS grade or equivalent
- Zinc Acetate - ACS grade or equivalent
- Phosphoric Acid - ACS grade or equivalent
- Ferrous Ammonium Sulfate - ACS grade or equivalent

The Laboratory Section also provides the following supplies used during sample collection activities:

- Security seals
- Total residual chlorine test strips
- Wide range pH test strips
- Narrow range pH test strips

6.3 Reuse of Bottles and Bottle Cleaning

Only 1000 mL plastic amber bottles, 1000 mL glass bottles, 500 mL plastic bottles, and 250 mL plastic bottles are cleaned and reused. In order to certify that the re-used containers are clean, random bottles are periodically analyzed for the target constituent when controls are not built into the analytical process. An outline of the cleaning procedures can be found in Table 8-1. The cleaned bottles are stored in the Sample Shipping/Receiving area (G-098) of the laboratory away from laboratory activities.

6.4 Sampling Containers, Preservatives and Holding Times

The sampling container types, preservation techniques and holding times for the parameters analyzed by the laboratory are summarized in Tables 6.1 (Water Quality) and 6.2 (Groundwater). These tables are adapted from *40 CFR, Chapter 1, Part 136, Table II*. Special attention should be paid to the footnotes for any deviations. The information for soil/sediment samples is adapted from *Test Methods for Evaluating Solid Waste, SW-846, Revision III*. Tissue samples are collected, filleted and frozen in metal tins prior to submission to the laboratory. Tissue samples are frozen indefinitely until preparation and analysis.

If the container, preservative or holding time requirements are not met for a sample, the sample may be rejected by the laboratory or the reports will be qualified using a data qualifier code and accompanied by a Sample Condition Upon Receipt (SCUR) report or Sample Anomaly Report (SAR). If criteria are not specified in a source document, internal DWQ Laboratory Section guidelines have been set and are footnoted in the tables.

"Analyze immediately" is an EPA designation reserved for tests which, for compliance monitoring projects, should be performed by field instrumentation or a laboratory " within 15 minutes or less" of sampling. The Laboratory Section does not perform these analyses.

6.4.1 Definition of Holding Time

The date and time of sampling documented on the field sheet establishes the date and time zero. For composite samples, the date and time the compositing cycle ended establishes the date and time zero. When the maximum allowable holding time is expressed in days, the holding time is based on day measured. Holding times, expressed in 72 hours or less, are measured from date and time zero. The first day of holding time ends twenty-four hours after sampling. Holding times for analyses include preparation, quantitation and any necessary reanalysis.

6.4.1.1 SVOA/Pesticides

Holding times for sample preparation for semi-volatile organics are measured from the date and time of sampling until the solvent contacts the sample. If a sample is to be extracted on the day of expiration, the actual time of extraction must be recorded on the sample preparation worksheet. Holding times for analysis are measured from the date and time of initiation of extraction to the time of injection into the gas chromatograph.

6.4.1.2 VOA

Holding times for volatile organics are measured from the date and time of sampling to the date and time of injection into the gas chromatograph. The time of initiation of purging is considered the injection time, but data systems record the start of the chromatographic run rather than the start of purging. Hence, if a sample is so near expiration that the start-of-purging time rather than the chromatographic run time is needed to document the integrity of the sample, the analyst must record the start-of-purging time in the instrument log.

6.4.1.3 Inorganics and Metals

For inorganics and metals analysis, with the exception noted below, the preparation/digestion/distillation must be started in time to allow the analysis step to be initiated as documented in the instrument log, instrument output, or analysis worksheet, within the maximum allowable holding time as measured from the sampling date and time.

6.4.1.4 Microbiologicals

For microbiological analyses such as coliform and BOD, the holding time is measured from the date and time of sampling to the date and time when incubation begins.

6.5 Scheduling Laboratory Capacity

Major sampling events must be scheduled with the laboratory prior to formal acceptance of the samples by the laboratory. Samples are accepted for analysis by logging them into the Sample Tracking and Reporting Laboratory Information Management System (DWQ STAR LIMS) and assigning tests. The Branch Manager or Supervisor is responsible for scheduling samples by assessing the capacity and previously scheduled workload of the laboratory and makes decisions regarding work assignments whenever laboratory capacity for any work group may be exceeded.

6.6 Processing Time-Sensitive Samples

With increased emphasis on emergency and enforcement actions, it is imperative that the laboratories meet all holding and incubation times. In order to properly process samples and keep overtime to a minimum, the Central Laboratory has established the following times for receipt of samples:

1. Samples for BOD, PO_4, Metals (Groundwater), Turbidity, Color, MBAS, and MF coliform analysis will not be accepted after 1:00 pm on Fridays and workdays that immediately precede a holiday. Employees planning to submit more than six samples on these days for these parameters should contact the laboratory to schedule the samples in advance.

2. MF coliform samples expected to meet the required six-hour holding time will not be accepted after 3:00 pm on normal Monday through Thursday workdays. Employees planning to submit more than five coliform samples in this category should contact the Bio/Chemistry Unit Supervisor to schedule the sample in advance.

3. All Tube coliform samples must be scheduled in advance by contacting the Bio/Chemistry Unit Supervisor.

4. Chlorophyll samples should not be submitted after 3:00 PM on any day since these must be filtered on the day of receipt, regardless of holding time.

5. Unpreserved samples for individual analysis of nitrate or nitrite should be scheduled with Cindy Green or the Nutrients group prior to submittal. With 48-hour hold times for these samples, analytical runs need to be specially scheduled to accommodate these samples. A concurrent preserved nutrients sample should be submitted for which nitrate+nitrite analysis has been requested.

The Asheville and Washington Regional Laboratories have similar policies, but tend to communicate daily with their samplers so allowances are often made based upon verbal agreements. The Asheville Regional laboratory has established the following policies for receipt of samples:

1. Samples for BOD, Turbidity, MF coliform analysis will not be accepted after 3:00 PM on Fridays or workdays that immediately precede a holiday. Staff planning to submit more than three samples on these days for these parameters should contact the laboratory to schedule these samples in advance.

2. Coliform bacteria samples expected to meet the required six-hour holding time will not be accepted after 4:00 PM on normal Monday through Thursday workdays.

3. All tube coliform samples must be scheduled in advance by contacting the laboratory.

The Washington Regional laboratory has established the following policies for receipt of samples:

1. Samples for BOD, Turbidity, MF coliform analysis will not be accepted after 3:00 PM on Fridays or workdays that immediately precede a holiday. Staff planning to submit more than three samples on these days for these parameters should contact the laboratory to schedule these samples in advance.

2. Coliform bacteria samples expected to meet the required six-hour holding time will not be accepted after 4:00 PM on normal Monday through Thursday workdays.

Figure 6.1 Required Containers, Preservation Techniques and Holding Times (Water Quality Samples)

COLLECTION AND PRESERVATION OF **WATER QUALITY** SAMPLES FOR THE NC DWQ LABORATORY SECTION
Reference: 40 CFR Part 136.3 Table II

Listed below is information on the collection and preservation of samples. The amount of sample listed is for average conditions; therefore, if you suspect that unusual conditions or interferences exist, please submit double the amount of sample. **Excluding purgeable organics and sulfide**, a one-half inch air space should be left in all bottles to allow for mixing before analysis. The parameters are listed in the same order as they appear on the DM-1 form.

Samples must be shipped to the Laboratory as soon as possible after collection.

Parameter [1]	Minimum Required Volume	Container [14] P-Plastic G-Glass	Preservation[25]	Maximum Holding Time[26]
A. BOD 5-day	1 liter	P	Cool, 4°C	48 hours[2]
COD[19]	200 ml	P	Cool, 4°C, 25% H_2SO_4 to pH<2	28 days
Coliform (Total,Fecal, E.coli and Enterococci)	250 ml **each**	P[3] (sterile)	Cool, 4°C, 0.008% $Na_2S_2O_3$[3]	6 hours[4]
Residue[19] (TSS, TDS, TS)	500 ml **each**	P	Cool, 4°C	7 days
A. pH[5]	Inappropriate for laboratory analysis.			Immediate – field measurement
A. Acidity[19]	200 ml	P	Cool, 4°C	14 days
A. Alkalinity[19]	200 ml	P	Cool, 4°C	14 days
TOC	200 ml	P	Cool, 4°C, conc. H_3PO_4 to pH<2	28 days
Turbidity	200 ml	P	Cool, 4°C	48 hours[2]
Chloride	200 ml	P	None required	28 days
Chlorophyll *a*[11]	500 ml	P (Brown)	14 days in dark[13] Cool, 4°C	14 days[13]
Color	200 ml	P	Cool, 4°C	48 hours[2]
Chromium, Hexavalent	200 ml	P (Disposable)	Cool, 4°C	24 hours
Cyanide, Total	2 liters (two 1 liter bottles)	P	Cool, 4°C, 0.6g ascorbic acid[6] 6N NaOH to pH>12	14 days[23]
Fluoride	500 ml	P	None required	28 days
Formaldehyde	500 ml	P (Disposable)	Cool, 4°C	NA
Oil & Grease	2 liters (two 1 liter bottles)[18]	G (Wide mouth quart jar, Teflon-lined cap)	Cool, 4°C, 1:1 H_2SO_4 to pH<2	28 days
Hardness, Total (Request by checking Ca and Mg on fieldsheet - can be included as part of metals sample)[27]	500 ml	P (Disposable)	1+1 HNO_3 to pH<2	6 months
MBAS	500 ml	P	Cool, 4°C	48 hours[2]
Phenols	2 liters (two 1 liter bottles)	G (Phenol Bottle) only	Cool, 4°C, 1:1 H_2SO_4 to pH <2 (1 ml FAS if sample contains oxidizer)	28 days
Sulfate	500 ml	P	Cool, 4°C	28 days
Sulfide	500 ml [10]	P	Cool, 4°C, add 1 ml of 2N zinc acetate plus 6N NaOH to pH>9, no headspace.	7 days
Specific Conductance	200 ml	P	Cool, 4°C	28 days
B. NH_3 as N	500 ml x 1	P(Disposable)	Cool, 4°C, 25% H_2SO_4 to pH<2[7] 0.008%$Na_2S_2O_3$ [12]	28 days
B. TKN as N	Combined with above	P(Disposable)	Cool, 4°C, 25% H_2SO_4 to pH<2[7] 0.008%$Na_2S_2O_3$ [12]	28 days
B. NO_2 + NO_3 as N	Combined with above	P(Disposable)	Cool, 4°C, 25% H_2SO_4 to pH<2[7]	28 days
B. P, Total as P	Combined with above	P(Disposable)	Cool, 4°C, 25% H_2SO_4 to pH<2[7]	28 days
P, Dissolved as P	200 ml	P(Disposable)	Filter immediately; Cool, 4°C, 25% H_2SO_4 to pH<2[7]	28 days
PO_4 as P	200 ml	P(Disposable)	Filter immediately; Cool, 4°C	48 hours[2]

C. Metals[24]: Ag , Al, As, Be, Ba, Ca, Cd, Co, Cr (Total), Cu, Fe, K, Li, Mg, Mn, Na, Ni, Pb, Sb, Sn, Se, Tl, V, Zn and Hg	500 ml x 1	P(Disposable)	1+1 HNO_3 to pH<2	6 months. (**28 days for Mercury**)
Semi Volatile Organics (B/NA extractables)	1 gal	G (amber), Teflon-lined cap	Cool, 4°C, 0.008%$Na_2S_2O_3$[12]	7 days until extraction[8] 40 days after extraction
Pesticides/PCB's (OP pest/ OCl pest/ON pest)	1 gal	G (amber), Teflon-lined cap	Cool, 4°C, 0.008%$Na_2S_2O_3$[12]	7 days until extraction[8] [16], 40 days after extraction
Acid Herbicides	1 gal	G (amber), Teflon-lined cap	Cool, 4°C, 0.008%$Na_2S_2O_3$[12]	7 days until extraction[8] [16], 40 days after extraction
Purgeable Organics (VOA)	40 ml x 3 [10]	G, Teflon-lined septum	Cool, 4°C, 0.6g Ascorbic Acid [12][13] Sodium Bisulfate ($NaHSO_4$) to pH<2 [13] [15][17]	14 days (7 days when sample is unpreserved and aromatics only requested)
TPH - GRO and BTEX (aq)	40 ml x 3 [10]	G, Teflon-lined septum	Cool, 4°C, 0.6g Ascorbic Acid [12][13] Sodium Bisulfate ($NaHSO_4$) to pH<2 [13] [15][17]	14 days
TPH - DRO (aq)	1 gal	G, Teflon-lined cap	Cool, 4°C	14 days, analyze extract within 40 days

SOIL SAMPLES

****WHEN SUBMITTING SOIL AND SLUDGE SAMPLES FOR ANALYSIS, A SEPARATE SAMPLE CONTAINER MUST BE COLLECTED FOR EACH OF THE ANALYTICAL GROUPS LISTED BELOW:**

Adopted from Tables 3-1 and 4-1 in *Test Methods for Evaluating Solid Waste*, SW-846, Third Edition, 1986 and First Update in 1987.

Parameter/Analytical Group [1]	Minimum Required Volume	Container [14] P-Plastic G-Glass	(F) Filtered (U) Unfiltered	Preservation [25]	Maximum Holding Time[26]
Wet Chemistry analyses[20]	8 oz jar	G, Teflon-lined cap	N/A	Cool, 4°C	refer to aqueous
Nutrient analyses[21]	8 oz jar	G	N/A	Cool, 4°C	refer to aqueous
Micro analyses[22]	8 oz jar	G	N/A	Cool, 4°C	refer to aqueous
Metals analysis	8 oz jar	G, Teflon-lined cap	N/A	Cool, 4°C	refer to aqueous
Pesticides/PCB's (OP/OCl/ONpesticides)	8 oz jar	G, Teflon-lined cap	N/A	Cool, 4°C	14 days to extract; analyze w/in 40 days
Acid Herbicides	8 oz jar	G, Teflon-lined cap	N/A	Cool, 4°C	14 days to extract; analyze w/in 40 days
Semi Volatile Organics (B/NA extractables)	8 oz jar	G, Teflon-lined cap	N/A	Cool, 4°C	14 days to extract; analyze w/in 40 days
Purgeable Organics (SVOA)	4 oz jar	G, Teflon-lined cap or septum	N/A	Cool, 4°C	14 days
TPH Gas Range (soil)	4 oz jar	G, Teflon-lined cap or septum	N/A	Cool, 4°C	14 days
TPH Diesel Range (soil)	8 oz jar	G, Teflon-lined cap	N/A	Cool, 4°C	14 days to extract; Analyze w/in 40 days

Footnotes:

(1)Determinations preceded by the same letter may be submitted in the same bottle if the bottle contains enough sample. If no letter precedes a parameter, it must be submitted in a separate bottle (i.e. A, B, C).

(2)48 hours is the maximum holding time, however, samples should be submitted to the Lab as soon as possible.

(3)Use the 250 ml wide-mouth sterile plastic bottles for all samples. All bottles contain sodium thiosulfate and EDTA reagents.

(4)Litigation samples should be delivered to the laboratory within 5 hours of sample collection.

(5)It is recommended that pH analysis be performed on site. “Immediately” is defined as within fifteen minutes after collection.

(6)Add 0.6 g of ascorbic acid only if sample contains residual chlorine.

(7)Caution: Addition of excessive amounts of acid will interfere with the test procedures. The 2.0 ml of 25% H_2SO_4 per 500 ml sample should be added using a graduated or precise volume dispensing device. If no dispenser is available you may add exactly 40 drops of the 25% H_2SO_4. In most cases, the addition of 2.0 ml (40 drops) of 25% H_2SO_4 to 500 ml of surface water will reduce the pH to <2; however, if the pH remains above 2, add acid dropwise with stirring until the pH is lowered to <2.

(8)In a glass container, submit a small quantity of the pure compound of any suspected material.

(9)If residual chlorine is present, add 0.008% sodium thiosulfate dropwise to just neutralize the chlorine.

(10)Fill the bottle to overflowing and cap, leaving no air space.

(11)EPA Method 445.0, Revision 1.2, September 1997.

(12)Should only be used in the presence of residual chlorine. Add sodium thiosulfate or ascorbic acid (as directed) to the container first; fill at least half way before adding acid.

(13)Used by the DWQ Chemistry Lab only at this time.

(14)The container types listed are those commonly used throughout the Division. Other container types may be acceptable. Please consult the laboratory about use of proper containers before deviating from those listed.

(15)Samples submitted for purgeable halocarbons only should not be acid-preserved.

(16)Samples submitted for pesticide and acid herbicide analyses must be extracted within 72 hours of collection if the pH is not adjusted in the lab to a pH range of 5-9.

(17)Samples submitted for purgeable aromatics receiving no pH adjustment must be analyzed within 7 days of collection.

(18)The entire contents must be used for analysis.

(19)COD, TS, TSS, Alkalinity and Acidity samples are to be shipped to the Central laboratory. They will then be repacked and routed to the Washington Regional laboratory for analysis. Samples collected in the Washington Region are sent directly to the Washington regional laboratory.

(20)Wet chemistry parameters include: Chloride, Chlorophyll *a*, Color, Hexavalent Chromium, Total Cyanide, Fluoride, Formaldehyde, Grease & Oils, MBAS, Phenols, Sulfate, Sulfide, Specific Conductance, and TDS.

(21)Nutrient parameters include: Ammonia Nitrogen, TKN, Nitrate+Nitrite Nitrogen, Total Phosphorous, Dissolved Phosphorous and Orthophosphate.

(22)Microbiology parameters include: BOD 5-day, Coliform (Total, Fecal, E. coli and Enterococci), Residue, pH, Acidity, Alkalinity, TOC, and Turbidity.

(23)Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be spot tested with lead acetate paper before pH adjustment in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of $CdNO_3$ powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH>12.

(24)For dissolved metals, samples should be filtered immediately on-site before adding preservative.

(25)Sample preservation should be performed immediately upon collection. For composite samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then the samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

(26)Samples should be analyzed as soon as possible after collection. The times listed are the MAXIMUM times that samples may be held before analysis and still be considered valid. Sample collectors must allow for preparation and analytical start-up. Some samples may not be stable for the maximum time period given in the table. Collectors are obligated to hold the sample for as short a time as possible especially if knowledge exists which shows this is necessary to maintain sample stability.

(27)Total Hardness = 2.497[Ca] + 4.118 [Mg].

Figure 6.2 Required Containers, Preservation Techniques and Holding Times (Groundwater Samples)

COLLECTION AND PRESERVATION OF **GROUNDWATER** SAMPLES FOR THE NC DWQ LABORATORY SECTION

Listed below is information to be used in the collection and preservation of samples. Filtered samples are requested for some parameters as recommended by the USGS manual. **If you are submitting filtered samples, write "DIS" (for dissolved) in the block beside applicable parameters on the GS-54 form. Excluding purgeable organics and sulfide**, a one-half inch air space should be left in all bottles to allow for mixing before analysis. The parameters have been grouped according to preservatives needed. **Samples must be shipped to the Laboratory as soon as possible after collection.**

Parameter [2]	Minimum Required Volume	Container [1][14] P-Plastic G-Glass	(F) Filtered (U) Unfiltered	Preservation[22]	Maximum Holding Time[23]
A. Alkalinity[18]	200 ml	P	U	Cool, 4°C	14 days
A. Carbonate	Request on fieldsheet and submit Alkalinity sample.				6 months
A. Bicarbonate	Request on fieldsheet and submit Alkalinity sample.				6 months
A. pH	Inappropriate for laboratory analysis.				Immediate - field measurement
A. Carbon Dioxide	Inappropriate for laboratory analysis.				Immediate - field measurement
Chromium, hexavalent	200 ml	P	U	Cool, 4°C	24 hours
Color	200 ml	P	U	Cool, 4°C	48 hours[6]
MBAS	500 ml	P	U	Cool, 4°C	48 hours[6]
Specific Conductance	200 ml	P	U	Cool, 4°C	28 days
Chloride	200 ml	P	U	None required	28 days
Fluoride	500 ml	P	U	None required	28 days
Hardness, Total (Request by checking Ca and Mg on fieldsheet - can be part of metals sample) Total Hardness = 2.497[Ca]+4.118[Mg]	500 ml	P	U	HNO_3 to pH<2, H_2SO_4 to pH<2	6 months
Hardness, Non-carbonate[3]	Submit samples for Total Hardness (Ca+Mg) and Alkalinity as specified.				6 months
Oil & Grease	2 liters (two 1 liter bottles)	G (wide-mouth quart jar, Teflon-lined cap)	U	Cool, 4°C, 1:1 H_2SO_4 to pH<2	28 days
Silica	200 ml	P	U	Cool, 4°C	28 days
Sulfate	200 ml	P	U	Cool, 4°C	28 days
Cyanide	2 liters (two 1 liter bottles)	P	U	Cool, 4°C, 0.6g ascorbic acid[4], 6N NaOH to pH>12	14 days[12]
Phenol	2 liters (two 1 liter bottles)	G only	U	Cool, 4°C, 1:1 H_2SO_4 to pH<2 (1 ml FAS is sample contains oxidizer)	28 days
B. *Metals:* Ag, Al, As, Ba, Be, Ca, Cd, Co, Cr (Total), Cu, Fe, Li, K, Mg, Mn, Na, Ni, Pb, Sb, Sn, Se, Tl, V, Zn and Hg	500 ml	P (Disposable)	U	1+1 HNO_3 to pH<2	72 hours (**should be delivered to lab within 48 hours to allow for sample prep**.) 28 days - Hg
BOD	1 liter	P	U	Cool, 4°C	48 hours[6]
COD[18]	200 ml	P	U	Cool, 4°C, 25% H_2SO_4 to pH<2	28 days
Coliform (Fecal or Total)	250 ml **each**	P (sterile)[7]	U	Cool, 4°C, 0.008% $Na_2S_2O_3$[7]	6 hours
TOC	200 ml	P	U	Cool, 4°C, H_3PO_4 to pH<2	28 days
Turbidity	200 ml	P	U	Cool, 4°C	To lab in <48 hours[6]
C. NH_3 as N	500 ml	P (Disposable)	U	Cool, 4°C, 25% H_2SO_4 to pH<2[11]	28 days
C. TKN as N	Combined w/ above	P (Disposable)	U	Cool, 4°C, 25% H_2SO_4 to pH<2[11]	28 days
C. NO_3 + NO_2 as N	Combined w/ above	P (Disposable)	U	Cool, 4°C, 25% H_2SO_4 to pH<2[11]	28 days
C. Total Phosphorous as P	Combined w/	P	U	Cool, 4°C, 25%	28 days

	above	(Disposable)		H_2SO_4 to pH<2[11]	
Dissolved Phosphorous as P	200 ml	P (Disposable)	F	Cool, 4°C, 25% H_2SO_4 to pH<2[11]	28 days
Orthophosphate as P	200 ml	P (Disposable)	F	Filter immediately, Cool, 4°C	48 hours[6]
Residue (TSS, TDS, TS)[18]	500 ml **each**	P	U	Cool, 4°C	7 days
Semi Volatile Organics (B/NA extractables)	1 gal	G (amber), Teflon-lined cap	U	Cool, 4°C	7 days until extraction 40 days after extraction
Pesticides/PCB's (OP/OCl/ON pest)	1 gal	G (amber), Teflon-lined cap	U	Cool, 4°C	7 days until extraction[16], 40 days after extraction
Acid Herbicides	1 gal	G (amber), Teflon-lined cap	U	Cool, 4°C	7 days until extraction[16], 40 days after extraction
Purgeable Organics(VOA)	40 ml x 3	G, Teflon-lined septum	U	Cool, 4°C, 0.6g ascorbic acid only if residual chlorine present, Sodium Bisulfate ($NaHSO_4$) to pH2 [13] [15][17] No headspace.	14 days (7days when unpreserved sample submitted for aromatics only)
TPH Gasoline Range (aq) and BTEX	40 ml x 3	G, Teflon-lined septum	U	Cool, 4°C, 0.6g ascorbic acid only if residual chlorine present, Sodium Bisulfate ($NaHSO_4$) to pH2 [13] [15][17] No headspace.	14 days
TPH Diesel Range (aq)	1 gal	G (amber), Teflon-lined cap	U	Cool, 4°C	14 days; analyze extract within 40 days

SOIL SAMPLES

****WHEN SUBMITTING SOIL AND SLUDGE SAMPLES FOR ANALYSIS, A SEPARATE SAMPLE CONTAINER MUST BE COLLECTED FOR EACH OF THE ANALYTICAL GROUPS LISTED BELOW:**

Adopted from Tables 3-1 and 4-1 in *Test Methods for Evaluating Solid Waste*, SW-846, Third Edition, 1986 and First Update in 1987.

Parameter [2]	Minimum Required Volume	Container [14] P-Plastic G-Glass	(F) Filtered (U) Unfiltered	Preservation [22]	Maximum Holding Time[23]
Wet Chemistry analyses[20]	8 oz jar	G, Teflon-lined cap	N/A	Cool, 4°C	refer to aqueous
Nutrient analyses[21]	8 oz jar	G	N/A	Cool, 4°C	refer to aqueous
Micro analyses[22]	8 oz jar	G	N/A	Cool, 4°C	refer to aqueous
Metals analysis	8 oz jar	G, Teflon-lined cap	N/A	Cool, 4°C	refer to aqueous
Pesticides/PCB's (OP/OCl/ON pest)	8 oz jar	G, Teflon-lined cap	N/A	Cool, 4°C	14 days to extract; analyze w/in 40 days
Acid Herbicides	8 oz jar	G, Teflon-lined cap	N/A	Cool, 4°C	14 days to extract; analyze w/in 40 days
Semi Volatile Organics (B/NA extractables)	8 oz jar	G, Teflon-lined cap	N/A	Cool, 4°C	14 days to extract; analyze w/in 40 days
Purgeable Organics(VOA)	4 oz jar	G, Teflon-lined cap or septum	N/A	Cool, 4°C	14 days
TPH Gas Range (soil)	4 oz jar	G, Teflon-lined cap or septum	N/A	Cool, 4°C	14 days
TPH Diesel Range (soil)	8 oz jar	G, Teflon-lined cap	N/A	Cool, 4°C	14 days to extract; Analyze w/in 40 days
Purgeable Organics (5035) High-level	EnCore Sampler x 2		N/A	Cool, 4°C	48 hours to extrusion into MeOH

References:

Analytical Procedures, Appendix D Leaking Underground Fuel Tank Field Manual: Guidelines for Site Assessment, Clean-up and Underground Storage Tank Closure; State of California State Water Resources Control Board, Sacramento, Ca., October 1989.

NPDES, Appendix A, Federal Register, 38, No. 75, Pt II

NOTE: All other organics will be analyzed using methods from the Federal Register (40 CFR, Part 136) when available. The Branch Supervisor must approve methods from any other source.

Footnotes:
(1)P-Plastic, G- Glass, P(Disposable) - Plastic disposable bottle.

(2)Parameters preceded by the same letter may be submitted in the same bottle if the bottle contains enough sample. If no letter precedes a parameter, it must be submitted in a separate bottle.

(3)When non-carbonate hardness is requested, samples for both metals (Ca+Mg) and alkalinity must be submitted.

(4)Add 0.6 g of ascorbic acid only if sample contains residual chlorine.

(5)Use one liter round glass bottles labeled phenol.

(6)48 hours is the maximum holding time; however, samples should be submitted to lab as soon as possible.

(7)Use the 250 ml wide-mouth sterile plastic bottles for all samples. All bottles contain sodium thiosulfate and EDTA reagents.

(8)Litigation samples should be delivered to the laboratory within 5 hours of sample collection.

(9)Caution: Addition of excessive amounts of acid will interfere with the test procedures. The 2.0 ml of 25% H_2SO_4 per 500 ml sample should be added using a graduated or precise volume dispensing device. If no dispenser is available, you may add exactly 40 drops of the 25% H_2SO_4. In most cases, the addition of 2.0 ml (40 drops) of 25% H_2SO_4 to 500 ml of groundwater will reduce the pH to <2; however, if the pH remains above 2, add acid dropwise with stirring until the pH is lowered to <2.

(10)In a glass container, submit a small quantity of the pure compound of any suspected material.

(11)If residual chlorine is present, add 0.008% sodium thiosulfate dropwise to just neutralize the chlorine.

(12)Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH>12.

(13)Used by the DWQ Chemistry Lab only at this time.

(14)The container types listed are those commonly used throughout the Division. Other container types may be acceptable. Please consult the laboratory about use of proper containers before deviating from those listed above.

(15)Samples submitted for purgeable halocarbons only should not be acid-preserved.

(16)Samples submitted for pesticide and acid herbicide analyses must be extracted within 72 hours of collection if the pH is not adjusted in the lab to a pH range of 5-9.

(17)Samples submitted for purgeable aromatics receiving no pH adjustment must be analyzed within 7 days of collection.

(18)COD, TS, TSS, Alkalinity and Acidity samples are to be shipped directly to the Washington Regional Office for analysis. Samples collected in the Washington Region are sent directly to the Washington regional laboratory.

(19)Wet chemistry parameters include: pH, Acidity, Alkalinity, Chloride, Chlorophyll a, Color, Hexavalent Chromium, Total Cyanide, Fluoride, Formaldehyde, Grease & Oils, MBAS, Phenols, Sulfate, Sulfide, Specific Conductance, and TDS.

(20)Nutrient parameters include: Ammonia Nitrogen, TKN, Nitrate+Nitrite Nitrogen, Total Phosphorous, Dissolved P and Orthophosphate.

(21)Microbiology parameters include: BOD 5-day, Coliform (Total, Fecal, E. coli and Enterococci), Residue, TOC, and Turbidity.

(22) Sample preservation should be performed immediately upon collection. For composite samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then the samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

(23) Samples should be analyzed as soon as possible after collection. The times listed are the MAXIMUM times that samples may be held before analysis and still be considered valid. Sample collectors must allow for preparation and analytical start-up. Some samples may not be stable for the maximum time period given in the table. Collectors are obligated to hold the sample for as short a time as possible especially if knowledge exists which shows this is necessary to maintain sample stability.

7.0 Sample Custody and Handling

Many of the errors in environmental analysis result from incorrect sample handling and lack of supporting documentation. Four factors that may ultimately affect the integrity of reported data include 1) obtaining a representative sample, 2) preventing contamination of the sample, 3) providing legal documentation of the sampling event, and 4) protecting the sample from chemical, physical or biological change prior to analysis.

7.1 Objective

The primary objective of sample custody is to maintain the integrity of samples and to generate documentation sufficient to trace a sample from its point of origin, through receipt in the laboratory, then analysis, reporting and disposal.

While the laboratory may not have control of field sampling activities, the laboratory has incorporated the following into its Quality Management Plan to ensure the validity of the laboratory's data.

- The laboratory has established procedures for the transportation, receipt, handling, protection, storage, retention and/or disposal of samples including all provisions necessary to protect the integrity of the sample and to protect the interests of the laboratory and the client. These procedures are communicated to Laboratory Section personnel and its clients in the *Sample Submission Guidance* document. This document is available for viewing on the Laboratory Section website at http://www.esb.enr.state.nc.us/lab/qa/sampsubguide.htm.

- The laboratory has adopted a system for identifying and tracking samples. This identification is retained throughout the life of the sample in the laboratory and ensures that samples cannot be confused physically or when referred to in records or other documents.

The sample management procedures used at the DWQ Laboratory Section are designed to ensure that sample integrity is maintained and documented. This documentation includes:

- Sample transmittal forms (fieldsheets and Chain-of-Custody)
- Sample preparation logs or worksheets
- Sample analysis logs or worksheets
- Calibration and quality control data associated with a sample set
- Instrument maintenance logs
- Sample disposal logs
- Final reports

7.2. Sample Custody Procedures

The Laboratory Section follows both routine and legal chain-of-custody (COC) procedures, depending on the requirements of the client submitting the samples. The DWQ Laboratory Section has adopted a policy of maintaining formal COC records on samples collected during enforcement or other investigations suspected to involve litigation. All samples processed by the Laboratory Section are kept discrete by assigning an individual laboratory number.

7.2.1. Routine Sample Custody

Samples are collected by field personnel utilizing procedures identified within their field SOPs or Quality Assurance Plans. The sample collection personnel must first consider the analyses to be performed so that proper shipping containers and sample containers with the appropriate preservatives are assembled. Holding times and field quality control measures must also be considered. All records required for the field personnel must complete documentation of field collection, including the pertinent data on sample labels/tags and applicable fieldsheets. Samples are packed so that they are segregated by site, sampling location, sample analysis type, sample priority or by final destination.

A sample transmittal form (also referred to as a fieldsheet) must accompany all samples that are submitted to the Laboratory Section. This record serves as a documented summary of the sample collection event and includes all records necessary to trace a sample from its point of origin through the final report. Each event or procedure to which the sample is subjected is recorded including sample collection, field preservation, sample receipt and sample log in. An example fieldsheet supplied by the Central Laboratory is given in Figure 7.1 (Water Quality Section fieldsheet). The minimum information required on these records include:

(a) a unique (for that date/time/collector) sample location/field ID combination.
(b) the date and time of sample collection (beginning and ending for composite samples)
(c) the collector's name
(d) the submitting entity (who to report to)
(e) the sample matrix
(f) the analyses requested
(g) sample priority
(h) method of shipment

If any of the above information is not present, an effort is made to reach the collector by phone. If the information cannot be obtained in a timely manner, the sample is subject to rejection. After collection, the samples are shipped to the laboratory by state courier, common carrier or are hand-delivered by the field staff.

Additional documentation recorded on the field sheets may include:

- Ambient field conditions
- Type of composite
- Temperature of samples in the field
- Field measurement data
- Field instrument calibration information

7.2.2. Legal Chain of Custody

Legal chain of custody is a special type of sample custody in which documentation is kept of all events (i.e., possession, transport, storage, and disposal) and time intervals associated with a specific sample. Legal chain of custody documentation includes chain-of-custody (COC) forms that have adequate space for dated, original signatures of all individuals who handled the samples, from the time of collection through laboratory receipt and distribution to the analytical unit. The custody of a sample is defined as one of the following:

(a) It is in the sampler's or transferee's actual possession;
(b) It is in the sampler's or transferee's view, after being in his/her physical possession;
(c) It was in the sampler's or transferee's physical possession and then he/she secured it or placed in a designated secure area to prevent tampering.

The purpose of the COC is to supply a detailed record of the sample description, collection information, and any transfer of custody from sample collection through sample receipt into the laboratory. The sample collector is responsible for the care and custody of the sample until properly dispatched to the analytical laboratory via State courier or turned over to a sample custodian or designee. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. Samples must be delivered to the laboratory as soon as possible after collection.

NOTE: The State couriers and independent couriers are not required to sign the COC form. The samples and COC are kept in the sealed sample cooler with the associated samples. The condition of the security seal is noted upon receipt at the lab. The freight bill from independent couriers is kept with the chain-of-custody documentation.

Chain of custody records are to be initiated by the sample collector and shall include the following information either by direct entry or by linkage to the fieldsheet.

- Time of day and calendar date of each transfer or handling procedure
- Signatures of transferors and transferees
- Location and security conditions of samples (when stored in the field)
- Storage conditions for sample including thermal preservation
- Unique lab ID for all samples
- Common carrier documents
- Sampling site description
- Date and time of sample collection
- Unique field ID code (optional)
- Collector's name
- Number of sample containers
- Requested analyses

Entries into all records must be written legibly and must be made with waterproof ink. All documentation entries shall be signed or initialed by responsible staff. Erasures or markings shall not obliterate entries in records. All corrections to record-keeping errors shall be made by one line marked through the error. The individual making the correction shall sign or initial and date the correction.

An example COC form is given in Figure 7.2 (Water Quality Section COC). A copy of this record is sent to the customer while the original is kept in the sample report file.

7.3 Sample Receipt Protocol

Sample acceptance, receipt, tracking and storage procedures are fully detailed in sample management SOPs. These procedures are summarized in the following sections.

The technicians at the Regional Laboratories and the Support Unit personnel at the Central Laboratory are responsible for receiving samples shipped from or delivered by field personnel that collect water, soil or tissue samples throughout the state. Laboratory staff receive deliveries of all samples and initiate the first in-house records for a sample. When samples arrive at the laboratory, staff inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the fieldsheet and COC (when applicable) and by visual checks of the container for possible damage or tampering. Any problems or anomalies are recorded on a Sample Condition Upon Receipt (SCUR) form and the sampler is notified. A course of action is determined and documented and the SCUR form is filed in the sample folder. A copy of this form is sent with the final report to the collector.

7.3.1 Procedure

Laboratory staff remove the samples from the container or cooler and organize the sample bottles according to sample location and fieldsheet. A sample may be composed of greater than one bottle since different preservatives, collection or handling techniques may be required to perform all analyses requested. Sample integrity and condition of all sample containers is verified for leakage, broken bottles, contaminated coolers, odors, etc.

Inspection of samples at the time of receipt include checking:

(a) Complete documentation to include sample identification, location, date and time of collection, collector's name, preservation type, sample type and any additional comments concerning the samples.

(b) Complete sample labels to include unique identification, preservation, analysis requested, date and time of collection and collector in indelible ink.

(c) Use of appropriate sample containers.

(d) Adherence to holding times as specified in the test method and/or summarized in Section 6.

(e) Adequate sample volume for the required analyses.

(f) Damage or signs of contamination to the sample container. Volatile organics vials and other volatile samples (e.g., sulfide) are also inspected for headspace by chemists in the Volatile Organics analytical unit.

(g) Checking and recording the temperature of samples that require thermal preservation.

Verification of chemical sample preservation as specified in 40 CFR Part 136 or the test method is performed prior to sample preparation or analysis in the analytical units after login and distribution and the process is documented on appropriate logs or laboratory bench worksheets.

At the time of receipt, laboratory staff check the temperature of the samples by measuring the temperature of the temperature blank. If there is no temperature blank present and if it does not compromise the integrity of the sample, the temperature of a representative sample is measured by pouring a small aliquot into a separate container, taking the temperature of this portion and then discarding it. Samples shall be deemed acceptable if arrival temperature is either within 0.1 to 6°C (with no evidence of freezing) or the method specific range. Samples that are hand-delivered immediately after collection may not be at the required temperatures; however, if there is evidence that the chilling process has begun, such as the arrival on ice or ice slurry and a downward trend in temperature is documented, the sample shall be considered acceptable. For samples with short transport times, samplers are asked to document a field temperature. Documentation of the actual sample temperature at the time of collection and upon receipt (and demonstrating a downward trend) will complete the preservation documentation requirements.

For COC samples, shipping documents are set aside and the shipping container examined, noting the presence and condition of any custody seals on the outside of the container before the sample is accepted for analysis. Any internal custody seals are then examined. Observations are recorded in the space provided on the COC form. The shipping container is opened fully and the sample custody documentation removed. If there is no COC or if it is improperly filled out, the deviation is documented on a SCUR form and chain of custody procedures are generally discontinued at this point. Carrier, freight bill or other tracking numbers in shipping documentation are recorded on and/or retained with the COC.

Any deviations from the checks above that question the suitability of the sample for analysis, or incomplete documentation of the tests required will be resolved by consultation with the sampler. If the sample acceptance criteria are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the sampler regarding the disposition of rejected samples, or

- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria. The condition of these samples shall be documented on the SCUR. The analytical results shall be appropriately qualified on the final report.

- Notify the customer of any non-conformance that may affect the integrity of the data. The samples may be rejected unless the client requests otherwise. Data from compromised samples is flagged with the appropriate data qualifier code(s) or comments and Sample Anomaly Report is issued with the report.

The custodian then assigns a chronological lab number to each sample and records their initials, shipment method and the date and time of receipt. The lab number is recorded on each sample label or tag and on the COC form when applicable. Each sample is assigned a unique sample identification number of the format XWYYYY or XGYYYY where X is the number denoting the year, then either W (representing Water Quality) or G (representing Ground Water) represents the type of sample and YYYY is an accession number beginning with '0001'.

If samples are identified for legal/evidentiary purposes on the fieldsheet, laboratory staff will retain the shipping record with the COC, initiate an internal COC for laboratory use by analysts and a sample disposal record. When a sample is removed from the Receiving Room, the custody is transferred from the sample custodian to an analyst. This person may be one and the same at the regional laboratories. The transaction is recorded in the "lab use only" section of the COC form by date, time and user.

Samples designated as 'Emergency' receive priority handling. Colored stickers denote their priority status for quick identification and all other work or sample analyses are often preempted by these samples.

Copies of the fieldsheets are routed through applicable analytical units and the originals are sent to a Processing Assistant for entry into the laboratory data management system. All samples received by the Laboratory Section are logged into the Sample Tracking and Reporting Laboratory Information Management System (DWQ STAR LIMS) to allow the laboratory to track and evaluate sample progress.

The samples are logged into the DWQ STAR LIMS with the following information.

(a) lab number
(b) sample location/id/description
(c) mode of sample delivery (e.g. common carrier, Federal Express, etc.) and date
(d) lab comments
(e) date and time collected
(f) date and time received
(g) sample type
(h) sample priority
(i) analyses requested
(j) initials of the log-in person
(k) matrix

This information must be unequivocally linked to the sample record or included as part of the record. If such information is recorded or documented elsewhere, the records shall be part of the lab's permanent records, easily retrievable upon request and readily available to individuals who will process the sample. Note: Information placed or recorded on the sample container or tag is not considered permanent record.

After all the sample information is logged into the system, the sample is authorized in the DWQ STAR LIMS. The corresponding fieldsheets are placed in a notebook or central location to await final report generation.

7.4 Procedure to Assess Capability to Meet Workload Requirements

It is the primary responsibility of the Section Chief, through the Branch Managers and Unit Supervisors, to manage workload in the lab. Availability of capability in the lab is contingent on both labor and instrumentation. All samples are logged into the DWQ STAR LIMS and given a unique lab number. It is the responsibility of the Supervisor for each analytical unit to review the incomplete worklist daily with the chemists/technicians and report any problems with scheduling to the Branch Manager. The Branch Manager maintains a detailed status report, which provides information on all samples that are logged into the lab. This information includes due date and incomplete summary. Scheduling, manpower and instrument issues are discussed on a unit by unit basis and resolved. The Branch Manager tracks analytical units with limited capacity or scheduling issues and passes this information to the Section Chief to notify samplers.

Sample collection dates/times are entered during sample login. The holding time deadline is calculated from this information and noted on the backlog report. Supervisors have the responsibility to ensure all analyses under their supervision are prepared and analyzed within the holding times. Chemists and technicians who schedule their work priority from backlog reports have the responsibility to complete analytical work within the holding time.

7.5 Storage conditions

After receipt and check-in, samples are transferred from the sample receiving area to the analytical units or sample storage areas. Storage areas must not contribute to deterioration, contamination, loss or damage to the sample. When samples must be stored under specified environmental conditions, these conditions shall be maintained monitored and recorded. The primary considerations for sample storage are temperature, holding times, contamination and security.

Samples are stored in the following areas within the Central Laboratory:

(a) temperature-controlled room (G-113): aqueous metal samples
(b) walk-in refrigerator (G035A): organic samples (SVOA, pesticides)
(c) walk-in refrigerator (G028): nutrients and wet chemistry samples
(d) walk-in refrigerator (G088): sediment metal and microbiology samples
(e) VOA lab refrigerator #1: VOA sample secondary aliquots
(f) VOA lab refrigerator #2: VOA aqueous and sediment samples
(g) lab freezer (G113): tissues and other samples requiring freezing
(h) lab freezer (G066): filtered chlorophyll samples

The regional laboratories store samples in refrigerators located in the analytical areas.

Section 6.0 summarizes the temperature and holding time protocols for various analyses. Samples, sample fractions, extracts, digestates or other sample preparation products that require thermal preservation shall be kept at +/- 2°C of the test method requirements. Those samples that have a specified storage temperature of 4°C may be stored at 0.1 to 6°C as long as there is no evidence of freezing. The temperature of cold storage areas are monitored and recorded daily and corrective action is taken as necessary.

All samples distributed into the laboratory are stored separately from standards and reagents used for analyses to prevent cross-contamination. Samples are also stored away from food and other potentially contaminating sources. Samples may not be stored in the refrigerator compartment of a unit that has standards stored in the freezer compartment. Sample fractions, extracts, digestates, and other sample preparation products shall be stored according to Section 6.0 (or according to the specifications in the test method) in controlled storage areas in the analytical unit.

The Laboratory Section laboratories are limited access, secure facilities. Only authorized personnel are permitted within the laboratory areas where sample access is possible. Access to the laboratory is controlled such that sample storage need not be locked at all times unless a particular case demands it. Samples are accessible to DWQ Laboratory Section personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of the Laboratory Section. Samples are returned to the appropriate refrigerator after sufficient sample has been obtained to complete the analysis.

7.6 Sample Disposal

Samples are normally maintained in the lab no longer than three months from receipt unless otherwise requested. If the sample is part of litigation, the affected legal authority, data user, and/or sample submitter must participate in the decision about the sample's disposal.

Disposal is performed in accordance with local, state, and US EPA-approved methods. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes:

- Date of disposal

- Nature of disposal (e.g., sample depletion)
- Names of individuals who conducted arrangements and physically completed the task.

Sample disposal may be handled in the following manner:

1) The sample may be consumed completely during analysis, or
2) The sample may be stored after analysis (samples are normally maintained no longer than 90 days from receipt unless otherwise requested and discarded when all laboratory analyses for that sample are complete.

7.7 Sample Custodians

Personnel working in the sample receiving room or sample receiving area are designated as sample custodians. The managers, chemists, technicians and QA/QC Coordinator may also be designated as sample custodians.

7.8 Inter-laboratory Custody

Samples that need to be subcontracted or routed to another laboratory within the Laboratory Section will show transfer to that lab on a sample transmittal form, which lists sample ID numbers and requested analyses. It will include the date/time it was sent out and the identity of the custodian responsible. For chain of custody samples, the COC form is completed and delivered to the receiving lab with the associated samples. The delivery technician and the recipient at the receiving lab must sign the COC indicating the transfer dates and times.

7.9 Sample Tracking and Reporting Laboratory Information Management System (DWQ STAR LIMS)

The laboratory uses a customized Excel© based LIMS, which runs on a Windows© operating system over a Novell Netware© LAN. The main server and the applications and hardware are maintained by the Bio/Chemistry Unit Supervisor.

To gain access to the LIMS, users must provide valid network and LIMS usernames and passwords.

When all the analyses for a sample are complete and the sample is authorized for release, two copies of the report are printed. The Branch Managers or the Section Chief certifies the reports by initialing them. One report is retained with the field data sheets in the laboratory. The other report is mailed with a copy of the fieldsheets to the client. Clients may also access the report via the state's WAN.

The LIMS software is being modified on a continuing basis by the section. The revisions of the codes are documented in the project history file of each application. The verification of the performance of the LIMS software and/or hardware is performed each time when any part of it is used. Any abnormalities are reported to the Bio/Chemistry Unit Supervisor immediately for quick corrective action(s).

Figure 7.1 Water Quality Section Fieldsheet.

DIVISION OF WATER QUALITY - LAB FORM

COUNTY : _____
RIVER BASIN : _____
REPORT TO : _____
SHIPPED BY : _____
COLLECTOR(S) : _____

PRIORITY
☐ AMBIENT ☐ QA
☐ COMPLIANCE ☐ CHAIN OF CUSTODY
☐ EMERGENCY

☒ W SAMPLE TYPE
☐ STREAM ☐ EFFLUENT
☐ LAKE ☐ INFLUENT
☐ ESTUARY

Lab Number : _____
Date Received : _____
Time Received : _____
Received By : _____
Data Released : _____
Date Reported : _____

Estimated BOD Range: Station Location: _____
Seed: _____ Chlorinated: _____ Remarks: _____

Station #	Date Begin (yy/mm/dd)	Date End (yy/mm/dd)	Time Begin	Time End	Depth - DM, DB, DBM	Value Type - A, H, L	Composite-T, S, B	Sample Type C G GNXX

	BOD 310	mg/L		Chloride 940	mg/L		NH3 as N 610	mg/L		Li- Lithium 1132	ug/L	
	COD High 340	mg/L		Chl a: 70953	ug/L		TKN as N 625	mg/L		Mg- Magnesium 927	mg/L	
	COD Low 335	mg/L					NO2 plus NO3 as N 630	mg/L		Mn- Manganese 1055	ug/L	
	Coliform: MF Fecal 31616	#/100 mls					P: Total as P 665	mg/L		Na- Sodium 929	mg/L	
	Coliform: MF Total 31504	#/100 mls		Color: True 80	c.u.		PO4 as P 70507	mg/L		As- Arsenic:Total 1002	ug/L	
	Coliform: Tube Fecal 31615	#/100 mls		Color: (pH) 83 pH	c.u.		P: Dissolved as P 666	mg/L		Se- Selenium 1147	ug/L	
	Coliform: Fecal Strep 31673	#/100 mls		Color: pH 7.6 82	c.u.		K- Potassium	mg/L		Hg- Mercury 71900	ug/L	
	Residue: Total 500	mg/L		Cyanide 720	mg/L		Cd- Cadmium 1027	ug/L		Ba- Barium	ug/L	
	Volatile 505	mg/L		Fluoride 951	mg/L		Cr- Chromium:Total 1034	ug/L		Organochlorine Pesticides		
	Fixed 510	mg/L		Formaldehyde 71880	mg/L		Cu- Copper 1042	ug/L		Organophosphorus Pesticides		
	Residue: Suspended 530	mg/L		Grease and Oils 556	mg/L		Ni- Nickel 1067	ug/L				
	Volatile 535	mg/L		Hardness Total 900	mg/L		Pb- Lead 1051	ug/L		Acid Herbicides		
	Fixed 540	mg/L		Specific Cond. 95	umhos/cm2		Zn- Zinc 1092	ug/L				
	pH 403	units		MBAS 38260	mg/L		V- Vanadium	ug/L		Base/Neutral&Acid Extractable Organics		
	Acidity to pH 4.5 436	mg/L		Phenols 32730	ug/L		Ag- Silver 1077	ug/L		TPH Diesel Range		
	Acidity to pH 8.3 435	mg/L		Sulfate 945	mg/L		Al- Aluminum 1105	ug/L				
	Alkalinity to pH 8.3 415	mg/L		Sulfide 745	mg/L		Be- Beryllium 1012	ug/L		Purgeable Organics (VOA bottle req'd)		
	Alkalinity to pH 4.5 410	mg/L		Boron: Total 1022	ug/L		Ca- Calcium 916	mg/L		TPH Gasoline Range		
	TOC 680	mg/L		Tannin & Lignin 32240	ug/L		Co- Cobalt 1037	ug/L		TPH/BTEX Gasoline Range		
	Turbidity 82079	NTU		Hexavalent Chromium 1032	ug/L		Fe- Iron 1045	ug/L		Phytoplankton		
	Coliform Total Tube 31508	#/100 mls										

COMMENTS : _____

LAB USE ONLY
Temperature on arrival (ºC):

Sample Point % (2)	Conductance (94)	Water Temp-C (10)	D.O. (300)	pH (400)	8.3 Alkalinity (82244)	4.5 Alkalinity (431)	4.5 Acidity (82243)	8.3 Acidity (82242)	Air Temp-C (20)
Secchi depth m	Salinity ppt (480)	Precipit-In/day (45)	Cloud Cover % (32)	Wind Dir-Deg (36)	Strm Flow Sev (1351)	Turbidity Severity (1350)	Wind Velocity-mph (3	Mean Strm Depth-ft (64)	Strm Width-ft (4)

Figure 7.2 Division of Solid Waste Management Chain of Custody Form

Report to: _____ DIVISION OF WASTE MANAGEMENT – WATER QUALITY SECTION (WQ) Page _____ of _____

CHAIN OF CUSTODY RECORD

NC DENR/DWQ LABORATORY (check one): [ ] CENTRAL [ ] ARO [ ] WaRO

For Investigation of:						**Incident No.**
Sample collector (print name) and DM-1 forms completed by: _____ Sample collector's signature: _____						
Field storage conditions and location (when applicable):						
Lab Use Only LAB NO.	SAMPLE ID	QUAD. NO.	LOCATION	DATE SAMPLED	TIME SAMPLED	NUMBER OF CONTAINERS
Relinquished by (signature):		Date	Time	Received by (signature):	Date	Time
Relinquished by (signature):		Date	Time	Received by (signature):	Date	Time
Relinquished by (signature):		Date	Time	Received by (signature):	Date	Time
Method of Shipment (circle one): State Courier Hand-delivered Federal Express UPS Other: _____						
Security Type and Conditions:		Sealed by:		Broken by:		

INTRALABORATORY CHAIN OF CUSTODY - Lab Use Only

LAB NUMBERS FROM	THROUGH	NUMBER BOTTLES	ANALYSES REQUESTED	RELINQUISHED BY:	RECEIVED BY:	DATE	TIME

8.0 ANALYTICAL PROCEDURES

The analytical methods utilized by the laboratory are listed in Section 5.0 of this QAM. Whenever possible, only EPA-approved methods are used. The reference methods are also documented in the laboratory's Standard Operating Procedures (SOPs). For information about the documentation and maintenance of laboratory SOPs, refer to SOP# QAG001 - *Guidance for Preparing Standard Operating Procedures*.

8.1 Reference Methods

The following compilations encompass the individual methods listed in Section 5.0 (listed by acronym designation as used in Section 5.0 tables).

8.1.1 EPA

- *Methods for Chemical Analysis of Water and Wastes;* USEPA Office of Research and Development, Cincinnati, OH, 3/83; EPA 600/4-79-020.
- *Methods for the Determination of Metals in Environmental Samples*, USEPA Office of Research and Development, Washington DC, 6/91, EPA/600/4-91/010.
- *Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods, SW-846*; 3rd edition (9/86), with Final Updates I (7/92), II (9/94), IIA (9/93), IIB (1/95) and III (12/96); USEPA Office of Solid Waste and Emergency Response, Washington, D.C.
- *Method for the Determination of Organic Compounds in Drinking Water, Supplement I*, EPA 500/4-90/020, July 1990.
- *Code of Federal Regulations, Title 40, Part 136*; U.S. Government Printing Office, Washington, D.C., July 1993.

8.1.2 SM##

- APHA, AWWA, WEF. 1992. Standard Methods for the Examination of Water and Wastewater, 18th Edition (designated as SM18 in Table 5.1).
- APHA, AWWA, WEF. 1995. Standard Methods for the Examination of Water and Wastewater, 19th Edition (designated as SM19 in Table 5.1).
- APHA, AWWA, WEF. 1998. Standard Methods for the Examination of Water and Wastewater, 20th Edition (designated as SM20 in Table 5.1).

8.1.3 CA LUFT

- Leaking Underground Fuel Tank Field Manual: Guidelines for Site Assessment, Cleanup, and Underground Storage Tank Closure, Method for Determination of Petroleum Range Organics October, 1989, State of California Leaking Underground Fuel Tank Task Force (designated CA LUFT).

8.1.4 Other Reference Procedures

Other reference procedures for non-routine analyses may include methods established by a specific state (e.g., MA DEP EPH) or by a vendor company such as HACH, QUIK CHEM or by organizations such as

USGS or ASTM. Sample type, source, instrumentation and the governing regulatory agency requiring the analysis will determine the method utilized.

8.2 Method Modifications

Many of the environmental sample analysis methods were written using the best available technology at the time of their publication. However, some of these methods have not been updated since that time and therefore do not reflect advances in technology. The Laboratory Section has modified some of these methods to take advantage of technological advances. The majority of these modifications are minor, do not have any impact on the quality of the data, and are included here for the sake of completeness. Some published methods are also not clear or are ambiguous about their requirements. Clarifications are made about these methods in this Section.

All modified methods are verified by performing an MDL study and are closely monitored for precision and accuracy. If the method performance is equivalent to that published in the method, the modification is adopted for routine use in the laboratory. The modification is summarized in the QAM and is described in detail in the SOP.

The following modifications have been made to methods indicated in Table 5.1:

8.2.1 EPA 200.2

Method 200.2 (hot plate) is modified and validated for use with a block digestor. 0.50 mL of nitric acid and 0.50 mL of 1+1 hydrochloric acid is added to 50 mL of sample in either a Teflon or disposable polypropylene tube and heated at 95°C for approximately 6 hrs. The sample is then brought back to a volume of 50 mL with deionized water. U.S. EPA Region 4 has provided written approval for the use of EPA Method 200.2 with this modification.

8.2.2 EPA 245.1

The QCS is not used to fortify an aliquot of LRB or sample matrix (ref. EPA Method 245.1, Section 3.11). Hydrochloric acid is used instead of sulfuric acid to prepare the stannous chloride solution as stated in Section 7.10. Stannous chloride is prepared per instructions from the instrument manufacturer. The lab is analyzing an LFM and LFMD to monitor precision (instead of LD_1 and LD_2 as stated in EPA Method 245.1, Section 3.5). The relative percent difference will determine if precision is acceptable.

8.2.3 EPA 245.6

An aqueous QCS is analyzed in addition to the SRM (ref. EPA Method 245.6, Section 3.10). Hydrochloric acid is used instead of sulfuric acid to prepare the stannous chloride solution as stated in Section 7.7. Stannous chloride is prepared per instructions from the instrument manufacturer. The "Stock Standard Solution" defined in Section 4.3 of SOP# MTA005R0 is equivalent to the "Mercury Stock Standard" required in EPA Method 245.6, Section 7.3. Calibration standards are prepared by diluting the stock standards solution and not by fortifying tissue samples as stated in Section 9. Potassium persulfate is not used in digesting tissue samples, as stated in Section 11.2. This deviation is based on historical data and percent recovery from analysis of a SRM.

8.2.4 EPA 245.5

An aqueous QCS is analyzed in addition to the SRM (ref. EPA Method 245.5, Section 3.10). Hydrochloric acid is used instead of sulfuric acid to prepare the stannous chloride solution as stated in Section 7.7. Stannous chloride is prepared per instructions from the instrument manufacturer. The "Stock Standard Solution" defined in Section 4.3 of SOP# MTA006R0 is equivalent to the "Mercury Stock Standard" required in EPA Method 245.5, Section 7.3. Sediment samples are not preserved with nitric acid as stated in EPA Method 245.5, Section 8.2. This is to comply with preparation of sediment samples for other metals using EPA Method 200.2, Section 8.2.

8.2.5 Standard Methods 5220 D

Standard Methods 5220 D allows the use of alternative digestion vessels and reagents (see Standard Methods 5220 D 2 (a) and 5220 C 2 (a)). Hach Company's digestion vessels, reagents and reactor are used to digest samples. The sample digestates are transferred from the Hach reaction tubes to 1.0-cm

spectrophotometer cells for colorimetric determination on the Shimadzu spectrophotometer. The modification has been validated through MDL and IDOC studies and ongoing digested QC standards.

8.3 Alternative or New Methods

When alternative procedures are employed or in cases where a test method is not mandated by regulation, the lab may choose to incorporate a new method or new instrumentation. Prior to sample analysis; however, the lab must meet the relevant start-up, calibration and ongoing validation and QC requirements. For regulated monitoring, an alternate test must be procured from EPA Region 4. An alternate test procedure is one that differs from a method previously approved by the U.S. EPA for determining the constituent of interest in National Pollutant Discharge Elimination System (NPDES) monitoring. The methods developed in-house and either validated or approved by Region 4 are outlined below:

8.3.1 ASTM D 6303-98

This method covers the determination of the formaldehyde monomer concentration in water and wastewater.

8.3.2 SM18 3030C

This method is for digesting Groundwater Section's water samples per memoranda, "Policy on Treatment of Groundwater Samples for Metals Determinations Required by 15A NCAC 2L", January 26, 1993, Arthur Mouberry, Groundwater Section Chief and "Request to Change Groundwater Section's Metals Policy", October 25, 2001, Arthur Mouberry, Groundwater Section Chief. The effective date for this method was March 1, 1993. A copy of this document is included in Figure 8-1.

8.3.3 EPA 200.2

U.S. EPA Region 4 has provided written approval to the NC DWQ Laboratory Section for the use of EPA 200.2 for NPDES compliance monitoring. A copy of this document is included in Figure 8-2.

8.3.4 EPA 200.8

U.S. EPA Region 4 has provided written approval to the NC DWQ Laboratory Section for the use of EPA 200.8 for NPDES compliance monitoring. A copy of this document is included in Figure 8-3 and an electronic mail notice of clarification regarding this approval is included in Figure 8-4.

8.3.5 EPA 200.9

U.S. EPA Region 4 has provided written approval to the NC DWQ Laboratory Section for the use of EPA 200.9 on wastewater. A copy of this document is included in Figure 8-5.

8.3.6 Platinum-Cobalt Color (SM18 2120 B)

U.S. EPA Region 4 has provided written approval to the NC DWQ Laboratory Section for the use of a spectrophotometer operating at a wavelength of 460 nm in place of the visual comparison method for wastewater samples. Copies of the original request for approval and of EPA's responses are included in Figures 8-6 and 8-7, respectively.

8.3.7 Anions by Ion Chromatography (EPA 300.0)

U.S. EPA Region 4 has provided written approval to the NC DWQ Laboratory Section for the use of EPA 300.0 on wastewater. PENDING

8.4 Standard Operating Procedures

The DWQ Laboratory Section has developed Standard Operating Procedures (SOPs) for all analytical procedures and laboratory operations. The method SOPs are derived from the most recently promulgated/approved published method. SOPs are an integral part of a successful quality system and facilitate consistency in the reliability and integrity of an end result. A SOP should describe the activity or analytical method used in the laboratory in sufficient detail that a competent analyst unfamiliar with the method could conduct a reliable review and/or obtain acceptable results. Each analytical test method SOP contains the following (where applicable): method title and

reference method, authorization signatures and approval dates, applicable matrices, practical quantitation limit, scope and application, components to be analyzed, procedure summary, deviations from referenced method, definitions, interferences, safety and waste handling, apparatus and equipment, reagents and standards, sample collection, preservation, shipment and storage, calibration and standardization, sample preparation, sample procedure, calculations, quality control, data validation procedures, preventive maintenance, troubleshooting and corrective actions for out-of-control or unacceptable data, referenced documents, personnel qualifications, attachments (including tables, diagrams, flowcharts, benchsheets, etc.), and revision history. Non-analytical SOPs follow a similar format where possible. General quality assurance SOPs are approved by the Section Chief, Branch Managers and the QA/QC Coordinator. All SOPs are controlled in the laboratory: numbered sequentially, approved and signed by the unit Supervisor, the Branch Manager, and QA Officer, dated with an effective date, placed in controlled manuals or placed in a read-only format on the network, and archived when updated. Procedures for preparation, review, revision and control are incorporated by reference to SOP# QAG001. SOPs are dynamic documents and may supersede some requirements in this document until the QAM annual update. SOPs must accurately reflect the operations of the Laboratory Section at any given time. They must be updated, verified and re-approved anytime procedures change. If no changes have taken place, SOPs must be reviewed at least annually. Any revisions must follow the prescribed approval process.

8.5 Requirements for Methods Start-up

Before the laboratory may institute a new method and begin reporting results, it must write a SOP, demonstrate satisfactory performance, and conduct a method detection limit study. There may be other requirements as stated within the published method or regulations (i.e., retention time window study, IDL, ATP approval from EPA R4, etc.).

In some instances a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QAM (i.e., SOP, MDL, and IDOC). If the sample is not for legal or regulatory purposes, the result may be reported as long as the following criteria are met: 1) the instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met, 2) the reporting limit is set at or above the first standard of the curve for the analyte and 3) the process is documented.

8.5.1 Initial Demonstration of Capability (IDOC)

An initial demonstration of capability (IDOC) must be made prior to using any test method to report results, and at any time there is a significant change in instrument type, personnel or test method.

Note: In laboratories with specialized "analytical units" (a well-defined group of analysts that together perform the method analysis), the group as a unit may meet the above criteria and this demonstration must be fully documented.

In general, this demonstration does not test the performance of the method in real world samples, but in the applicable and available clean matrix, e.g., water, solids or biological tissue. Actual sample spikes may also be used for this purpose, but only prior to reporting analytical results and only if the data was generated within the last twelve months. For analytes that do not lend themselves to spiking, e.g., TSS, the demonstration of capability may be performed using quality control samples.

All demonstrations shall be documented through the use of the IDOC Certification Statement form in Figure 4-6.

The following steps, which are adapted from the EPA test methods published in 40 CFR Part 136, Appendix A, shall be performed.

a) A quality control sample shall be obtained from an outside source. If not available, the spiking standard may be prepared by the laboratory using stock standards that are prepared independently from those used in instrument calibration.

b) The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method, or if unspecified, to a concentration approximately 10 times the method-stated or laboratory-calculated method detection limit.

c) At least 4 aliquots shall be prepared and analyzed according to the test method either concurrently or over a period of days.

d) Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations of the population sample (n-1), in the same units, for each parameter of interest. When it is not possible to determine mean and standard deviations, such as for presence, absence and logarithmic values, the laboratory will assess performance against criteria described in the Method SOP.

e) Compare the information obtained from (d) above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory-generated acceptance criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

f) When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must either:

- Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with c) above.
- Beginning with c) above, repeat the test for all parameters that failed to meet criteria. Repeated failure; however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with c) above.

g) A Certification Statement shall be used to document the completion of each IDOC. A copy of the certification is archived in a method/instrument folder and a copy is archived in the analyst's training folder.

h) Methods on line prior to the effective date of this Section shall be updated to the procedures outlined above as new analysts perform their demonstration of capability. A copy of the new record will replace that which was used for documentation in the past. At a minimum the precision and accuracy of four mid-level laboratory control samples must have been compared to the laboratory quality control acceptance limits.

8.6 Laboratory Reagent Water

Laboratory reagent water is used for the preparation of reagents and standards, the dilution of samples and blank analysis. Reagent water should have no detectable concentration of the compound or element to be analyzed at the detection limit of the analytical method. Reagent water must be free of substances that interfere with analytical methods. Laboratory reagent water is generally prepared by passing tap water through a reverse osmosis system or a still and meets or exceeds ASTM Type II Reagent Grade Water requirements. It must have a resulting specific conductance of less than 2 μmhos/cm. The conductivity is checked and recorded monthly. If the water's specific conductance exceeds the specified limit, the analyst must immediately initiate corrective action. Non-chlorinated well water is used for organic analyses. For volatile organics analyses, this well water is also passed through an activated charcoal filter before use.

8.7 Reagents and Standards

The nature of the analytical laboratory demands that all material used in any of the procedures is of a known quality. All standards and reagents are prepared from reagent grade materials, primary standards or are purchased from reputable vendors. Standards and reagents are prepared using balances in which the calibration is verified daily, Class A volumetric glassware or pipettors which are calibrated in accordance with ISO 8655-6 and ASTM Type II reagent water. The wide variety of materials and reagents available makes it advisable to specify the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP. The material is dated and initialed upon receipt and upon opening.

Material Safety Data Sheets (MSDS) are kept in a central location known to all personnel and each analytical unit shall have copies of MSDS for the chemicals used in that unit. Anyone may review these for relevant information on the safe handling and emergency precautions of chemicals used and stored on-site. In addition, laboratory SOPs describe precautionary measures (listed in the *Safety and Waste Handling* section and at the critical steps in the procedure) for particularly hazardous chemicals and known or suspect carcinogens.

8.7.1 Specifications

There are many different grades of analytical reagents available to the analyst. All methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, it may be assumed that it is not significant in that procedure and; therefore, any grade reagent may be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of the reagent grade.

Records of manufacturer's certification and traceability statements are maintained in files or binders in each analytical unit. These records include date of receipt, lot number (when applicable) and expiration date (when applicable). Commercial materials purchased for preparation of calibration solutions, spike solution, etc. are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material. Wherever possible, standards must be traceable to NBS/NIST standards, and records to that effect are maintained in the area in which the standard is to be used.

Logbooks are utilized to document all information needed to maintain proper traceability of all standards and reagents prepared or purchased by the Laboratory. Logbooks document the date of preparation or opening of purchased material, expiration date, a list of standards/reagents or solutions used, lot numbers and the preparer's name. Calibrated instruments (e.g., balance or autopipette) used in the preparation of standards, must be identified in the logbook by serial or assigned ID number. Additional information, such as pH, may also be recorded. For purchased standards/reagents, the logbook is used to record the vendor, date opened, lot number and expiration date. Reagents or working standards that are prepared in-house shall be recorded in a logbook, dated, initialed by the analyst preparing the reagent or standard, and is assigned a unique designation for tracking purposes. All reagents and solutions in the laboratory areas shall be labeled to indicate identity, analyst, titer or concentration, solvent (when applicable), preparation date and expiration date. If a vial or container is too small for all the information listed above, use an ID number to link the vial to the logbook entry containing this information. Storage requirements are generally described in the associated SOP. Deteriorated or outdated reagents and solutions shall not be used.

Expiration dates for standards and reagents are usually specified in the methods or by the manufacturer and are adhered to unless degradation prior to this date is observed. Deterioration may be recognizable by changes in physical appearance such as a change in color or clarity, a change in volume, clumping or the formation of solids. Purchased materials are labeled with the date received and the date opened. Reagents are stored according to method or manufacturer's instructions and discarded upon expiration. When expiration dates are not specified, the following guidelines are used:

- Stock Standards used for calibration can be used for up to one year if properly preserved and stored. Standard solutions, such as ammonia and TKN standards, may need to be prepared more frequently.

- Titrating solutions need to be either restandardized or a new bottle of vendor-certified standard opened each month. Titrating solutions used by the Laboratory Section include 0.02*N* sulfuric acid (alkalinity) and 0.025*N* sodium thiosulfate (BOD and total phenol), 0.0192*N* silver nitrate (cyanide), 0.1*N* HCl (formaldehyde), and 0.0141*N* silver nitrate (chloride).

- Calibration or spiking standards are dilutions of stock standards used to calibrate an instrument. These standards are to be prepared daily unless specified otherwise in the method SOP.

- Acids can be used for up to three years; however, additional care must be taken with nitric and sulfuric acid, as exposure to sun and heat will accelerate decomposition.

- Organic solvents may be used for up to one year.

- Dry, inorganic reagents and specially denatured alcohol formulations may be used for up to five years.

All other solutions are used for no more than a year. They are valid for that length of time only if evaporation is minimized and proper preservation and storage techniques are used. If a bottle is opened often or is much less than half full more frequent preparation may be required. If a solution, such as a buffer, is expected to degrade rapidly after opening, it will be labeled with the date opened and an adjusted expiration date based on the date opened. Solutions are always poured off from the original bottle and unused portions are never returned to the original bottle. If degradation becomes apparent the solution is discarded immediately and time period of valid use of that solution is reduced.

The stability of standard solutions can be demonstrated by comparing the analysis of freshly prepared solutions periodically with older preparations. The age of the standards must be limited using expiration dating so that no significant difference can be detected between older solutions and freshly prepared solutions. The lab analyst may also refer to the decomposition data available on a chemical's Material Safety Data Sheet.

Attempts should be made to control the quality of chemicals by purchasing in quantities fitting for the volume to be used. Smaller containers are appropriate for low-volume use and for products that have short shelf life while larger containers may be appropriate for high-volume use and products with indefinite shelf life.

8.7.2 Chemical Storage

All reagents and solvents are dated upon receipt. All manufacturer expiration dates are observed. If an expiration date is not specifically stated on the manufacturer's label, a holding time may be assigned and the expiration date written on the label. The date the reagent was opened is also written on the label.

Acids, except portions that are dispensed into small, labeled containers for immediate use, are stored in the original containers in the operational area in an acid cabinet or in the chemical supply room separate from alkaline bases and other unsuitable chemicals as stated in the MSDS.

Bases, except portions that are dispensed into small, labeled containers for immediate use, are stored in the original containers in the operational area or in the chemical supply room separate from acids.

Solvents, except portions that are dispensed into small, labeled containers for immediate use, are stored in the original containers in a separate area of the chemical supply room designated for solvent storage or in vented, explosion-proof cabinets in the operational area.

Dry reagents are stored in the dry chemical storage area of the chemical supply room. Organic and inorganic reagents are stored on separate shelves. Reactive chemicals are isolated from other materials. The dry chemical storage area is air-conditioned.

Light-sensitive reagents may be stored in amber/brown-glass containers. Specific storage instructions are detailed in the laboratory SOPs.

Organic extracts and stock solutions are stored in a freezer in the appropriate operational unit. Working solutions are refrigerated or frozen as appropriate. Neat standards are stored at room temperature in the analytical area. Inorganic digestates, distillates and stock and working solutions may either be refrigerated or stored at room temperature. Instructions are detailed in the analytical SOP.

8.8 Waste Disposal Methods

The Laboratory Section collects, stores, packages, labels, ships and disposes of wastes in a manner which ensures compliance with all federal, state, and local laws, regulation, and ordinances. Procedures are designed to minimize employee exposure to hazards associated with laboratory-generated wastes and to afford maximum environmental protection. Waste handling procedures are detailed in the laboratory SOPs and Chemical Hygiene Plan (CHP).

A waste is a hazardous waste if it is listed in 40 CFR Part 261.30-261.33 or fails any of the criteria in 40 CFR Part 261 Subpart C. Personal knowledge of the waste's characteristics must also be considered. Hazardous wastes must be segregated, labeled appropriately, stored in a designated waste disposal area, and disposed of by a commercial waste disposal company. The Safety Officer is responsible for maintaining the on-site system to prepare the wastes for disposal, scheduling removal by the contractor, maintaining records, and assuring that the contractor is permitted. The selection of the waste transporter must be predicated on their being permitted to transport hazardous wastes coupled with an absence of RCRA/DOT violations and a proven record of successful performance.

Processes generating waste organic solvents in the laboratory include organic sample preparation and standard/reagent preparation. Laboratory wastes are stored in labeled four-liter or 2.5 liter glass bottles. These containers are stored closed in fume hoods in the appropriate analytical units. At the Central Laboratory, when the waste bottle is full, the contents are transferred into a 55-gallon drum labeled "Waste Flammable Liquid "in the waste storage room. Fifty-five gallon drums are maintained by the laboratory in compliance with RCRA regulations for disposal of waste solvents. All records of waste disposal are maintained. Records include waste disposal manifests, correspondence from disposal firms and any other information necessary to document the disposal of laboratory wastes. Organic solvents containing PCB's are segregated for disposal with the appropriate manifest.

Solvent extracts are stored chronologically in appropriate refrigerators in the laboratory units. Upon expiration of required holding times, sample extracts are disposed of by pouring the extract into the appropriate solvent storage container and placing the empty extract container into the appropriate solid waste container.

Only completed samples (including raw samples, extracts, and digestates) with authorized reports (checked from DWQ STAR LIMS) are disposed. The Central Laboratory has a two-stage sewage system. Laboratory drains are separated from the sanitary drains. The laboratory room drains are collected and passed through a calcium carbonate filter to neutralize acids, and then are passed into the normal sanitary sewer system. The sanitary drains

bypass the pretreatment phase and drain directly to the sanitary sewer system. Non-hazardous, unpreserved aqueous samples are poured down the sink drain while flushing with tap water. Non-hazardous, preserved aqueous samples are neutralized with sodium bicarbonate and then poured down the sink drain while flushing with tap water. Non-hazardous solid samples are disposed of in the city garbage.

Biological wastes are placed in an autoclavable biohazard bag and sterilized prior to disposal in the city garbage.

8.9 Labware

8.9.1 Labware specifications

All volumetric glassware must be Class A. Pyrex glass or equivalent should be used where possible. For safety purposes, thick-wall glassware should be used where available.

8.9.2 Labware cleaning

The proper technique for cleaning labware depends upon the intended use of the labware being cleaned. The goal is to remove all substances from the labware that might interfere with the analysis. Generally, water-soluble substances can be removed with tap water followed with multiple rinses with laboratory-grade water. In some instances, detergent may be required. Detergent washing should be followed by a series of analyte-free water rinses. General procedures for cleaning laboratory glassware and other labware for specific applications are outlined in Table 8-1.

Table 8-1. Labware Cleaning Protocols.

Parameter group	**Cleaning Protocols (in order specified)**
Extractable Organics	1,2,4,5 (6 optional)
Purgeable Organics	1,2,4,5,6
Metals	1,2,3,4,7
Nutrients	1,2,3*, 4,7 *For nutrients, nitric acid should be replaced by hydrochloric acid, or hydrochloric acid may be used after the nitric acid rinse.
Minerals, Demand, and other Wet Chemistry	1,2,4,7
Oil and Grease	1,2,3*,4 (5,6 optional) *For oil and grease, nitric acid should be replaced by hydrochloric or sulfuric acid.
Residues	1,2,4,9
Bacteriologicals	1,2,7,8

Key to cleaning protocols:

1. Wash with hot water and a brush to scrub inside glassware and stopcocks, using a suitable laboratory-grade detergent (generally Detergent-8 which is phosphate-free or Alconox). Bacteriologicals - must pass an inhibitory residue test.
2. Rinse thoroughly with tap water.
3. Rinse with 1:1 nitric acid solution.
4. Rinse thoroughly with deionized water.
5. Rinse thoroughly with pesticide-grade acetone or methanol
6. Oven-dry at 105°C to 125°C for at least 1 hour. Note: Class A volumetric glassware should not be baked.
   Note: Oven dried containers (tightly capped) should remain in the oven or in a contaminant-free environment until being dispatched to the field or used for laboratory operations.
7. Invert and air-dry in contaminant-free environment.
8. Autoclave containers (the tops of which are covered with aluminum foil and an autoclave indicator strip is placed in the autoclave with the containers and tops.
9. Bake crucibles at 105°C or 180°C for 1 hour (prior to use as per method).

8.9.3 Labware storage

Once cleaned, labware is capped, inverted or covered for storage in a designated cabinet or drawer, away form bulk chemicals or reagents.

Figure 8-1. Memorandum from the Groundwater Section implementing the use of Method 3030C for the preparation of Groundwater water samples.

Division of Water Quality

Groundwater Section

October 25, 2001

Memorandum

RECEIVED
OCT 29 2001
DWQ
LABORATORY SECTION

To: Steve Tedder
Chief, Laboratory Section

From: Arthur Mouberry
Chief, Groundwater Section

Subject: Request to Change Groundwater Section's Metals Policy

The Groundwater Section's current metals policy requires use of Standard Method 3030C, "Treatment for Acid-Extractable Metals," and applies to all groundwater samples collected for metals except mercury. This policy was adopted to address problems due to various amounts of sediment and turbidity in many groundwater samples and to ensure consistent treatment of samples by samplers and laboratories. Standard Method 3030C requires field acidification of unfiltered samples and acid extraction in the laboratory followed by filtration. Use of 0.45 micron filters and a 72-hour holding time from sample collection to filtration were specified as conditions.

In an effort to maximize use of laboratory staff time, the Division of Water Quality Chemistry Lab recently recommended changes to the Groundwater Section's 3030C metals preparation policy that would eliminate some low turbidity samples from the filtration requirement and allow a larger filter size for others. The Lab's recommendations, and other metals policy options, were presented at the Groundwater Section Supervisors' Meeting held October 17-19, 2001.

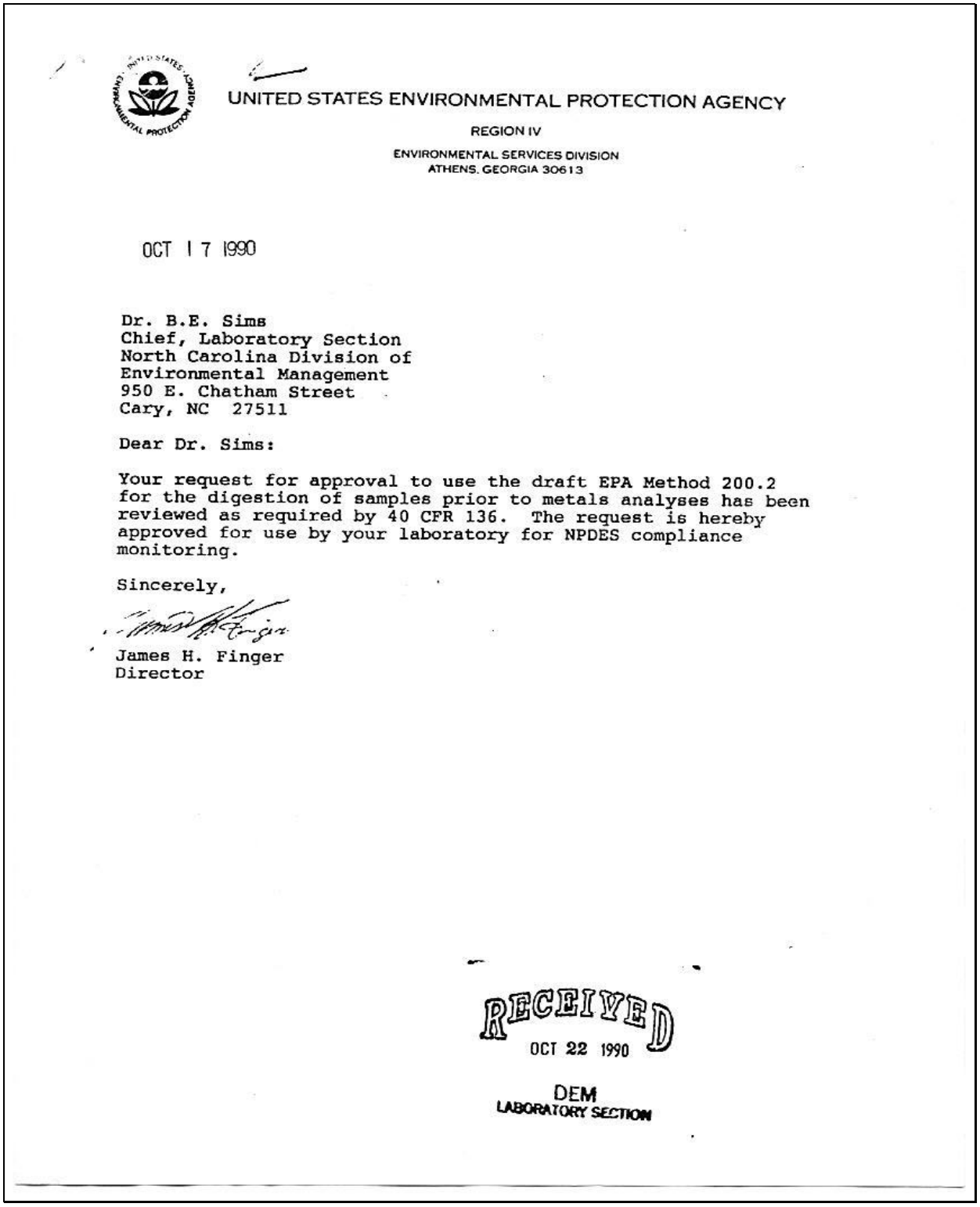
After reviewing the Lab's recommendations and other options, the Groundwater Section has decided to generally exclude water supply well samples from the 3030C metals preparation procedure requirement. Field staff will be reminded to check "water supply well" on sample tags and to list "water supply well" on field sheets when appropriate. Water supply well samples may be analyzed for "total metals," in accordance with Standard Method 3030A (Introduction to "Preliminary Treatment of Samples"), which will eliminate most of those samples from the digestion and/or filtration requirement due to low turbidity (<1 NTU). Water supply well samples with turbidity >1 NTU, and all monitor well samples must be run using the 3030C procedure. A few specific Groundwater Section investigations may involve a network of water supply wells and associated monitor wells. For such investigations, samplers may request both "total metals" analysis and the "3030C metals preparation" analysis for affected water supply wells, to ensure consistency in methodology when comparing monitor well/water supply well results.

The Groundwater Section decided to not make filter size changes to the 3030C metals preparation procedure at this time. Should there be a need to review the Groundwater Section's metals policy again, we would be happy to accept your offer to assist us with a study.

If you need additional information, please contact me at (919) 715-6170 or Betty Wilcox at (919) 715-6167.

cc: Regional Supervisors
Greg Thorpe
Ted Bush
Debra Watts
Larry Ausley
Roy Byrd
Dana Satterwhite
Betty Wilcox
Files

Figure 8-2. EPA Region 4 approval to use EPA Method 200.2.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION IV

ENVIRONMENTAL SERVICES DIVISION
ATHENS, GEORGIA 30613

OCT 17 1990

Dr. B.E. Sims
Chief, Laboratory Section
North Carolina Division of
Environmental Management
950 E. Chatham Street
Cary, NC 27511

Dear Dr. Sims:

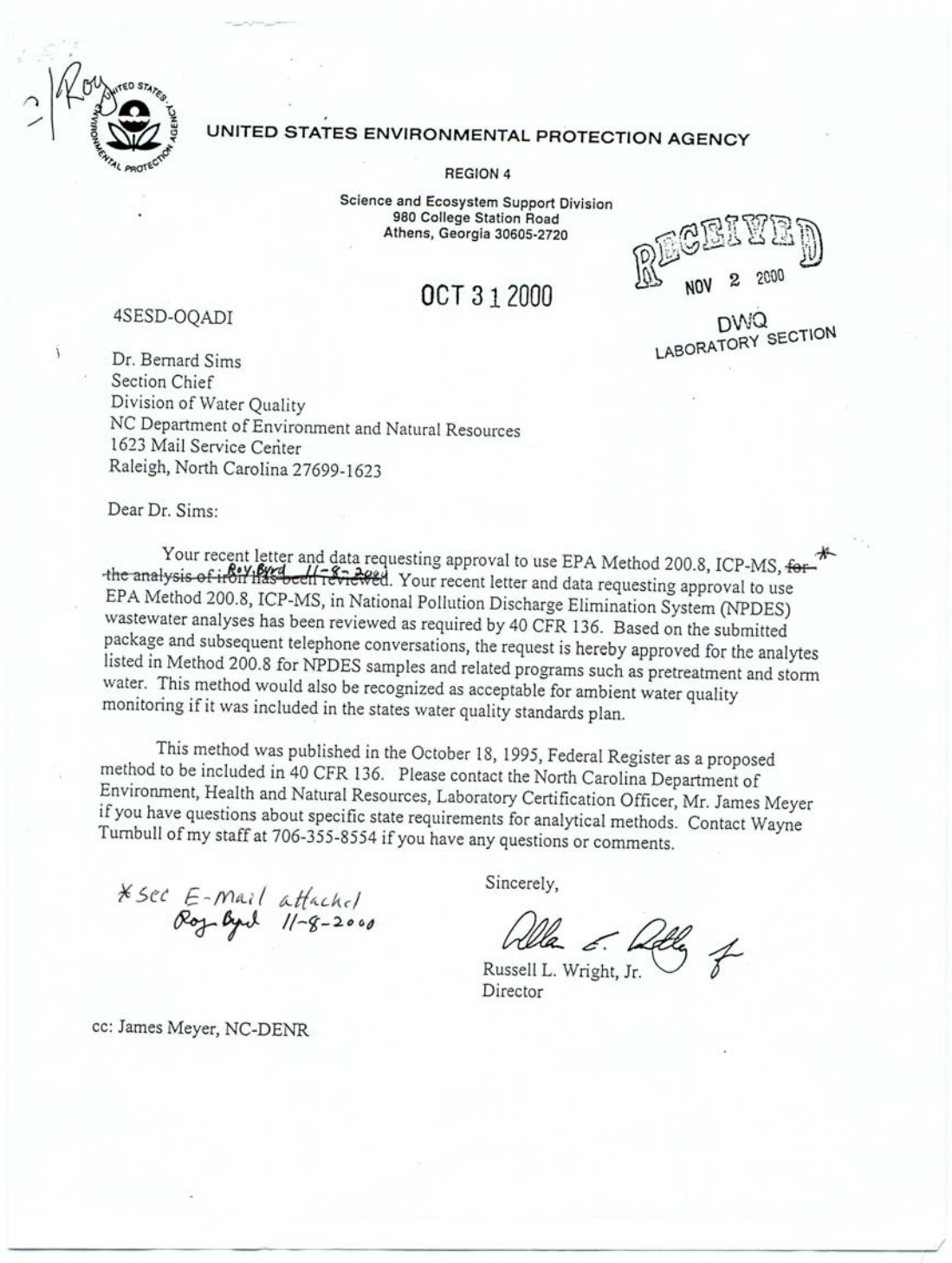
Your request for approval to use the draft EPA Method 200.2 for the digestion of samples prior to metals analyses has been reviewed as required by 40 CFR 136. The request is hereby approved for use by your laboratory for NPDES compliance monitoring.

Sincerely,

James H. Finger
Director

RECEIVED
OCT 22 1990
DEM
LABORATORY SECTION

Figure 8-3. EPA Region 4 approval for EPA Method 200.8.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION 4

Science and Ecosystem Support Division
980 College Station Road
Athens, Georgia 30605-2720

RECEIVED
NOV 2 2000
DWQ
LABORATORY SECTION

OCT 31 2000

4SESD-OQADI

Dr. Bernard Sims
Section Chief
Division of Water Quality
NC Department of Environment and Natural Resources
1623 Mail Service Center
Raleigh, North Carolina 27699-1623

Dear Dr. Sims:

Your recent letter and data requesting approval to use EPA Method 200.8, ICP-MS, ~~for the analysis of iron has been reviewed~~. Your recent letter and data requesting approval to use EPA Method 200.8, ICP-MS, in National Pollution Discharge Elimination System (NPDES) wastewater analyses has been reviewed as required by 40 CFR 136. Based on the submitted package and subsequent telephone conversations, the request is hereby approved for the analytes listed in Method 200.8 for NPDES samples and related programs such as pretreatment and storm water. This method would also be recognized as acceptable for ambient water quality monitoring if it was included in the states water quality standards plan.

This method was published in the October 18, 1995, Federal Register as a proposed method to be included in 40 CFR 136. Please contact the North Carolina Department of Environment, Health and Natural Resources, Laboratory Certification Officer, Mr. James Meyer if you have questions about specific state requirements for analytical methods. Contact Wayne Turnbull of my staff at 706-355-8554 if you have any questions or comments.

Sincerely,

Russell L. Wright, Jr.
Director

cc: James Meyer, NC-DENR

Figure 8-4. Electronic mail clarification from EPA Region 4 regarding 200.8 approval.

ATP Clarification for method 200.8

Subject: ATP Clarification for method 200.8
Date: Wed, 08 Nov 2000 10:16:31 -0500
From: Turnbull.Wayne@epamail.epa.gov
To: Roy.Byrd@NCmail.net

November 8, 2000

Dr. Bernard Sims and Mr. Roy Byrd:

An alternate test procedure approval letter for Method 200.8 was sent on October 31, 2000 to NC Department of Environment and Natural Resources, Division of Water Quality . Clarification of the first sentence in the letter is required. The intent was that all analytes listed in method 200.8 be approved as stated in the third sentence of the letter. The phrase "for the analysis of iron" was inadvertently left in the letter from a previous request by another North Carolina laboratory.

I apologize for the confusion in this matter. If you have any questions or need further clarification please give me a call at 706-355-8554. If you need a signed statement, I will be glad to FAX you a signed copy of this statement.

Wayne Turnbull
Chemist, ATP Coordinator
US EPA, Region 4

1 of 1 11/8/2000 2:50 PM

Figure 8-5. EPA Region 4 approval for EPA Method 200.9.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION 4

Science and Ecosystem Support Division
980 College Station Road
Athens, Georgia 30605-2720

NOV 16 2001

RECEIVED
NOV 19 2001
DWQ
LABORATORY SECTION

4SESD-OQADI

Mr. Steve Tedder
Section Chief
Division of Water Quality
North Carolina Department of Environmental Resources
1623 Mail Service Center
Raleigh, North Carolina 27699-1623

Dear Mr. Tedder:

Your request for approval to use EPA Method 200.9 for the analyses of lead, arsenic, selenium, cadmium, copper, nickel, and silver in waste water has been reviewed as required by 40 CFR 136. The request is hereby approved to use Method 200.9 for analyzing the metals listed above provided all quality assurance criteria are met for the method. To analyze additional metals using 200.9, please submit the supporting documentation for the additional metals. Because of the sensitivity of the methodology, the quality control criteria must be closely monitored to help assure acceptable data quality. This approval covers National Pollutant Discharge Elimination System (NPDES) discharges, storm water discharges and pre-treatment discharges to publicly owned treatment works. Other types of water monitoring, including stream samples, groundwater samples and any other type of monitoring used to meet state water quality standards, must be approved by individual states in their water quality plans (standards). EPA Region 4 recognizes Method 200.9 as an appropriate procedure for analyzing samples related to state water quality monitoring.

For your information, this method was published in the October 18, 1995, Federal Register as a proposed method to be included in 40 CFR 136. We anticipate this method being promulgated in the Federal Register in the future. If you have any questions or need additional clarification, please call Wayne Turnbull of my staff at 706-355-8554.

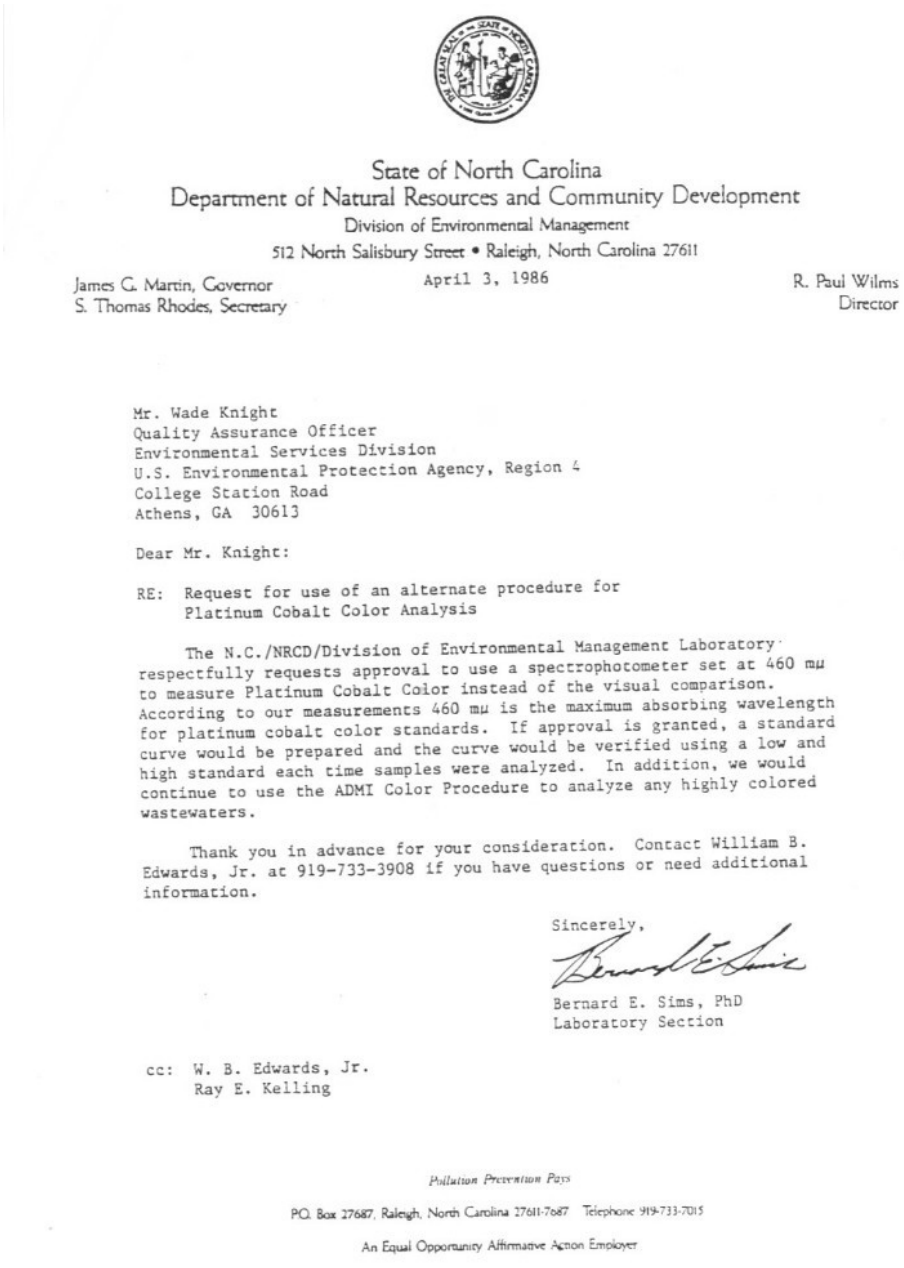
Sincerely,

Jerry Burnett /for

Allan E. Antley
Acting Director

cc: Mr. James Meyer, NC DEHNR

Figure 8-6. Request for approval for spectrophotometric determination of Platinum Cobalt color.



State of North Carolina
Department of Natural Resources and Community Development
Division of Environmental Management
512 North Salisbury Street • Raleigh, North Carolina 27611

James G. Martin, Governor
S. Thomas Rhodes, Secretary

April 3, 1986

R. Paul Wilms
Director

Mr. Wade Knight
Quality Assurance Officer
Environmental Services Division
U.S. Environmental Protection Agency, Region 4
College Station Road
Athens, GA 30613

Dear Mr. Knight:

RE: Request for use of an alternate procedure for
Platinum Cobalt Color Analysis

The N.C./NRCD/Division of Environmental Management Laboratory respectfully requests approval to use a spectrophotometer set at 460 mμ to measure Platinum Cobalt Color instead of the visual comparison. According to our measurements 460 mμ is the maximum absorbing wavelength for platinum cobalt color standards. If approval is granted, a standard curve would be prepared and the curve would be verified using a low and high standard each time samples were analyzed. In addition, we would continue to use the ADMI Color Procedure to analyze any highly colored wastewaters.

Thank you in advance for your consideration. Contact William B. Edwards, Jr. at 919-733-3908 if you have questions or need additional information.

Sincerely,

Bernard E. Sims, PhD
Laboratory Section

cc: W. B. Edwards, Jr.
Ray E. Kelling

Pollution Prevention Pays

P.O. Box 27687, Raleigh, North Carolina 27611-7687 Telephone 919-733-7015

An Equal Opportunity Affirmative Action Employer

Figure 8-7. EPA Region IV approval for spectrophotometric determination of Platinum Cobalt color.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION IV
ENVIRONMENTAL SERVICES DIVISION
ATHENS, GEORGIA 30613

REF: 4ES/AS

May 5, 1986

Dr. Bernard E. Sims
Laboratory Section
NC Division of Environmental Management
P O Box 27687
Raliegh, NC 27611-7687

Dear Dr. Sims:

Your request to use a spectrophotometer instead of visual comparison to measure Platinum Cobalt Color in wastewater and water quality samples has been reviewed. In my opinion, use of a spectrophotometer would not be considered an alternate test procedure. This opinion is shared by Mr. Terry Covert, Chief, Equivalency Staff, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

Sincerely yours,

Wade Knight

Wade Knight, Chief
Laboratory Evaluation & QA Section

9.0 Calibration Procedures and Frequency

The NC DWQ laboratories are equipped with state-of-the-art instrumentation. Major equipment lists for each of the laboratories are found in Tables 4-1, 4-2 and 4-3 of this document. Laboratory personnel routinely calibrate all instruments and equipment used within the Laboratory Section. Some instruments and/or measurement devices are also annually calibrated by an external calibration service following ISO Guide 25 protocol. A summary of calibration procedures for individual instruments and tests is provided in this section. This information is summarized in Table 9-1, *Calibration Frequency, Procedures, Standards, and Acceptance Criteria for Support Equipment* and Table 9-2, *Calibration Frequency, Procedures, Standards, and Acceptance Criteria for Operational Equipment*. It is the laboratory's policy that method calibration requirements will be followed if more stringent than those described in these sections. Calibration and continuing instrument calibration verification procedures are described in detail in the laboratory SOPs.

9.1 Standards Receipt, Preparation and Traceability

Standards are purchased from commercial sources in stock solutions or mixes designed for the specific methods or as neat analytes. Certificates of analysis are shipped with each standard material by the vendor. When possible, standards are certified to meet or exceed the criteria established by the US EPA or are traceable to NIST standards.

Standards traceability logbooks are maintained by all analytical units in the Section to track the receipt, preparation, and disposition of all standard materials. A unique laboratory identification number is assigned to each standard material. The standard material is labeled with this number, which is then documented in the standard traceability logbook along with the date of preparation, date of receipt, a descriptive name of the standard, initials of the analyst, concentration (or purity), expiration date, and solvent (when applicable). If required, a standard preparation narrative is also provided in this logbook to document the preparation steps for each stock standard. The unique laboratory identification number is recorded on all appropriate data sets.

9.1.1 Analytical standard verification

Accuracy of calibration standards is verified by analyzing independently prepared standards against calibration curves prepared using the calibration standards. These initial calibration verification standards are prepared using materials that are from a different source than those used for the initial calibration standards. It is acceptable to use standards from the same manufacturer as used for the initial calibration standards, as long as the primary standards used for the purchased solution can be shown to be from a different source (i.e., lot number). However, the preferred approach is to use standards from a different supplier altogether.

9.1.2 Standard preparation

Calibration standards are prepared using the procedures indicated in the *Reagents and Chemicals* section of the determinative method SOP. However, general procedures are described below.

- For each analyte and surrogate (when applicable) of interest, prepare calibration standards at the minimum number of concentrations as summarized in Tables 9-1 and 9-2. If a reference or mandated method does not specify the number of calibration standards, the minimum number is 3, not including blanks.

- The lowest concentration calibration standard that is analyzed during an initial calibration is generally equivalent to the practical quantitation limit and based on the final volume of extract (or sample) described in the appropriate sample preparation SOP. In some cases, the lowest concentration standard may be less than the PQL, but a standard is analyzed at the PQL concentration either as part of the curve or as a daily check. In all cases, the reporting level will be within the range of the calibration curve.

- The other concentrations define the working range of the instrument/method or correspond to the expected range of concentration found in actual samples that are also within the working range of the

instrument/method. Results of samples not bracketed by initial instrument calibration standards (i.e., not within calibration range) must be diluted to fall within the range of calibration or be reported as having less certainty by means of defined qualifiers or case narratives (with the exception of ICP methods or other methods where the referenced method does not specify two or more standards).

- Given the number of target compounds addressed by some of the organic methods, it may be necessary to prepare several sets of calibration standards, each set consisting of the appropriate number of solutions at different concentrations. The initial calibration will then involve the analysis of each of these sets of the appropriate number of standards.

- All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard when available.

- Spiking solutions are prepared according to method specifications. If no specifications are provided, they are prepared at a concentration near the middle of the calibration range such that the spiking volume is not excessive.

9.2 Laboratory Instrument Calibration

Calibration requirements are divided into two parts: requirements for analytical support equipment and requirements for operational instrument calibration.

9.2.1 Analytical Support Equipment Calibration

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include, but are not limited to balances, ovens, refrigerators, freezers, incubators, water baths, autoclaves, temperature measuring devices, and volumetric dispensing devices if quantitative results are dependent on their accuracy (as in standard preparation and dispensing or dilution into a specified volume). Support equipment requiring calibration checks can be found in Table 9-1.

Table 9-1 also includes calibration check frequency and acceptance limits. Records of these calibration checks must be documented and include (when applicable):

- Instrument model number or specific lab identification.
- Identification of standards used for the calibration check.
- Performance tolerances.
- Results of the calibration checks, the initials of the individual making the check, and the date of the check.
- A reference for the procedure used to perform the calibration check.

9.2.2 Operational Instrument Calibration

The frequency and acceptance criteria of instrument calibration and standardization are summarized in Table 9-2. Method specific SOPs expand on the following general discussion.

9.3 General Calibration Procedures

Instrument calibration and reagent standardization for the analyses performed in the lab are in accordance with the procedures specified in the referenced method (see Section 5).

9.3.1 Calibration Documentation

All calibration records including raw data, response factors, standard concentrations, curves, reduced data, and instrument settings or conditions are stored and archived as hard or electronic copy according to

laboratory standard operating procedures. Current chromatograms, curves, and results transcribed onto forms are kept in the analytical units and periodically archived into a data storage area. Initial and continuing calibrations are sorted by date for ease of location. All assigned unique standard identification numbers appear on graphs, plots, chromatograms, or curves for traceability purposes.

9.3.2 Protocol for Determining the Test Method Range of Applicability

During the development of new test methods and during initial demonstrations of capability (method validation studies), cursory evaluation will be made of the dynamic range over which the method is applicable. That evaluation will take into consideration the type of calibration protocol (linear, nonlinear), the change in sensitivity over the tested calibration region, the detection limit of the method and the practical quantitation limit. Once a valid range of applicability is established, calibration standards will be used to bracket the range over which quantitation will occur. Results reported from data that were generated outside the determined range of applicability will be flagged as estimates (unless the sample was diluted prior to analysis in order to bring concentrations within the established test method range of applicability).

During the establishment of the test method range of applicability, calibration standards will be prepared and analyzed over the estimated or published range of applicability. For inorganic parameters, if a linear calibration protocol is to be used, the correlation coefficient of the calibration values plotted against their respective responses (absorbance, concentration, etc.), must be greater than or equal to 0.995. For organic parameters, if a linear calibration protocol is to be used, either a) the correlation coefficient of the calibration values plotted against their respective response factors must be greater than or equal to 0.995, b) the relative response factors (response factor/calibration value) over the range of calibration must have a relative standard deviation of less than or equal to 10% or c) conditions for linearity specified in the applied, published method must be met.

If the above conditions are not met, either the linear dynamic range must be decreased until those conditions are met or, in some cases, a non-linear calibration protocol may be used. Whenever a non-linear calibration protocol is utilized, a minimum of 5 calibration points must be defined for a second order fit; a third order fit requires a minimum of 6 calibration points. When using non-linear calibration procedures, loss in sensitivity (Δ response/Δ concentration) can occur at high concentrations. To ensure that signals are not quantified in regions of poor sensitivity, control standards must be analyzed at the highest point of the nonlinear calibration curve during method validation and must meet the reference method acceptance criteria for calibration.

The lower limit of the test method range of applicability is normally established at the practical quantitation limit. The initial demonstration of capability includes establishment of the method detection limit and practical quantitation limit which is generally set at three to five times the calculated method detection limit.

9.3.3 General GC Calibration Procedures

General calibration procedures are described below for GC procedures using non-MS detection. The calibration procedures for other techniques are described within the applicable method SOP.

9.3.3.1 External Standard Calibration Procedure

External standard calibration involves comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards. Sample peak areas (or peak heights) are compared to peak areas (or peak heights) of the standards. The ratio of the detector response to the amount (mass) of analyte in the calibration standard is defined as the calibration factor (CF).

$$CF = \frac{\text{Peak Area (or Height) of the Compound in the Standard}}{\text{Mass of the Compound Injected (in nanograms)}}$$

For multi-component analytes, see the appropriate method SOP for information on calibration.

The CF can also be calculated using the concentration of the standard rather than the mass in the denominator of the equation above. However, the use of concentrations in CFs will require changes to the equations that are used to calculate sample concentrations.

Alternatively, software programs are used that calculate sample concentrations directly from the calibration curve.

9.3.3.2 Internal Standard Calibration Procedure

Internal standard calibration involves the comparison of instrument responses from the target compounds in the sample to the responses of specific standards added to the sample or sample extract prior to injection. The ratio of the peak area (or peak height) of the target compound in the sample or sample extract to the peak area (or peak height) of the internal standard in the sample or sample extract is compared to a similar ratio derived for each calibration standard. The ratio is termed the response factor (RF), and may also be known as a relative response factor in other methods.

In many cases, internal standards are recommended. These recommended internal standards are often brominated, fluorinated, or stable isotopically labeled analogs of specific target compounds, or are closely related compounds whose presence in environmental samples is highly unlikely. If internal standards are not recommended in the method, then the analyst needs to select one or more internal standards that are similar in analytical behavior to the compounds of interest, and not expected to be found in the sample otherwise. The use of specific internal standards is available in the method SOP.

Whichever internal standards are employed, the analyst needs to demonstrate that the measurement of the internal standard is not affected by method analytes and surrogates or by matrix interferences. In general, internal standard calibration is not as useful for GC methods with non-MS detectors because of the inability to chromatographically resolve many internal standards from the target compounds. The use of MS detectors makes internal standard calibration practical because the masses of the internal standards can be resolved from those of the target compounds even when chromatographic resolution cannot be achieved.

When preparing calibration standards for use with internal standard calibration, add the same amount of the internal standard solution to each calibration standard, such that the concentration of each internal standard is constant across all of the calibration standards, whereas the concentrations of the target analytes will vary. The internal standard solution will contain one or more internal standards and the concentration of the individual internal standards may differ within the spiking solution (e.g., not all internal standards need to be at the same concentration in this solution). The mass of each internal standard added to each sample extract immediately prior to injection into the instrument or to each sample prior to purging must be the same as the mass of the internal standard in each calibration standard. The volume of the solution spiked into sample extracts should be such that minimal dilution of the extract occurs (e.g., 10 µL of solution added to a 1 mL final extract results in only a negligible 1% change in the final extract volume which can be ignored in the calculations).

An ideal internal standard concentration would yield a response factor of 1 for each analyte. However, this is not practical when dealing with more than a few target analytes. Therefore, as a general rule, the amount of internal standard should produce an instrument response (e.g., area counts) that is no more than 100 times that produced by the lowest concentration of the least responsive target analyte associated with the internal standard. This should result in a minimum response factor of approximately 0.01 for the least responsive target compound.

For each of the initial calibration standards, calculate the RF values for each target compound relative to one of the internal standards as follows:

$$RF = \frac{A(s) \times C(is)}{A(is) \times C(s)}$$

Where:
A(s) = Peak area (or height) of the analyte or surrogate
A(is) = Peak area (or height) of the internal standard
C(s) = Concentration of the analyte or surrogate, in μg/L
C(is) = Concentration of the internal standard, in μg/L

Note that in the equation above, RF is unitless, i.e., the units from the two area terms and the two concentration terms cancel out. Therefore, units other than μg/L may be used for the concentrations of the analyte, surrogate, and internal standard, provided that both C(s) and C(is) are expressed in the same units. The mass of the analyte and internal standard may also be used in calculating the RF value.

9.3.3.3 Evaluating the Linearity of the Initial Calibration

To evaluate the linearity of the initial calibration, calculate the mean CF (external standard calibration) or RF (internal standard calibration), the standard deviation (SD) and the RSD as follows:

$$\text{Mean CF} = \overline{CF} = \frac{\sum_{i=1}^{n}(CF(i))}{n}$$

$$\text{Mean RF} = \overline{RF} = \frac{\sum_{i=1}^{n}(RF(i))}{n}$$

The variance and standard deviation of a data set measures the spread of the data about the mean of the data set.

The variance of a sample of size *n* represented by s^2 is given by:

$$s^2 = \frac{[\text{The sum of } (x - \text{mean})^2]}{(n-1)}$$

The standard deviation (SD) can be calculated by taking the square root of the variance.

RSD = (SD/mean CF) x 100
RSD = (SD/mean RF) x 100

If the RSD of the calibration or response factors is less than or equal to the acceptance limit stated in Table 9-2 over the calibration range, then linearity through the origin may be assumed, and the average calibration response factor may be used to determine sample concentrations.

9.3.6 Percent RSD Corrective Action

Given the potentially large numbers of analytes that may be analyzed in some methods, it is likely that some analytes may exceed the acceptance limit for the RSD for a given calibration. In those instances, the following steps are recommended, but not required.

- The first step is generally to check the instrument operating conditions. This option will apply in those instances where a linear instrument response is expected. It may involve some trade-offs to optimize performance across all target analytes. For instance, changes to the operating conditions necessary to achieve linearity for problem compounds may cause the RSD for other compounds to increase, but as long as all analytes meet the RSD limits for linearity, the calibration is acceptable.

- If the RSD for any analyte is greater than the applicable acceptance criteria in Table 9-2, the analyst may wish to review the results (area counts, calibration or response factors, and RSD) for those analytes to ensure that the problem is not associated with just one of the initial calibration standards. If the problem appears to be associated with a single standard, that one standard may be reanalyzed and the RSD recalculated. Replacing the standard may be necessary in some cases.

- A third alternative is to narrow the calibration range by replacing one or more of the calibration standards with standards that cover a narrower range. If linearity can be achieved using a narrower calibration range, document the calibration linearity, and proceed with analyses. Note: Changes to the upper end of the calibration range will affect the need to dilute sample above the range, while changes to the lower end will affect the overall sensitivity of the method. Consider the regulatory limits or action levels associated with the target analytes when adjusting the lower end of the range.

NOTE: As noted in Section 9.3.2, the practical quantitation limit is equal to the concentration of the lowest standard analyzed during the initial calibration. Hence, narrowing the calibration range by changing the concentration of the lowest standard will change the practical quantitation limit. When the purpose of the analysis is to demonstrate compliance with a specific regulatory limit or action level, the laboratory must ensure that the practical quantitation limit is at least as low as the regulatory limit or action level.

In those instances where the RSD for one or more analytes exceeds the acceptance criteria, the initial calibration may still be acceptable if the following conditions are met:

- The mean of the RSD values for all analytes in the calibration is less than or equal to the acceptance criteria. The mean RSD is calculated by summing the RSD value for each analyte and dividing by the total number of analytes. If no analyte has an RSD above the acceptance criteria, then the mean RSD calculation need not be performed.

- The mean RSD criterion applies to all analytes in the standards, regardless of whether or not they are of interest for a specific sample. In other words, if the target analyte is part of the calibration standard, its RSD value is included in the evaluation.

- The data user must be provided with either a summary of the initial calibration data or a specific list of those compounds for which the RSD exceeded the acceptance criteria and the results of the mean RSD calculation.

NOTE: The analyst and the data user should be aware that the mean RSD approach described above will lead to greater uncertainty for those analytes for which the RSD is greater than the acceptance criteria. The analyst and the data user should review the associated quality control results carefully, with particular attention to the matrix spike and the laboratory control sample results, to determine if the calibration linearity poses a significant concern. If this approach is not acceptable for a particular application, then the analyst may need to employ another calibration approach or adjust the instrument operating conditions and/or the calibration range until the RSD meets the acceptance criteria.

- If all of the conditions above are met, then the average calibration or response factor may be used to determine sample concentrations.

Use of other types of calibration (i.e., linear calibration using a least squares regression or non-linear calibration) may be described in manufacturer's manuals or within a published method. These procedures must be reviewed, incorporated into the appropriate SOP and approved by the QA/QC Coordinator prior to their use.

9.3.7 Retention Time Windows

Retention time windows are crucial to the identification of target compounds. Absolute retention times are used for compound identification in all GC methods that do not employ internal standard calibration. Retention time windows are established to compensate for minor shifts in absolute retention times as a result of sample loadings and normal chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results. Tight retention time windows may result in false negatives and/or may cause unnecessary reanalysis of sample when surrogates or spiked compounds are erroneously not identified. Overly wide retention time windows may result in false positive results that cannot be confirmed upon further analysis.

The following subsection describes the approach used to establish retention time windows for GC methods. Note: The criteria listed in this section are provided for GC procedures using non-MS detection. Identification procedures are different for GC/MS and are detailed in the analytical SOPs.

9.3.7.1 Before establishing retention time windows, make sure that the chromatographic system is operating reliably and that the system conditions have been optimized for the target analytes and surrogates in the sample matrix to be analyzed. Make three injections of all single component standard mixtures and multi-component analytes (such as PCBs) over the course of a 72-hour period. Serial injections or injections over a period of less than 72 hours may result in retention time windows that are too tight.

Record the retention time for each single component analyte and surrogate to three decimal places (e.g., 0.007). Calculate the mean and standard deviation of the three absolute retention times for each single component analyte and surrogate. For multi-component analytes, choose three to five major peaks (see the determinative methods for more details) and calculate the mean and standard deviation of those peaks.

If the standard deviation of the retention times for a target compound is 0.000 (i.e., no difference between the absolute retention times), then the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes. (Recording retention times to three decimal places rather than only two should minimize the instances in which the standard deviation is calculated as 0.000).

The width of the retention time window for each analyte, surrogate, and major constituent in multi-component analytes is defined as +/- 3 times the standard deviation of the mean absolute retention time established during the 72 hour period. If the default standard deviation in the above example is employed, the width of the window will be 0.03 minutes.

Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

The laboratory must calculate absolute retention time windows for each analyte and surrogate on each chromatographic column and instrument. New retention time windows must be established when a new GC column is installed.

If the instrument data system is not capable of employing compound-specific retention time windows, then the analyst may choose a window that minimizes false negatives and positives and apply it to all compounds. As noted above, other approaches may also be employed, but must be documented by the analyst. In general, you should not use a window greater than 0.2 to 0.3 minutes. If windows larger than this have been determined a cause should be looked for and the windows should be re-determined.

The surrogates are added to each sample, blank, and QC sample and are also contained in each calibration standard. Although the surrogates may be diluted out of certain sample extracts, their retention times in the calibration standards may be useful in tracking retention time shifts. Whenever the observed retention time of a surrogate is outside of the established retention time window, the analyst is advised to determine the cause and correct the problem before continuing analyses.

9.3.8 Calibration Verification

The calibration relationship established during the initial calibration must be verified at periodic intervals as specified in Table 9-2. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models.

NOTE: The process of calibration verification referred to is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach amounts to a daily single-point calibration, and is neither appropriate nor permitted in SW-846 chromatographic procedures for trace environmental analyses.

As a general rule, the initial calibration must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample or standard that can be injected within 12 hours of the beginning of the shift. Continuing instrument calibration verification must be repeated at the beginning and end of each analytical batch for non-GC/MS methods. The concentration of the calibration verification shall be varied within the established calibration range. If an internal standard is used, i.e., GC/MS, only one continuing calibration verification must be analyzed per analytical batch.

If the response (or calculated concentration) for an analyte is within the acceptance limits of the response obtained during the initial calibration, then the initial calibration is considered still valid and the analyst may continue to use the CF or RF values from the initial calibration to quantitate sample results. If the response (or calculated concentration) for any analyte varies from the mean response obtained during the initial calibration by more than the acceptance criteria, then the initial calibration relationship may no longer be valid. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, then either the laboratory has to demonstrate performance after corrective action with two consecutive successful calibration verifications, or a new initial instrument calibration must be performed. However, sample data associated with an unacceptable calibration verification may be reported under the following special conditions:

1. When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported with the appropriate qualification. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

2. When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported (with the appropriate qualification) if the sample results exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

In keeping with the approach described for initial calibration, if the average of the responses for all analytes are within that required in Table 9-2, then the calibration has been verified. However, the conditions in Section 9.3.6 also apply, e.g., the average must include all analytes in the calibration, regardless of whether they are target analytes for a specific project, and the data user must be provided with the calibration verification data or a list of those analytes that exceeded the limit. The effect of using the average of the response for all analytes for calibration verification will be similar to that for the initial calibration - namely, that the quantitative results for those analytes where the difference is greater than the limit will include a greater uncertainty. If the calibration does not meet the limit (either on the basis of each compound or the average across all compounds), check the instrument operating conditions, and if necessary, restore them to the original settings, and inject another aliquot of the calibration verification standard. If the response for the analyte is still not within the acceptance criteria, then a new initial calibration must be prepared.

9.3.9 Verification of Linear Calibrations

Calibration verification for linear calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the procedure specified in the method SOP.

$$\% \text{ Drift} = \frac{\text{Calculated concentration - Theoretical concentration}}{\text{Theoretical concentration}} \times 100$$

Where the calculated concentration is determined using the mean calibration factor or response factor from the initial calibration and the theoretical concentration is the concentration at which the standard was prepared.

$$\% \text{ Difference} = \frac{CF(v) - \overline{CF}}{CF} \times 100 \quad \text{or} \quad = \frac{RF(v) - \overline{RF}}{RF} \times 100$$

Where CF(v) and RF(v) are the calibration factor and the response factor (whichever applies) from the analysis of the verification standard, and CF and RF are the mean calibration factor and mean response factor from the initial calibration. Except where superseded in certain determinative methods, the % difference or %drift calculated for the calibration verification standard must be within +/- 15% for each analyte, or averaged across all analytes, before any sample analyses may take place.

9.3.10 Verification of Non-Linear Calibrations

Calibration verification of a non-linear calibration is performed using the percent drift calculation described in Section 9.3.9. Calibration verification must be acceptable before any sample analyses may take place. It may also be appropriate to employ two standards at different concentrations to verify the calibration. This is outlined in the method SOP when used.

Regardless of whether a linear or non-linear calibration model is used, and the percent drift difference criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications in the method SOP. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating

conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

All target analytes and surrogates, including those reported as non-detects, must be included in a periodic calibration for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met. The frequency is noted in Table 9-2.

Samples analyzed using external standards must be bracketed by periodic analysis of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The results from these bracketing standards must meet the calibration verification criteria and the retention time criteria. However, if the standard analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit, and the analyte was not detected in any of the previous samples during the analytical shift, then the sample extracts do not need to be reanalyzed, as the verification standard has demonstrated that the analyte would have been detected were it present.

9.4 Instrument-Specific Calibration Procedures

The brief narratives describing instrument calibration procedures listed below meet or exceed cited method requirements. All calibrations are recorded in the raw data or on bench worksheets for that analytical run.

9.4.1 Support Equipment

pH meter

Each pH meter is calibrated daily with two or three standard buffers, generally at pH 4.0, 7.0 and 10.0, and checked with a third buffer at or near pH 7.0, which must indicate $\pm$0.10 pH units of its given value. Manual or automatic temperature compensation is performed, depending on the meter. Additional checks of the pH meter must be performed with buffers other than 4 or 10 if samples are outside the pH range of 4-10.

Calibration information from manual determinations is recorded in a calibration logbook for the pH meter and/or on laboratory bench worksheets.

Analytical Balance

Electronic analytical balances are calibrated daily with internal mechanisms, if available. The calibration of the balance must be checked daily by the analysis of two Class S (or equivalent) weights that bracket the approximate weight of material that is being determined. The balance must be checked quarterly by the analysis of a series of at least 3 weights that the lab routinely determines. The daily and quarterly calibration checks must be documented in a logbook kept with the balance or on laboratory bench worksheets. In addition, on a yearly basis, all analytical balances are calibrated, cleaned and certified by an independent company.

Thermometer Calibration and Temperature Checks

Equipment such as refrigerators, freezers, ovens, waterbaths, hot blocks, and incubators are periodically checked with calibrated or NIST traceable thermometers. Refrigerators and freezers are checked daily and the temperatures documented in a notebook or on laboratory bench worksheets. The temperature of microbiological incubators and waterbaths must be checked and recorded twice daily. Sample storage refrigerators should be set to 4°C. They must maintain a temperature less than 6°C, and must not freeze aqueous samples. All

thermometers are calibrated annually against an NIST-certified thermometer. Thermometers are replaced when they are not within allowable tolerances, otherwise they are labeled and the proper correction is applied.

9.4.2 Metals Calibration Protocols

ICP-AES

The Inductively Coupled Plasma-Atomic Emission Spectrometer (ICP-AES) is calibrated with each analytical batch and whenever the response of the continuing calibration verification (CCV) standard varies by greater than ± 10% from the initial calibration. The initial calibration curve is generated using an instrument blank and a minimum of four standards encompassing the concentration range of interest. The curve fit is linear, first order. The initial calibration curve must meet the following criteria:

- Accuracy of a daily Quality Control Check Sample (QCS) must be in the range of 90-110 %.
- Calibration/instrument blank must exhibit a response < ½PQL;
- An interference check standard is analyzed and values must agree within ± 15 % of the components' true values.

A Continuing Calibration Verification (CCV) Standard at the mid-point of the calibration curve and an instrument blank are analyzed every 10 samples and at the end of the run to insure the continuing validity of the initial calibration. The CCV must agree within ± 10 % of the initial calibration. In addition, the blank must exhibit a response < ½PQL of the analysis components.

All calibration runs and sample results to which the calibration applies are recorded on the system hard disk. All data are archived to the network, where they are stored permanently on optical disk. All records are filed by run date.

ICP-AES (Optima 3000 XL) Calibration Protocol Summary.

Calibration Check	**200.7 Criteria**
Minimum number of calibration points	4
Initial Instrument Performance Check (IPC)	±5% of true value
Initial Calibration Blank (ICB)	<½PQL
PQL standard	Detected; ±50% of true value
Interference Check Solutions	±20% of true value
Continuing Calibration Verification (CCV)	±10% of true value
Continuing Calibration Blank (CCB)	<½PQL

ICP-MS

The Inductively Coupled Plasma-Mass Spectrometer's (ICP-MS) performance is verified prior to the beginning of an analysis run and every 12 hours thereafter, using a multi-element check solution containing Ce, Ba, Pb, Mg, In and Rh at 10 ug/L each. The performance analysis must meet the following criteria:

- Ba^{++}/Ba^{+} 69 ratio is ≤ 3 %;
- CeO^{+}/Ce^{+} 155.9 ratio is ≤ 3 %;
- Mg 4 intensity must be ≥ 20,000 ions/sec:
- In 114.9 intensity must be ≥ 300,000 ions/sec;
- Rh 102.9 intensity must be ≥ 150,000 ions/sec.
- Pb 208.0 intensity must be ≥ 100,000 ions/sec:

The results from this optimization/tune are recorded in the instrument's daily operating log.

The ICP-MS is calibrated with each analytical batch and whenever the continuing calibration verification standard (CCVS) varies by greater than 10% from the initial calibration. The initial calibration curve is generated using an instrument blank and four standards. The curve fit is linear, first order. Quantitations are carried out using the internal standard technique. The initial calibration curve must meet the following criteria:

- Accuracy of a daily Quality Control Check Standard (QCS) must be in the range of 90-110 %.
- Calibration/instrument blank must exhibit a response < ½PQL.

A Continuing Calibration Verification Standard (CCVS) at the mid-point of the calibration curve and an instrument blank are analyzed at least every 10 samples. The CCVS must agree within 10% of the initial calibration. In addition, the blank must exhibit a response below the MDL of the analysis components.

The internal standard acceptance criteria for natural water samples is 60-125% of the internal standard's initial intensity for the analytical run as per EPA Method 200.8. The internal standard acceptance criteria for solid and waste samples is 30-130% of the internal standard's initial intensity for the analytical run as per EPA Method 6020.

The summary data for each run is archived to floppy disk and to the network, where it is stored permanently on optical disk.

ICP-MS (Elan 6100) Calibration Protocol Summary.

Calibration Check	**200.8 Criteria**
Minimum number of calibration points	4
Initial Instrument Performance Check (IPC)	±5% of true value
Initial Calibration Blank (ICB)	<½PQL
PQL standard	Detected; ±50% of true value
Continuing Calibration Verification (CCV)	±10% of true value
Continuing Calibration Blank (CCB)	<½PQL

AA (Atomic Absorption)

Atomic absorption spectrophotometers are calibrated daily with the specified number of calibration standards, including a calibration blank. The curve fit is linear, first order. The correlation coefficient of the regression curve must be greater than or equal to 0.995. An initial calibration verification (ICV) standard is analyzed immediately upon calibration and must meet acceptance criteria. Continuing calibration verification (CCV) standards are analyzed after every 10 samples and at the end of the sequence and must meet the acceptance criteria. A calibration blank (ICB or CCB) is analyzed immediately after the verification standards and must meet the acceptance criteria.

All calibration acceptance criteria and pass/fail status are documented on raw calibration data files. Calibration data is filed by run date and method number. The sample numbers to which calibrations apply are recorded on calibration records.

GFAA (Graphite Furnace Atomic Absorption) Calibration Protocol Summary.

Calibration Check	**200 series**	**200.9**
Minimum number of calibration points	4	4
Initial Calibration Verification (ICV)	±10% of true value	+5% of true value

Initial Calibration Blank (ICB)	<½PQL	<½PQL
Continuing Calibration Verification (CCV)	±10% of true value	+10% of true value
Continuing Calibration Blank (CCB)	<½PQL	<½PQL

All sample results must be bracketed by acceptable calibration standards.

FLAA (Flame Atomic Absorption) Calibration Protocol Summary.

Calibration Check	**200 series**
Minimum number of calibration points	4
Initial Calibration Verification (ICV)	±10% of true value
Initial Calibration Blank (ICB)	<½PQL
Continuing Calibration Verification (CCV)	±10% of true value
Continuing Calibration Blank (CCB)	<½PQL

All sample results must be bracketed by acceptable calibration standards.

CVAA (Cold Vapor Atomic Absorption - Mercury) Calibration Protocol Summary.

Calibration Check	**200 series**
Minimum number of calibration points	6
Initial Calibration Verification (ICV)	±5% of true value
Initial Calibration Blank (ICB)	<½PQL
Continuing Calibration Verification (CCV)	±10% of true value
Continuing Calibration Blank (CCB)	<½PQL

All sample results must be bracketed by acceptable calibration standards.

9.4.3. General Chemistry

Flow Injection Auto Analyzers

A calibration curve containing 5-8 calibration standard levels is analyzed daily, at the start of each analytical run sequence. External standard calibration is utilized. The calibration curve must meet the following criteria:

- The correlation coefficient for the linear regression must be ≥ 0.995 using a regression fit;
- Accuracy of a daily QC Check Standard must be in the range of 90-110 % or within the manufacturer's accuracy acceptance range, unless historical data indicate that tighter control limits can be routinely maintained;
- Calibration/instrument blank must exhibit a response < ½PQL.

Continuing Calibration Check Standards (CCCS) at the concentration mid-point of the initial calibration are analyzed every 20 samples, to insure the continuing validity of the initial calibration. The CCCS must agree within +/-10 % of the response of the initial calibration to be valid. If this check fails, the instrument is re-calibrated. In addition, the calibration/instrument blank, which is analyzed every 10 samples, must exhibit a response below < ½PQL. Samples analyses must be bracketed by calibration verification standards that meet control criteria.

Calibration information is recorded on the computer printout of raw data. The calibration runs are also recorded on the system hard disk and stored on floppy diskette. All initial calibration raw data is filed by run date and method number. The sample numbers to which calibration apply are recorded on calibration records. Applicable calibration run dates are recorded on sample raw data records.

Ion Selective Electrode (ISE)

Ion selective electrodes are calibrated daily with a minimum of three standards and a blank. The calibration curve is established by linear regression applied to the standard concentrations versus the corresponding millivolt values. The calibration curve must meet the following criteria:

- The correlation coefficient must be greater than or equal to 0.995.
- The slope for the 1 to 10 ppm standards should be -59 $\pm$ 4 mV /decade and the efficiency (of meter) of -1.00 $\pm$0.08.
- Accuracy of a daily Quality Control Check Standard (QCS) must be in the range of 90-110 % or within the manufacturer's accuracy acceptance range, unless historical data indicate that tighter control limits can be routinely maintained.

Calibration/instrument blank must exhibit a response < ½PQL. A Continuing Calibration Check Standard (CCCS) is analyzed every 10 samples, to insure the continuing validity of the initial calibration. The CCCS must agree within $\pm$10% of the response of the initial calibration to be valid. If this check fails, the instrument is re-calibrated. Data must be bracketed by calibration standards that meet control criteria to be acceptable. In addition, the calibration/instrument blank, which is analyzed every 20 samples, must exhibit a response < ½PQL.

ISE calibration information is recorded on raw data bench worksheets.

Turbidimeter

Initial calibration is performed upon instrument startup and whenever the continuing calibration verification standard fails the acceptance criteria. Sealed standards are calibrated against formazin standards initially and then quarterly. The instrument is calibrated daily with one sealed standard for each range of interest and a blank. The calibration/instrument blank must exhibit a response below 0.05 NTU.

Calibration information is recorded on the laboratory bench worksheets.

Ion Chromatograph

Initial calibration is performed for every analytical run and whenever the response factor of the continuing calibration check standard varies by more than $\pm$15 % from the latest initial calibration. A calibration curve is prepared for all target analytes (using a minimum of three standard concentration levels) with the lowest standard concentration at or below the reporting limit and the remaining standards defining the working range of the detector. Either linear regression or quadratic curve fitting is used, depending on the analyte. All quantitations are carried out using the external standard technique. The initial calibration curve must meet the following criteria:

- The correlation coefficient must be $\geq$ 0.995;
- Accuracy of a daily Quality Control Check Standard (QCS) must be in the range of 90-110 %, unless historical data indicate that tighter control limits can be routinely maintained;

- Calibration/instrument blank must exhibit a response < ½PQL.

Continuing Calibration Verification (CCV) standards are analyzed at the concentration mid-point of the initial calibration. The CCV are analyzed every 10 samples, to insure the continuing validity of the initial calibration. The CCV must agree within ±10 % of the response of the initial calibration to be valid. Sample analyses must be bracketed by calibration verification standards that meet the acceptance criteria. In addition, the Cal/instrument blank, which is analyzed every 10 samples, must exhibit response < ½PQL.

Calibration information is recorded on the computer printout of raw data. The calibration runs are also recorded on the system hard disk.

Ultraviolet-Visible (UV/VIS) Spectrophotometer

The spectrophotometer is calibrated with a minimum of five standards at least annually (some procedures/instruments may require daily calibration), when a new stock standard solution is prepared or when the continuing calibration verification standard varies by greater than ±10% from the initial calibration. All quantitations are carried out using the external standard technique. The initial calibration curve must meet the following criteria:

- The correlation coefficient must be ≥ 0.995 using a regression fit;
- Calibration/instrument blank must exhibit a response < ½PQL.

Continuing Calibration Verification (CCV) standards at the concentration mid point of the initial calibration curve are analyzed immediately following the calibration standards (initial or continuing), after every 10 samples (or after every three samples in the case of sulfate turbidimetric analyses), and at the end of each run. The CCV must agree within ±10 % of the response of the initial calibration to be valid. Data must be bracketed by calibration verification standards that meet control criteria. In addition, the instrument blank, which is analyzed every 10 samples, must exhibit response < ½PQL.

Wavelength calibration checks are performed every three years according to manufacturers' instructions. The process is documented and filed with the instrument manual.

Calibration information is recorded on the computer printout of raw data and on the system hard disk. All initial calibration data is filed by run date and method number. Calibration run dates are recorded on all raw data sample records.

Conductivity Meter

The cell constant of each meter is verified, at a minimum, annually by the analysis of a KCl standard. To verify the instrument operation, a minimum of three standards and a blank are analyzed at the beginning of each working day, using KCl standards in the expected range of the sample. The standard percent recovery must be within ±10% of the known value (except for the 14.9 µmhos/cm standard, which must be ±20% of the known value). For meters not having automatic temperature compensation, samples are either analyzed at 25°C ±2 °C or a manual temperature correction is employed.

Total Organic Carbon (TOC)

A minimum of five calibration standards is analyzed. The concentration of the calibration standards is such that the known or expected linear response range of the instrument is bracketed. The lowest calibration point is equivalent to the practical quantitation limit.

A calibration curve is fitted to the calibration points using least squares techniques by the data processing software. In most cases, a straight-line fit can be achieved. Calibration curves must have a correlation coefficient (r) equal to or greater than 0.995. This is equivalent to a coefficient of determination (r^2) of 0.990.

The continuing calibration verification (CCV) standard is a mid-range standard that is analyzed immediately upon calibration, after every 10 samples and at the end of the analytical batch to verify that the instrument has remained in calibration during sample analysis. The acceptable range of recovery is 90-110%. If the CCV is unacceptable, the instrument must be recalibrated and verified. Sample analyses must be bracketed by acceptable calibration verification standards; therefore, all samples analyzed since the last acceptable CCV must be reanalyzed.

Calibration information is recorded on the computer printout of raw data and on the system hard disk. All initial calibration data is filed by run date and method number. Calibration run dates are recorded on all raw data sample records.

9.4.4 Gas Chromatography (GC)

Volatiles by GC

Volatile organic compounds (VOCs) are analyzed by three protocols: EPA 600 series, EPA 8000 series and the State of California Leaking Underground Fuel Tank Field Manual (CA LUFT-TPH). These analyses are generally performed using external standard calibration and quantitation; therefore the absolute retention time is used to determine the identification of the target compounds. The retention time window is calculated as three times the standard deviation obtained from a 72-hour sequence or default windows of 0.03 minutes are used for compounds where the calculated window is too restrictive or zero. Bracketing by CCV will be required for external standard calibrations as specified in the method or SOP. If internal standard calibration and quantitation is used, the relative retention time, as defined in the respective SOPs, will be used to determine the identification of the target compounds and bracketing by CCV will not be required unless specified in the method.

Initial calibration is performed upon instrument startup and whenever the continuing calibration verification standard fails the acceptance criteria. A calibration curve is prepared for all target compounds with the lowest standard concentration at or below the reporting limit and the remaining standards defining the working range of the detector. After the initial calibration standards are injected, a calibration curve is constructed using either internal standard or external standard methodology. The analyst inspects the curves before proceeding with sample analysis. The correlation coefficient of the calibration curve must be greater than or equal to 0.99. An alternative to quantitation from a calibration curve is quantitation from an average calibration factor or average response factor. If the %RSD is less than or equal to the acceptance criteria, the average response factor can be used for quantitation.

A mid-level calibration verification standard must be analyzed periodically as a check on the validity of the initial calibration. If the percent difference or percent drift is within the acceptance criteria, the curve is acceptable for quantitation of samples.

Calibration Check	600 series	8000 series	CA LUFT - TPH GRO
Initial calibration	Minimum of 3 standards	Minimum of 5 standards	Minimum of 3 standards
%RSD criteria (1)	RF≤10%	CF or RF≤20%	N/A
CCV criteria (%difference or %drift)	Within Q-table values	±15%	±10%
Frequency of CCV	Daily	Every 12 hours	Daily

(1) Alternatively, a regression curve (linear, quadratic, etc.) may be constructed. If the correlation coefficient of the regression curve is greater than or equal to 0.99, the curve may be used for quantitation of samples.

External standard CCV - Samples analyzed by external standard calibration require bracketing by CCV. If the CCV standard analyzed after the samples fails to meet the acceptance criteria and the response of the midpoint standard is above the criteria (that is the response of the analytical system has increased), samples which have no target compounds detected above the RL may be reported as <RL, since the compounds would have been detected if present. (SW-846 Method 8000 B).

Semivolatiles by GC

Semivolatile organic compounds (SVOCs) are analyzed by four protocols: EPA 500 series, EPA 600 series, EPA 8000 series and the State of California Leaking Underground Fuel Tank Field Manual (CA LUFT - TPH). If internal standard calibration is used; relative retention time, as defined in the respective SOPs, will be used to determine the identification of the target compounds and bracketing by CCV will not be required unless specified in the method. If external standard calibration is used, the absolute retention time window is calculated as three times the standard deviation obtained from a 72-hour sequence or default windows of 0.03 minutes are used for compounds where the calculated window is too restrictive or zero. Bracketing by CCV will be required for external calibrations if specified in the method or SOP.

Initial calibration is performed upon instrument startup and whenever the continuing calibration verification standard fails the acceptance criteria. A calibration curve is prepared for all target compounds with the lowest standard concentration at or below the reporting limit and the remaining standards defining the working range of the detector. After the initial calibration standards are injected, a calibration curve is constructed using the internal standard or external standard methodology. The analyst inspects the curves before proceeding with sample analysis. The correlation coefficient of the calibration curve must be greater than or equal to 0.99. An alternative to quantitation from a calibration curve is quantitation from an average response factor. If the %RSD is less than or equal to the acceptance criteria, the average response factor can be used for quantitation.

A mid-level calibration verification standard must be analyzed periodically as a check on the validity of the initial calibration. If the percent difference or percent drift is within the acceptance criteria, the curve is acceptable for quantitation of samples.

Calibration Check	500 series	600 series	8000 series	CA LUFT - TPH DRO
Initial calibration	Minimum of 3 standards (as the calibration range is extended, the number of points must be increased)	Minimum of 3 standards	Minimum of 5 standards	Minimum of 3 standards

%RSD criteria (1)	RF≤20%	RF≤10%	CF or RF≤20%	N/A
CCV criteria (%difference or %drift)	±20%	±15% (non-40 CFR Methods are ±10%)	±15%	±10%
Frequency of CCV	Every 8 hours	Daily	Every 12 hours	Daily

(1) Alternatively, a regression curve (linear, quadratic, etc.) may be constructed. If the correlation coefficient of the regression curve is greater than or equal to 0.99, the curve may be used for quantitation of samples.

External standard CCV - Samples analyzed by external standard calibration require bracketing by CCV. If the CCV standard analyzed after the samples fails to meet the acceptance criteria and the response of the midpoint standard is above the criteria (that is the response of the analytical system has increased), samples which have no target compounds detected above the RL may be reported as <RL, since the compounds would have been detected if present. (SW-846 Method 8000 B).

9.4.5 Gas Chromatography/Mass Spectrometry (GC/MS)

Volatiles by GC/MS

Volatile organic compounds (VOCs) are analyzed by two protocols: 600 series and 8000 series. Hardware tuning is performed on each GC/MS prior to calibration as specified in the applicable EPA methods. Ion abundance acceptance criteria for VOC tuning with BFB are given below. Mass calibration is performed as an integral part of tuning. The tune check and calibration check must be performed daily for the 600 series and every 12 hours for the 8000 series. The tune analysis must meet the criteria listed in EPA methods 624 and 8260 for a 25-ng injection of bromofluorobenzene (BFB).

VOLATILE ORGANIC GC/MS TUNING AND MASS CALIBRATION (BFB)		
Ion Abundance Criteria		
m/e	**624**	**8260**
50	15-40% of mass 95	15-40% of mass 95
75	30-60% of mass 95	30-60% of mass 95
95	Base peak, 100% relative abundance	Base peak, 100% relative abundance
96	5-9% of mass 95	5-9% of mass 95
173	<2% of mass 174	<2% of mass 174
174	>50% of mass 95	>50% of mass 95
175	5-9% of mass 174	5-9% of mass 174
176	>95% but <101% of mass 174	>95% but <101% of mass 174
177	5-9% of mass 176	5-9% of mass 176

Initial calibration is performed upon instrument startup and whenever the continuing calibration verification standard fails the acceptance criteria. A calibration curve is prepared for all target compounds with the lowest standard concentration at or below the reporting limit and the remaining standards defining the working range of the detector. A minimum of 3 levels is required for the 600 series and a minimum of 5 levels is required for the 8000 series.

After the initial calibration standards are injected, a calibration curve is constructed using internal standard methodology. The analyst inspects the curves before proceeding with sample analysis. The correlation coefficient of the calibration curve must be greater than or

equal to 0.99. An alternative to quantitation from a calibration curve is quantitation from an average response factor. If the %RSD is less than or equal to the acceptance criteria, the average response factor can be used for quantitation.

A mid-level calibration verification standard must be analyzed periodically (daily for 600 series and every 12 hours for the 8000 series) as a check on the validity of the initial calibration. If the percent difference or percent drift is within the acceptance criteria, the curve is acceptable for quantitation of samples.

Volatile GC/MS		
Method	**Initial Calibration Check Criteria**	**Continuing Calibration Check Criteria**
624	All targets ≤35% RSD, or alternatively, construct calibration curve.	QC Check Sample (20 µg/L) meets limits specified in method - Table 5, Range for Q
8260	CCC ≤30% RSD Target analytes <15% RSD	CCC ≤20% difference or drift from initial calibration
	SPCC (minimum RF) Chloromethane 0.10 1,1-Dichloroethane 0.10 Bromoform >0.10 Chlorobenzene 0.30 1,1,2,2-Tetrachloroethane 0.30 (0.10 for 25-mL purge) (1) Others ≥0.050	

(1) The purging efficiency of 1,1,2,2-tetrachloroethane relative to the internal standard is such that the SPCC criteria cannot be met consistently for a 25 mL purge. The response factor is generally in the 0.1 to 0.3 range. The alternate criteria is adopted from the EPA CLP Low Level Statement of Work, a protocol similar in scope and application to SW-846 Method 8260.

A Quality Control Check Standard (QCCS) is used to check the accuracy of the initial calibration curve for each compound and to insure that the standards used to generate the curve have maintained their integrity. The QCCS is analyzed every time the instrument is calibrated and during every 12 or 24 hour analytical shift. The QCCS also contains the Calibration Check Compounds (CCCs) and System Performance Check Compounds (SPCCs) so that these checks can be accomplished in a single analysis.

Method 8260 - After the CCC and SPCC are evaluated, all target compounds are evaluated for linearity. If the %RSD is less than or equal to 15%, the average response factor can be used for quantitation. If the %RSD exceeds 15%, a regression curve (linear, quadratic, etc.) may be used for quantitation if the correlation coefficient is greater than 0.99.

Each instrument is calibrated according to the procedures specified within the relevant EPA method. In all cases, the minimum requirements and specifications given in the methods are met or exceeded. A brief description of the calibration requirements and practices of the laboratory are discussed here. Refer to the specific EPA method protocols for additional details.

The internal standard responses and retention times of each standard and sample analyzed are evaluated after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from retention times in the most recent QCCS, then the chromatographic system must be inspected for malfunctions and corrections must be made. If the response for any internal standard varies by more than a factor of two (-50% to +100%) from the most recent calibration sequence, the GC/MS system must be inspected for

malfunctions and corrections must be made, as appropriate. Any standard or sample failing these internal standard checks are re-analyzed. The system is re-calibrated, if necessary.

Analytical standards for the internal standards, surrogates, initial calibration, continuing calibration check, system performance check standards and standard spiking solutions must be certified and NIST- traceable. The standard solutions for the calibration and matrix spiking solutions must be from independent sources. The term "independent source" means that the origin of the standard preparations are known to be different from one another. In practical terms this requires that the solutions be prepared by two different suppliers or at a minimum, have different lot numbers from the same supplier.

Paper copies of the calibration and quantitation reports are stored in a file folder labeled appropriately. All raw electronic data files are initially stored on the MS system hard disk, then later archived to tape or optical disk for permanent storage.

Semivolatiles by GC-MS

Semivolatile compounds are analyzed by two protocols: 600 series and 8000 series. Hardware tuning is performed on each GC/MS prior to calibration as specified in the applicable EPA methods. Ion abundance acceptance criteria for SVOC tuning with a 50-ng injection of decafluorotriphenyl phosphine (DFTPP) are given below. Mass calibration is performed as an integral part of tuning. The tune check and calibration check must be performed daily for 600 series and every 12 hours for 8000 series.

SEMIVOLATILE ORGANIC GC/MS TUNING AND MASS CALIBRATION (DFTPP)		
Ion Abundance Criteria		
m/e	**625**	**8270**
51	30-60% of mass 198	30-60% of mass 198
68	<2%of mass 69	<2% of mass 69
69	(reference only)	(reference only)
70	<2% of mass 69	<2% of mass 60
127	40-60% of mass 198	40-60% of mass 198
197	<1% of mass 198	<1% of mass 198
198	Base peak, 100% relative abundance	Base peak, 100% relative abundance
199	5-9% of mass 198	5-9% of mass 198
275	10-30% of mass 198	10-30% of mass 198
365	>1% of mass 198	>1% of mass 198
441	Present but less than mass 443	Present but less than mass 443
442	>40% of mass 198	>40% of mass 198
443	17-23% of mass 442	17-23% of mass 442

Initial calibration is performed upon instrument startup and whenever the continuing calibration verification standard fails the acceptance criteria. A calibration curve is prepared for all target compounds with the lowest standard concentration at or below the reporting limit and the remaining standards defining the working range of the detector.

After the initial calibration standards are injected, a calibration curve is constructed using internal standard methodology. The analyst inspects the curves before proceeding with sample analysis. The correlation coefficient of the calibration curve must be greater than or equal to 0.99. An alternative to quantitation from a calibration curve is quantitation from an average response factor. If the %RSD is less than or equal to the acceptance criteria, the average response factor can be used for quantitation.

A midpoint calibration verification standard must be analyzed periodically as a check on the validity of the initial calibration. If the percent difference or percent drift is within the acceptance criteria, the curve is acceptable for quantitation of samples.

Semivolatile GC/MS		
Method	**Initial Calibration Check Criteria**	**Continuing Calibration Check Criteria**
625	All targets ≤35% RSD, or alternatively, construct calibration curve.	All targets ≤20% difference from initial calibration.
8270	CCCs ≤30% RSD; RF of SPCCs ≥0.050	RF of CCCs ≤20% difference or drift from initial calibration; RF of SPCCs ≥0.050

SW-846 Method 8270 - After the CCC and SPCC are evaluated, all target compounds are evaluated for linearity. If the %RSD is less than or equal to 15%, the average response factor can be used for quantitation. Alternatively, a regression curve (linear, quadratic, etc.) may be used for quantitation if the correlation coefficient is greater than 0.99.

Each instrument is calibrated according to the procedures specified within the relevant EPA method. Clarification of the calibration requirements and practices of this laboratory are discussed here. Refer to the specific EPA method protocols for additional detail.

The internal standard responses and retention times of each standard and sample analyzed are evaluated after data acquisition. If the retention time for any internal standard changes by more than 30 sec. from the last CCCS, the chromatographic system must be inspected for malfunctions and corrections must be made. If the response for any internal standard varies by more than a factor of two (-50% to +100%) from the last CCCS, the GC/MS system must be inspected for malfunctions and corrections must be made, as appropriate. Any standard or sample failing these internal standard checks is re-analyzed. The system is re-calibrated, if necessary.

Analytical standards for the internal standards, surrogates, initial calibration, continuing calibration, QC check standards and standard spiking solutions must be certified and NIST-traceable. The standard solutions for the calibration and QC Check Standard must be from independent sources. The term “independent source” means that the origins of the standard preparations are known to be different from one another. In practical terms this requires that the solutions be prepared by two different suppliers or at a minimum, have different lot numbers from the same supplier.

Paper copies of the calibration and quantitation reports are stored in a file folder labeled with the initial calibration data file name. All raw electronic data files are initially stored on the MS system hard disk, then later archived to RW CD for permanent storage.

9.5 Standardization of titrating solutions

The titrants for all titrametric procedures are standardized against primary standards before each use. Table 9-3 shows standardization of titrating solutions.

Table 9-1. Calibration Frequency, Procedures, Standards, and Acceptance Criteria for Support Equipment

Instrument/Analyte	Frequency	Procedure	Standard	Acceptance Criteria
pH Meter (primarily for Color and Alkalinity analyses)	Daily	Calibration (2 points)	Vendor Certified Buffer Solutions	Within Certified Values
	Daily	Third buffer Check	Vendor Certified Buffer Solution	± 0.1 pH units
Analytical Balances	Daily	Calibrated according to manufacturer's instructions		Manufacturer specified
	Daily	1 point verification	Class S or equivalent weights	ASTM tolerances
	Quarterly	3 point verification	Class S or equivalent weights	ASTM tolerances
Ovens	Daily	Temperature checked and recorded	NIST traceable thermometer	Varies according to use - see determinative SOP
Incubators	Twice Daily	Temperature checked and recorded	NIST traceable thermometer	Varies according to use - see determinative SOP
Autoclaves	Daily	Maximum temperature and pressure recorded	NIST traceable thermometer and pressure gauge	Varies according to use - see determinative SOP
Water baths	Daily	Temperature checked and recorded	NIST traceable thermometer	Varies according to use - see determinative SOP
Refrigerators	Daily	Temperature checked and recorded	NIST traceable thermometer	1 to 6°C with no evidence of freezing
Freezers	Daily	Temperature checked and recorded	NIST traceable thermometer	-10°C to - 20°C
Thermometers, Hg or spirit-filled	Annually	Verified against an NIST or NIST traceable thermometer	NIST traceable thermometer	Varies according to use - see thermometer calibration SOP
Thermometers, Digital	Quarterly	Verified against an NIST or NIST traceable thermometer	NIST traceable thermometer	Varies according to use - see thermometer calibration SOP
Pipettors	Quarterly	Verified gravimetrically	Class S or equivalent weights	±1% full scale volume

Table 9-2. Calibration Frequency, Procedures, Standards, and Acceptance Criteria for Operational Equipment

Instrument/Analytes	Frequency	Procedure	Standard	Acceptance Criteria
AA spectrophotometer -metals (flame) -metals (furnace) -mercury (cold vapor) ***ICP spectrophotometer***	Daily or failure of CCV	Calibration (4-6 points)	Vendor certified standard. Plasma grade-ICP	Correlation coefficient >0.995
	Immediately following calibration, 10% and end of run	ICV/CCV	Mid-range calibrant	±5% immediately following calibration then ±10% of initial value after every 10 samples and at the end of the run.
	Daily following CCV and at end of run	Second source QC	Certified reference material	±10% of true value
Ion chromatograph	Daily or failure of ICV/CCV	Calibration (3-5 points)	Vendor certified standards	Correlation coefficient > 0.995
	Immediately following calibration and end of run.	CCV	Mid-range calibrant	±5% initially ±10% thereafter
Autoanalyzers	Daily	Calibration (6-8 points)	Reagent grade chemicals	Correlation coefficient > 0.995
	10% and end of run	CCV	Mid-range calibrant	±10%
	10-20%	Second source QC	Certified reference material	Within certified values
	10% and end of run	Cd column check	Nitrate standard	±10% of true value
pH meter	Daily	Calibration (2-3 points)	Vendor certified buffers	Within certified values
	Following calibration	Mid point check	Vendor certified buffers	Within ±0.1 pH units
Conductivity meter	Daily	Calibration verification (3 points)	Vendor certified standards	±10% of certified values
	Annually	Cell constant verification	Vendor certified standards	±10% of certified values
Spectrophotometer	Daily	Calibration (3 to 5 points)	Vendor certified standards	Correlation coefficient >0.995
	Annually	Calibration (5 points)	Vendor certified standards	Correlation coefficient >0.995
	Daily	Second source QC	Certified reference material	±10% of certified value
Turbidity meter	Daily	Calibration check (Each range used)	Secondary sealed standard	±10%
	Monthly	Calibration (3 NTU levels)	Primary calibration standards	±10%
DO meter	Weekly	Barometric pressure calibration	Barometer	
Fluorometer	Daily	Calibration (1 point)	Chlorophyll *a* standard	±10%
	Daily	QCS	Chlorophyll *a* standard	±5%

Table 9-2. ***cont'd.***

GC Semivolatiles	Initially and every 12 or 24 hours	Injection port contamination check	DDT/Endrin	<15% degradation
	Initially or upon failure of CCC	Calibration (3 to 5 points)	Vendor certified standards	Coefficient of determination >0.990
	After initial calibration	Second source standard	Vendor certified standard	<20% difference
	Every 12 or 24 hours	CCC	Mid-level standard	<20% difference
GC Volatiles	Initially or upon failure of CCC	Calibration (5 points)	Vendor certified standards	Coefficient of determination >0.990
	Every 12 or 24 hours	CCC	Mid-level standard	<15% difference
	Every run or every 12 hours	Second source standard	Vendor certified standard	<20% of true value
GC/MS Semivolatiles	Every 12 or 24 hours	Instrument tune	DFTPP	Method specified criteria
	Initially, upon failure of CCC and every 12 or 24 hours	Calibration (3 to 5 points) SPCC CCC	Vendor certified standards	SPCC minimum RF 0.05 CCC $\leq$30% RSD
	After initial calibration and at end of batch	Second source QC	Vendor certified standard	$\leq$30% difference
GC/MS Volatiles	Every 12 or 24 hours	Instrument tune	BFB	Method specified criteria
	Initially and upon failure of CCC	Calibration (3 to 5 points)	Vendor certified standard	Minimum RF 0.05
	After initial calibration and end of every 12 or 24 hour run	Second source QC	Vendor certified standard	$\leq$30% difference from initial calibration
	Every 12 or 24 hours	CCC	50 ppb calibrant	<30% difference form initial calibration
	Every 12 or 24 hours	SPCC	50 ppb calibrant	Minimum RF 0.3
Fluoride meter	Daily	Calibration (3 points)	Vendor certified standard	Correlation coefficient > 0.995
	Daily	Second source QC	Vendor certified standard	Within certified value
	Daily	Slope check	Calibrants	54 ± 4 mV
TOC analyzer	Daily	Calibration (3 points)	Vendor certified standard	Correlation coefficient > 0.995
	Daily	Second source QC	Vendor certified standard	Within certified value
ICP/MS	Daily or failure of CCV	Calibration (4 points)	Vendor certified standard.	Correlation coefficient >0.995
	Immediately following calibration, 10% and end of run	ICV/CCV	Mid-range calibrant	±10% immediately following calibration then ±15% of initial value after every 10 samples and at the end of the run.
	Daily following CCV and at end of run.	Second source QC	Certified reference material	±10% of true value

Table 9-3. Standardization of Titrating Solutions.

Titrating Solution	Primary Standard	Source of Primary Standard	Frequency of Standardization
Sulfuric Acid	Sodium carbonate solution	Commercial supplier	Every 7 days.
EDTA	Calcium carbonate solution	Commercial supplier	Each day of use.
Sodium Thiosulfate	Potassium bi-iodate solution	Commercial supplier	Every month.
Mercuric Nitrate	Sodium chloride solution	Commercial supplier	Each day of use.
Silver Nitrate	Sodium chloride solution	Commercial supplier	Each day of use.
Cyanide Standard	Silver nitrate solution	Commercial supplier	Each day of use.
Ferrous Ammonium Sulfate (FAS)	Potassium Dichromate Solution	Prepared in lab from reagent grade source	Each day of use.
Formaldehyde	0.1N HCl	Prepared and standardized in lab from reagent grade source	Every 6 months.
Phenol	0.025N Sodium Thiosulfate	Prepared and standardized in lab from reagent grade source	Each day of use.

10.0 Preventive Maintenance

The Central Laboratory is equipped with computerized instrumentation. A preventive maintenance schedule has been developed to minimize instrument downtime, and to obtain reliable data over the life of the instrument. Analysts and supervisors are primarily responsible for routine maintenance and repair of the instruments. Service agreements are kept for some major instruments in the laboratory. Major repairs that go beyond the expertise of the analysts, Supervisors and Managers are contracted to external specialists.

Table 10.1 lists the types of analytical equipment utilized to perform analyses and the frequency of routine preventive maintenance tasks performed to ensure data quality. The service intervals are designated as follows: D = daily; W = weekly; M = monthly; Q = quarterly; SA = semi-annually; A = annually; AN = as needed. The preventive maintenance schedules are based primarily on manufacturer guidance, recommendation in the literature, and the experience of the analysts, Supervisors and Managers. Some of the items will be performed as an integral part of each procedure. Others will be followed as closely as possible, balancing to the extent possible the workload and the urgency of the need for preventive maintenance. Common sense and familiarity with the performance of each instrument will dictate whether the preventive maintenance schedule needs to be accelerated or delayed for that instrument. Trends and excursions from accepted quality assurance requirements such as QC sample results, degradation of peak resolution, a shift in the calibration curve, and loss of sensitivity are monitored to determine if there is instrument malfunction, and in such cases preventive maintenance is provided on an as-needed basis.

10.1 Documentation

An instrument maintenance logbook documenting instrument problems, instrument repair and maintenance activities shall be kept for all major pieces of equipment. It is the responsibility of each Unit Supervisor to ensure that instrument maintenance logs are kept for all equipment in his/her Unit. Documentation must include all major maintenance activities such as contracted preventive maintenance and service, and in-house activities such as the replacement of electrical components. An extensive spare parts inventory is maintained for routine repairs at the laboratory facilities, consisting of GC columns, AA lamps, fuses, printer heads, tubing, and other instrument components or adjustment to instrument settings. Entries must include the date, the problem, the corrective actions taken, the name of the person performing the service and when appropriate, a statement that the instrument has returned to control and is available for use (also state what was used to determine a return to control - e.g., CCV acceptable). When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be stapled into the logbooks adjacent to pages describing the maintenance performed.

10.2 Contingency Plan

The laboratory has several pieces of analytical equipment in duplicate. This redundancy allows the laboratory to keep performing critical analyses on one instrument while the other is out of service.

In the event of instrument failure or if critical holding times are approaching, the following options exist:

1. Portions of the sample load may be diverted to duplicate instruments within a facility.
2. The analytical technique may be switched to an alternate approved technique (e.g., Total Hardness by ICP to titration).
3. Samples may be shipped to another State lab. When shipping samples to another facility, COC procedures are followed as required.

In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacture for repair. Back up instruments, which have been approved for the analysis,

shall perform the analysis normally carried out by the malfunctioning instrument. If the back up is not available and the analysis cannot be carried out within the needed timeframe, sample collection personnel may be asked to postpone sampling events or to send the samples to a certified commercial laboratory.

Any item of equipment which has been subjected to overloading or mishandling, which gives suspect results, or has been shown to be defective shall be taken out of service. The instrument will be clearly identified and, wherever possible, stored in a different location until it has been repaired and shown by calibration, verification or test to perform satisfactorily. The laboratory shall examine the effect of this defect on previous calibrations or tests.

10.3 Uninterruptible Power Supply

As a further precaution, the Central Laboratory keeps some major instrumentation connected to individual Uninterruptible Power Supply (UPS) units which provide line conditioning and backup power.

Table 10-1. Laboratory Equipment Preventive Maintenance Schedule.

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
Wet Chemistry	SERVICE INTERVAL							
EQUIPMENT/INSTRUMENT	**D**	**W**	**M**	**Q**	**SA**	**A**	**AN**	**SERVICE**
Conductivity Meter (YSI)								
Cell							X	Replatinize cell when 1 µmho/cm range exceeds 90-100%, and when erratic readings cannot be corrected.
Check Standard Calibration	X							Make new solutions
UV/VIS Spectrophotometers (Shimadzu, Milton Roy)								
Wavelength					X			Verify wavelength(s)
Cells	X							Inspect daily for chips/scratches
Lamps							X	Replace if blown and realign
Ion Selective Electrodes (Orion)								
Electrode (Fluoride)							X	Polish.
Probe (Fluoride)							X	Fill with Single Junction Reference Electrode Filling Solution
Probe (pH)							X	Add 4*M* KCl solution if cell is low
Ionanalzyer (Orion)								
ATC						X		Verify against NIST thermometer
Fluorometer (Turner Designs)								
Meter	X							Calibrated with primary standard and checked with secondary standard (solid)
Lamp							X	Replace if blown and realign
Analytical Balances (Sartorious)								
Balance Calibration	X							Verify calibration with Class S weights
Balance						X		Checked and adjusted by service contractor
Weights						X		Checked against Class S weights
Centrifuges (Damon, Beckman Coulter)								
Centrifuge operation							X	Check warranty
Compartment							X	Clean
8" Drill Press benchtop (Chlorophyll grinder)								
Drill press operation							X	Check warranty
Thermometers								
Hach COD Reactor						X		Verify against NIST thermometer
Convection Ovens						X		Verify against NIST thermometer
Waterbaths								
Compartment							X	Clean with hot soapy water, fill with DI water
Thermometer						X		Verify against NIST thermometer

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
Wet Chemistry continued	SERVICE INTERVAL							
EQUIPMENT/INSTRUMENT	**D**	**W**	**M**	**Q**	**SA**	**A**	**AN**	**SERVICE**
Freezer								
Cleaning							X	Defrost and clean with hot soapy water
Thermometer						X		Verify against NIST thermometer
Cooler								
Cleaning							X	Sweep and mop
Stored samples		X						Discard samples by Discard List
Thermometer						X		Verify against NIST thermometer
LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
Nutrients	SERVICE INTERVAL							
EQUIPMENT/INSTRUMENT	**D**	**W**	**M**	**Q**	**SA**	**A**	**AN**	**SERVICE**
Flow Injection Auto Analyzers and Ion Chromatograph (Lachat)								
Flow cell – flare tubing and o-rings						X		Replace.
Manifold Tubing						X		Replace.
Pump Tubing			X					Replace.
Manifold / Valve o-rings					X			Replace.
Pump and pump cartridges			X					Inspect and Clean.
Transmission / Waste tubing							X	Replace.
Cadmium column							X	Replace.
IC Guard Column					X			Replace.
IC Eluent Pump			X					Check, replace if needed.
Autoclave								
Pressure verification	X							Check and document; replace seals as needed.
Temperature verification	X							Check with autoclave thermometer; document
Cleaning			X					Wash with soapy water; visually inspect for leaks and degradation.
Seals							X	Visually inspect and replace as needed.
Timing				X				Check with stopwatch
Block Digestor								
Digestion Block	X							Inspect and clean using DI water.
Digestion Tubes							X	Replace with new tube(s).
Digestion Tubes, Cold Fingers	X							Clean and check for cracks.
Timing							X	Check with watch, adjust as needed.
Racks			X					Clean.
pH Meter								
Probe			X					Inspect, add/replace KCl filling solution.
Probe	X							When not in use, keep lower end of probe in beaker of standard 6.86.
pH buffer standards							X	Prepare as needed.
ATC						X		Verify with NIST thermometer

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
Nutrients continued	SERVICE INTERVAL							
EQUIPMENT/INSTRUMENT	**D**	**W**	**M**	**Q**	**SA**	**A**	**AN**	**SERVICE**
Digital Balance								
Balance Pan	X							Clean.
Balance Level	X							Check that balance is level.
Calibration	X							Check with standard weights, each day used.
Balance						X		Contract service/cleaning
Weights						X		Verify against Class S weights
Ultrasonic Cleaner								
Solution in Tank	X							Maintain correct level; renew solution as needed.
Tank							X	Empty, clean with warm water, and wipe with non-abrasive cloth.
Reagent/Standard Refrigerator								
Temperature	X							Verify temp with thermometer.
Shelves					X			Clean.

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
Microbiology	SERVICE INTERVAL							
EQUIPMENT/INSTRUMENT	**D**	**W**	**M**	**Q**	**SA**	**A**	**AN**	**SERVICE**
BOD Meter								
Probe (electrolyte)		X						Change solution.
Probe (membrane)							X	Replace membrane.
Barometer			X					Calibrate.
Turbidimeter								
Meter	X							Verify calibration with sealed standards.
Lamp							X	Replace.
Meter			X					Calibrate with sealed HF Scientific standards
TOC Analyzer								
Carrier gas.	X							Check flow. Should be 200 cc/min $\pm$10%
DDI H_2O	X							Replace.
Corrosive scrubber (Cu+Sn)	X							Check for tarnish. Replace as needed.
8-port valve thumbscrews	X							Hand-tighten.
IC sparger		X						Clean with mild soap and water.
Sparger & water traps	X							Empty.
Permeation dryer	X							Inspect for damage or water accumulation.
Combustion tube and/or catalyst							X	Change and/or repack.
Baseline							X	Adjust.
Microscope								
Lens							X	Clean.
Lamp							X	Replace.

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
Microbiology continued	SERVICE INTERVAL							
EQUIPMENT/INSTRUMENT	**D**	**W**	**M**	**Q**	**SA**	**A**	**AN**	**SERVICE**
Incubator (Coliform)								
Temperature	X							Check and document daily
Thermometer						X		Verify against NIST thermometer
Compartment			X					Clean
Coils						X		Clean coils
Analytical Balance								
Balance Calibration	X							Verify calibration with Class S weights
Balance						X		Contract service/cleaning
Weights						X		Verify against Class S weights
Autoclave								
Pressure verification	X							Check and document; replace seals as needed.
Temperature	X							Check and document
Temperature verification		X						Check with maximum hold thermometer
Cleaning			X					Wash with soapy water; visually inspect for leaks and degradation.
Seals							X	Visually inspect and replace as needed.
Timing				X				Check with stopwatch; replace as needed.

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
Metals	SERVICE INTERVAL							
EQUIPMENT/INSTRUMENT	**D**	**W**	**M**	**Q**	**SA**	**A**	**AN**	**SERVICE**
ICP – Optima 3000 XL								
Pump tubing	X							Replace every 8 hours of operation.
Peristaltic pump and drain	X							Check that drain tube is firmly attached to spray chamber drain fitting and liquid flows smoothly through pump.
Inspect waste and rinse water fluid levels	X							Empty or fill as needed.
Nebulizer							X	Clean.
Filters			X					Inspect monthly, clean or replace as needed.
Spray Chamber							X	Clean.
Optical Window			X					Clean or replace if needed.
Quartz torch							X	Clean and align.
Circulating cooler		X						Check water supply and for dust buildup on cooling coils.
Replace torch							X	Replace with new quartz tube and o-rings. Perform X-Y align.
Air Supply for Shear Gas	X							Check pressure and for condensation in traps. Output pressure should be a minimum of 60 PSI.

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
Metals continued	SERVICE INTERVAL							
EQUIPMENT/INSTRUMENT	**D**	**W**	**M**	**Q**	**SA**	**A**	**AN**	**SERVICE**
Liquid argon tanks attached to manifold system	X							Insure gas supply will last the day and there is sufficient pressure (90-120 PSI).
Nitrogen Tank	X							Insure gas supply will last the day. Output pressure should be a minimum of 40 PSI.
THGA Graphite Furnace and AS-800 Autosampler								
Graphite tubes	X							Inspect for deposits around injection hole and cracks in tube. Clean or replace as needed.
Graphite contacts	X							Inspect for deposits and cracks in the contacts. Clean or replace as needed.
Furnace windows							X	Clean or replace as needed.
Water level in cooling system		X						Make sure water level is at the max.
Autosampler external surfaces		X						Wipe over the surfaces with a damp lint-free cloth
Complete rinsing system	X							Fill and flush the rinsing system before the start of every analysis run.
Valves							X	Clean or replace seals, valves are covered under maintenance agreement.
Wash bottle	X							Check daily and empty as needed.
Rinse bottle	X							Make sure rinse bottle is filled with 18-MΩ water.
Pipet tip	X							Check pipet tip for damage and repair or replace.
Argon gas (UHP or 99.996% purity)	X							Outlet gauge minimum pressure is 50 PSI and maximum 58 PSI.
Special gas (95% Ar + 5% H)	x							Outlet gauge minimum pressure is 50 PSI and maximum 58 PSI.
Mercury Analyzer FIMS 400								
Pump tubing	X							Inspect daily and replace as needed.
FIMS-cell window							X	Measure the absorbance of the cell windows regularly, if >0.75, clean.
FIMS-cell inner surface							X	Clean if sensitivity drops not attributable to other factors.
Air filter						X		Replace sooner if needed.
Waste bottle	X							Empty after each analytical run.
FIAS-valve							X	Take apart and clean per maintenance manual.
Argon gas (UHP or 99.996% purity)	X							Outlet gauge pressure is 52 PSI.
Fume trap (for fumes emitted from FIMS-cell)	X							Change charcoal in trap as needed.
Elan 6100 ICP/MS								
Pump tubing	X							Replace every 8 hours of operation.

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE										
Metals continued	SERVICE INTERVAL									
EQUIPMENT/INSTRUMENT	**D**	**W**	**M**	**Q**	**SA**	**A**	**AN**	**SERVICE**		
Peristaltic pump and drain	X							Check that drain tube is firmly attached to spray chamber drain fitting and liquid flows smoothly through pump.		
Nebulizer							X	Clean.		
Filters			X					Inspect monthly, clean or replace as needed.		
Spray Chamber							X	Clean.		
Liquid argon tank	X							Insure gas supply will last the day and there is sufficient pressure (90-120 PSI).		
Inspect waste and rinse water fluid levels	X							Empty or fill as needed.		
Inspect roughing pump oil level and cooler	X							Add or change if dark brown color.		
Inspect condition of drain and rinse station pump tubing							X	Replace if needed.		
Vacuum pressure (Plasma On)	X							Pressure should be around 1.60E-05. Lower pressure may require interface cones to be replaced.		
Daily performance check	X							Take corrective actions necessary to pass. See table below.		
Daily performance check list								Analyte	Mass	Intensities (cps)
								Mg	4	>20,000
								Rh	102.9	>150,000
								In	114.9	>300,000
								Pb	208.0	>100,000
								Ba++/Ba+	69	<0.03
								CeO/Ce	155.9	<0.03
								Bkgd	220.0 amu	<30
X-Y optimization							X	Usually after changing torch = or interface cones.		
Nebulizer optimization							X	To increase sensitivity.		
Auto lens optimization							X	To increase sensitivity and after changing out the lens.		
Mass calibration and resolution			X					Use tuning solution and instrument software to calibrate the mass and adjust peak resolution.		
Dual detector calibration							X	Use multi-element standard solution and software to perform dual detector calibration		
Circulating cooler		X						Check coolant and for dust buildup on cooling coils.		

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
Volatile Organics	SERVICE INTERVAL							
EQUIPMENT/INSTRUMENT	**D**	**W**	**M**	**Q**	**SA**	**A**	**AN**	**SERVICE**
Gad Chromatograph - VOA								
ELCD reactor temp.	X							Inspect daily.
ELCD reaction tube							X	Replace as needed.

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
Volatile Organics continued	SERVICE INTERVAL							
EQUIPMENT/INSTRUMENT	**D**	**W**	**M**	**Q**	**SA**	**A**	**AN**	**SERVICE**
ELCD propanol flow	X							Inspect daily and measure as needed.
ELCD propanol							X	Replenish as needed.
ELCD resin bed							X	Replace as needed.
Check PID sensitivity	X							Check daily standard and adjust as needed.
Change PID lamp							X	Replace.
Chromatographic column							X	Replace or cut as needed.
Leak check							X	Check column and fittings as needed/drift or poor sensitivity
Inlet Septum							X	Replace as needed.
Gas Cylinders	X							Inspect daily, change when pressure reads < 500 psi.
Hydrocarbon/Moisture Trap					X			Replace.
Teflon transfer line								Replace as needed.
Heated transfer lines							X	Bake as needed.
Gas Chromatograph/Mass Spectrometer - VOA								
Inlet septum							X	Replace as needed
GC Column							X	Replace/cut as needed/poor sensitivity
Filament							X	Replace as needed/poor sensitivity
MS Source							X	Clean as needed/poor sensitivity
Leak check pumps			X					Inspect visually and Standard Spectral Tune
Pump fluid					X			Replace pump fluid
Calibration vial					X			Check level and refill as needed.
Inlet liner and O-rings							X	Replace as needed/contamination
System check		X						Standard Spectral Tune
Check gas flow							X	As needed
Gas Cylinder	X							Inspect daily, change when pressure reads <500 psi.
Hydrocarbon/Moisture Trap			X					Replace.
Purge and Trap								
Disposable purge tubes	X							Replace
Sorbent trap							X	Change as needed/poor sensitivity
Purge flow					X			Inspect semi-annually; adjust as needed.
Rinse purge ports	X							Use charcoal filtered water.
Leak check lines							X	As needed/poor sensitivity
Bake system and transfer lines							X	As needed/ contamination

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
Pesticides	SERVICE INTERVAL							
EQUIPMENT/INSTRUMENT	**D**	**W**	**M**	**Q**	**SA**	**A**	**AN**	**SERVICE**
Gas Chromatograph - PESTICIDES								
Column							X	Replace.
Septum		X						Replace
Gas Cylinder	X							Inspect daily, change when pressure reads < 300 psi.

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
Pesticides continued	SERVICE INTERVAL							
EQUIPMENT/INSTRUMENT	**D**	**W**	**M**	**Q**	**SA**	**A**	**AN**	**SERVICE**
Hydrocarbon/Moisture Trap						X		Replace.
Inlet liner			X					Replace
Automated Sample Processing System (GPC)								
Column							X	Resolvate.
Gas Cylinder	X							Inspect daily, change when pressure reads <300psi.
Methylene Chloride reservoir	X							Add solvent.
LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
Semivolatile Organics	SERVICE INTERVAL							
EQUIPMENT/INSTRUMENT	**D**	**W**	**M**	**Q**	**SA**	**A**	**AN**	**SERVICE**
Gas Chromatographs - SVOA								
Column			X				X	Cut off 1 foot or Replace.
Septum			X				X	Replace
Gas Cylinder		X					X	Inspect gauge change when pressure reads < 200 psi.
Hydrocarbon/Moisture Trap						X	X	Replace.
Inlet, inlet liner			X				X	Clean, replace and clean
FID						X	X	Clean, replace jet
Wash Bottles	X						X	Inspect, refill, replace, clean
Syringe	X						X	Inspect, replace
Check gas leaks							X	After column change
Mass Spectrometer - SVOA								
Run Standard Spectrum tune							X	Check base line operation, air leaks, tighten vacuum system
Vacuum Pump		X				X	X	Check oil level, change oil
Ion source	X						X	Check, Clean when performance not in controls
LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
Asheville Regional Laboratory	SERVICE INTERVAL							
EQUIPMENT/INSTRUMENT	**D**	**W**	**M**	**Q**	**SA**	**A**	**AN**	**SERVICE**
BOD Meter								
Probe (electrolyte)		X						Change solution
Probe (membrane)							X	Replace membrane
Barometer			X					Calibrate
Turbidimeter								
Meter	X							Verify calibration with sealed standards
Lamp							X	Replace
Meter			X					Calibrate with sealed HF Scientific standards.
Microscope								
Lens							X	Clean.
Lamp							X	Replace

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
Asheville Regional Laboratory continued	SERVICE INTERVAL							
EQUIPMENT/INSTRUMENT	**D**	**W**	**M**	**Q**	**SA**	**A**	**AN**	**SERVICE**
Orion 920A Meter								
pH Probe							X	Clean, add filling solution
Ammonia Probe		X						Change membrane & filling solution
ATC						X		Calibrate against NIST thermometer
Analytical Balance								
Balance Calibration	X							Verify calibration with Class S weights
Balance						X		Contract service/cleaning
Weights						X		Verify against Class S weights
Balance Pan	X							Clean
Balance Level	X							Check that balance is level
DI Water System								
Filters							X	Change.
System							X	Contract service.
Autoclave								
Pressure verification	X							Check and document; replace seals as needed
Temperature	X							Check and document
Temperature verification		X						Check with maximum hold thermometer
Cleaning			X					Wash with soapy water; visually inspect for leaks and degradation; add DI water
Seals							X	Visually inspect and replace as needed
Timing				X				Check with stopwatch
Incubators (BOD)								
Temperature	X							Check and document daily
Thermometer						X		Verify against NIST thermometer
Compartment			X					Clean
Coils						X		Clean coils
Incubator (Coliform)								
Temperature	X							Check and document daily
Thermometer						X		Verify against NIST thermometer
Compartment			X					Clean
Coils						X		Clean coils
Refrigerators (sample storage)								
Temperature	X							Check and document daily
Thermometer						X		Verify against NIST thermometer
Compartment			X					Clean
Coils						X		Clean

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
Washington Regional Laboratory	SERVICE INTERVAL							
EQUIPMENT/INSTRUMENT	**D**	**W**	**M**	**Q**	**SA**	**A**	**AN**	**SERVICE**
Autoclave								
Pressure verification	X							Check and document; replace seals as needed
Temperature	X							Check and document
Temperature verification		X						Check with maximum hold thermometer
Cleaning			X					Wash with soapy water; visually inspect for leaks and degradation; add DI water
Seals							X	Visually inspect and replace as needed
Timing				X				Check with stopwatch
BOD Meter								
Probe (electrolyte)		X						Change solution
Probe (membrane)							X	Replace membrane
Barometer			X					Calibrate
De-Ionizing Water System								
Canisters					X			Replace.
Conductivity	X							Check if within limits
Incubators (BOD)								
Temperature	X							Check and document daily
Thermometer						X		Verify against NIST thermometer
Compartment			X					Clean
Coils						X		Clean coils
Incubator (Coliform)								
Temperature	X							Check and document daily
Thermometer						X		Verify against NIST thermometer
Compartment			X					Clean
Coils						X		Clean coils
Ovens (residue)								
Temperature	X							Check and document daily
Thermometer						X		Verify against NIST thermometer
Compartment			X					Clean
Muffle Furnace (residue)								
Temperature	X							Check and document daily
Thermometer						X		Verify against NIST thermometer
Compartment			X					Clean
pH Meter								
probe	X							Calibrate
							X	Clean
Refrigerators (sample storage)								
Temperature	X							Check and document daily

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
Washington Regional Laboratory continued	SERVICE INTERVAL							
EQUIPMENT/INSTRUMENT	**D**	**W**	**M**	**Q**	**SA**	**A**	**AN**	**SERVICE**
Thermometer						X		Verify against NIST thermometer
Compartment			X					Clean
Coils						X		Clean
Turbidimeter								
Meter	X							Verify calibration with sealed standards
Lamp							X	Replace
Meter			X					Calibrate with sealed HF Scientific standards.
Vacuum Pump								
Oil						X		Change oil
Pump					X			Add oil and de-water
Water bath (Coliform)								
Thermometer						X		Verify against NIST thermometer
Bath				X				Clean and change water
Water							X	Add water
Water distillation system								
Still			X					Clean
Heating elements							X	Change
Microscope								
Lens							X	Clean.
Lamp							X	Replace.
Analytical Balance								
Balance Calibration	X							Verify calibration with Class S weights
Balance						X		Contract service/cleaning
Weights						X		Verify against Class S weights
Balance Pan	X							Clean
Balance Level	X							Check that balance is level

11.0 Quality Control Checks and Routines to Assess Precision and Accuracy and Calculation of Method Detection Limits

The key to a successful QA/QC program is strict adherence to the program during all phases of the project including pre-sampling discussions, sample collection, preservation, storage and analysis, and validation and reporting of results. Field and laboratory quality control checks are a part of each sampling trip and laboratory analysis. Quality control checks are used to establish quality assurance objectives in the laboratory (see Section 5). Once the quality assurance objectives are set, QC samples and elements are used to continuously monitor the quality of the data against those objectives. By using laboratory QA targets and QC check results, the user knows the limits of data precision and accuracy and if these objectives were met for a given set of data.

11.1 QC Checks

QC samples must be scheduled with each batch of samples of a given matrix analyzed for a given parameter. This section discusses the QC checks used by the Laboratory Section on a routine basis. However, the analytical methods used and occasionally the client define the QC checks that are required for each test. If the quality control requirements of a particular method or client are more stringent than those presented here, the requirements of that method or client will be followed.

11.1.1 Field QC Checks

When field QC sample collection and analysis are required for a project, it is the responsibility of the sampling personnel to ensure that this sampling is performed correctly and at the required frequency. Field QC samples may or may not be identified as such to the laboratory and are considered by the laboratory as field samples for the purpose of QC batching, sample preparation and analysis. Field QC sample results are reported in the same manner as actual field samples, unless a specific deliverable is requested by a client. No correction of the analytical data for associated field samples is done in the laboratory based on the analysis of field QC samples. Recommended field QC may include field duplicates, split samples, field blanks, equipment blanks and trip blanks. Trip blanks are the only field QC required for sample submission to the Laboratory Section. When VOA samples are received without an associated trip blank, a Sample Condition Upon Receipt report is completed and the collector is notified immediately of the infraction. Re-sampling is generally recommended. Any contamination problem discovered in a trip blank initiates an immediate investigation which generally involves comparison with the associated batch method blanks and discussion with the sample submitter. A description of the preparation and handling of trip blanks follows.

Trip Blanks

Volatile organic samples are susceptible to contamination by diffusion of organic contaminants through the septum of the sample vial. A trip blank must accompany volatile organic samples. The purpose is to determine if contamination has occurred as a result of improper sample container cleaning, contaminated blank source water, sample contamination during storage and transportation due to exposure to volatile organics (e.g., gasoline fumes) and other environmental conditions during the sampling event and subsequent transportation to the lab. Trip blanks are prepared prior to the sampling event either by the laboratory providing sample containers, or by sample collection personnel who are responsible for the initial preparation of sample containers and field equipment. Trip blanks are prepared by filling 40 mL VOA vials (with no headspace) with organic-free water. Any appropriate preservatives must be added at the time that the blanks are prepared. The sample containers are sealed, labeled appropriately, and transported to the field in the same manner as the sample vials. These blanks are NOT to be opened in the

field. They are to be transferred to the sample cooler and transported with the samples to the laboratory. Trip blanks are prepared for each cooler expected to be used to store and transport VOA samples.

11.1.2 Laboratory QC Checks

Laboratory performance QC is required to ensure the laboratory systems (instrumentation, samples preparation, analysis, data reduction, etc.) are operating within acceptable QC guidelines during data generation as required to meet method requirements or the client's objectives. Determination of the validity of sample results is based on the acceptance criteria being met by the control samples. The acceptance criteria for each type of control samples are defined in the appropriate SOP. These acceptance criteria are per method requirements or calculated annually from historical data.

Laboratory QC samples consist of method blanks, instrument blanks, laboratory control samples and calibration verification samples. In addition to laboratory performance QC, matrix-specific QC is utilized to determine the effect of the sample matrix on the data being generated. Typically, this includes matrix spikes matrix spike duplicates, sample duplicates and the use of surrogate compounds. Following is a brief description of these QC checks.

Batch
Environmental samples which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A ***preparation batch*** is composed of one to 20 environmental samples of the same matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An ***analytical batch*** is composed of prepared environmental samples (extracts, digestates or concentrates) and/or those samples not requiring preparation, which are analyzed together as a group using the same calibration curve or factor. An analytical batch can include samples originating from various environmental matrices and can exceed 20 samples.

Blind sample
A sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

Calibration
To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other device, or the correct value for each setting of a control knob. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements.

Initial Calibration Verification Standard
An Initial Calibration Verification Standard (ICV) is a second source standard analyzed immediately following calibration and indicates whether sample analysis can proceed.

Continuing Calibration Verification Standard (equivalent to Calibration Check Standard or CCC)
A Continuing Calibration Verification Standard (CCV) is an analytical standard that is reanalyzed with test samples to verify calibration of the analytical system. CCVs are usually mid-range standards that are analyzed at the beginning and end of an analytical run and after every 10 or 20 samples for large analytical runs.
Confirmation

A confirmation shall be performed to verify the compound identification when positive results are detected in a sample from a location that has not been previously tested by the laboratory. Such confirmations shall be performed on organic tests such as pesticides, herbicides or acid extractable or when recommended by the analytical test method except when the analysis involves the use of a mass spectrometer.

When samples results are confirmed using two dissimilar columns or with two dissimilar detectors, the agreement between the quantitative results should be evaluated after the identification has been confirmed. Calculate the relative percent difference (RPD) between the results using the formula described in Section 12 where R1 and R2 are the results for the two columns and the vertical bars in the equation indicate the absolute value of the difference. Therefore, RPD is always a positive value.

If one result is significantly higher (e.g., >40%), check the chromatograms to see if an obviously overlapping peak is causing an erroneously high result. If not overlapping peaks are noted, examine the baseline parameters established by the instrument data system (or analyst) during peak integration.

If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems, report the higher results. This approach is conservative relative to protection of the environment. The data user should be advised of the disparity between the results of the two columns.

Method Blank (equivalent to a laboratory reagent blank or LRB)
The method blank is a QC sample that consists of all reagents specific to the method and is carried through every aspect of the procedure, including preparation, cleanup and analysis. The method blank is used to identify any interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. Potential sources of contamination include solvent, reagents, glassware, other sample processing hardware, or the laboratory environment. In general, the method blank is a volume of deionized laboratory water or well water for water samples, or Ottawa sand for soil/sediment samples, that is processed as a sample. The volume or weight of the method blank must be approximately equal to the sample volume or sample weight processed. A method blank shall be prepared with each group of samples processed. Method blanks are also referred to as laboratory reagent blanks.

The source of contamination must be investigated and measures taken to correct, minimize or eliminate the problem if the blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated sample batch. Any sample associated with the contaminated blank shall be reprocessed for analysis or the results reported with the appropriate data qualifier code.

Instrument Blank
The instrument blank is an unprocessed aliquot of reagent water (or a dry tube purge as in the case of volatile organics analyses) used to monitor the contamination of the analytical system at the instrument. System contamination may lead to the reporting of elevated analyte concentrations or false positive data. The instrument blank does not undergo the entire analytical process and generally consist of an aliquot of the same reagent(s) used for a sample dilution. Instrument blanks are also referred to as continuing calibration blanks.

Laboratory control sample (equivalent to a laboratory fortified blank or LFB)
A laboratory control sample (LCS) is a sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes. It

is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

The fortified blank is analyzed exactly like a sample. Fortified blanks are used to obtain a recovery from the solution used to spike a matrix spike sample. Results are used to validate or reject matrix spike recovery results. A low or high sample matrix spike recovery can be justified if the fortified blank also shows a similar bias and all other QC data is acceptable. This may indicate analyst error in the preparation of the spiking solution. If sample recovery results are outside control limits and the fortified blank recovery results are acceptable it is reasonable to assume a sample matrix effect is biasing results. Analysts may attempt to eliminate the interference or else flag the sample results with a sample qualifier code.

Matrix Spike (equivalent to laboratory fortified matrix or LFM)
A matrix spike (MS) is an environmental sample to which known concentrations of target analytes have been added. MS samples are analyzed to evaluate the effect of the sample matrix on the analytical methodology. MS samples are generated by taking a separate aliquot of an actual client sample and spiking it with the selected target analyte(s) prior to sample extraction. The MS sample then undergoes the same extraction and analytical procedures as the unfortified client sample. Due to the potential variability of the matrix of each sample these results may have immediate bearing only on the specific sample spiked and not on all samples in the QC batch.

If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the LCS and MS. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number (at a minimum 10%) of the listed components may be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

Matrix Spike Duplicate (equivalent to laboratory fortified matrix duplicate or LFMD)
A matrix spike duplicate (MSD) is a second aliquot of a sample that is spiked with the selected target analyte(s) and analyzed with the associated sample and MS sample. The results of the MS and MSD are used together to determine the effect of a matrix on the accuracy and precision of the analytical process. Due to the potential variability of the matrix of each sample, the MS/MSD results may have immediate bearing only on the specific sample spiked and not all samples in the QC batch.

Sample Duplicate
A sample duplicate is a second aliquot of an environmental sample taken from the same sample container that is processed identically with the first aliquot of that sample. That is, sample duplicates are processed as independent sample within the same QC batch. The results are compared to determine the sample homogeneity and the precision of the analytical process.

Surrogates
Surrogates are organic compounds that are similar in chemical composition and behavior to the target analytes but that are not normally found in environmental samples. Surrogate compounds must be added to all samples, standards, and blanks for all organic

chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery.

Tuning Solution
Tuning solutions are used to determine acceptable instrument performance prior to calibration and sample analysis for GC/MS analysis.

Post-Digestion Spike
A recommended quality control sample whenever a new or unusual sample matrix is encountered. The spike is added to the sample after digestion. It is a test for matrix interference (positive or negative bias). The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrument detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected.

Interference Check Sample
An interference check sample (ICS) is a solution containing known concentrations of both interfering and analyte elements. Analysis of this sample can be used to verify background and inter-element correction factors.

Internal Standards
An internal standard (IS) is a compound or element with similar chemical characteristics and behavior in the analysis process of the target analytes, but in not normally found in environmental samples. The internal standard is usually added after sample preparation. The primary function of the internal standard is quantitation, however it also provides a short-term indication of instrument performance. For isotope dilution methods, internal standards are added during sample preparation and are used for quantitation.

Quality Control Check Samples
In general, these samples are prepared similarly to an LCS, except that the reagent water is spiked with all compounds of interest. It must be from an independent source from the calibration standards. The standard is generally required in 40 CFR Part 136 methods (e.g., 624) due to the long list of analytes and the risk that the spiked sample may have some analytes outside of control limits. Note the required concentration of the standard as described within the published method or laboratory SOP.

Range.
The difference between the minimum and the maximum of a set of values.

11.2 Methods of Calculations for QC

11.2.1 Precision - Generating control limits for P and A

Precision is estimated from the relative percent difference (RPD) of the concentrations (not the recoveries) measured for matrix spike/matrix spike duplicate pairs, or for duplicate analyses of unspiked samples. For each matrix spike/matrix spike duplicate or sample and sample duplicate analyzed, calculate the relative percent difference, as described in Section 12.1.4.4 (Data Reduction, Verification and Reporting). If calculated form three or more replicates, relative standard deviation (RSD) is calculated as described in Section 9.3.3.3, rather than RPD.

Note: Range is a better measurement of precision than RPD as analytical results approach the MDL (20 x the MDL is a reasonable figure). This is especially important for those

analyses that do not lend themselves to spiking (e.g., BOD and solids). For each sample and sample duplicate, calculate range as follows:

$$\text{Range} = |C_{(1)} - C_{(2)}|$$

Where:
$C_{(1)}$ = Measured concentration of the first sample aliquot
$C_{(2)}$ = Measured concentration of the second sample aliquot

Calculate the average (p) and the standard deviation (s) for each of the duplicated compounds after analysis of 20-30 duplicate samples of the same matrix.

Calculate control and warning limits for each compound (since RPD or range are expressed as a positive number, there can be no lower control limit, as that value would be a negative number), as follows:

$$\text{Control limit} = p + 3s$$
$$\text{Warning limit} = p + 2s$$

Control limits approximate a 99% confidence interval around the mean, while warning limits approximate a 95% confidence interval. Statistically, sixty-eight percent of all results should fall within one standard deviation of the mean. Statistically, seven consecutive results on one side or the other of the mean indicate an anomaly that should be corrected, while three consecutive results exceeding warning limits also indicate an event that should be investigated.

Any matrix spike, surrogate, or laboratory control sample (LCS) result outside of the control limits requires evaluation by the laboratory. Such actions should begin with a comparison of the results from the sample or matrix spike sample with the LCS results. If the recoveries of the analytes in the LCS are outside of the control limits, then the problem may lie with the application of the extraction and/or cleanup procedures applied to the sample matrix or with the chromatographic procedures. Once the problem has been identified and addressed, corrective action may include the re-analysis of sample, or the extraction and analysis of new sample aliquots, including new matrix spike sample and LCS. When the LCS results are within the control limits, the problem may either be related to the specific sample matrix or to an inappropriate choice of extraction, cleanup, and determinative methods. For a further discussion of corrective action, see Section 13.

Control (acceptance) limits and warning limits are printed and updated at least annually. Once limits are updated, the new limits are posted in the laboratory (dated and approved by the QA Officer) and entered into a master log. The QA Officer maintains an archive of all limits used within the laboratory with the start and ending effective dates. The control and warning limits used to evaluate sample results are those that are in place at the time of sample analysis.

For methods and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

Results used to develop acceptance criteria must meet all other QC criteria associated with the determinative method. For instance, matrix spike recoveries from a GC/MS procedure are generated from samples analyzed after a valid GC/MS tune and a valid initial calibration that includes the matrix spike compounds. Another example is that analytes in GC methods must fall within the established retention time windows in order to be used to develop acceptance criteria.

It is advisable to consider the effects of the spiking concentration on matrix spike control limits, and to avoid censoring of data. The acceptance criteria for matrix spike recovery and precision are often a function of the spike concentration used. Therefore, caution must be used when pooling matrix spike/matrix spike duplicate data to generate control limits. Not only should the results all be from a similar matrix, but the spiking levels should also be approximately the same (within a factor of 2). Similarly, the matrix spike and surrogate results should all be generated using the same set of extraction, cleanup and analysis techniques. For example, results from solid samples extracted by ultrasonic extraction are not mixed with those extracted by Soxhlet.

Another common error in developing acceptance criteria is to discard data that do not meet a preconceived notion of acceptable performance. This results in a censored data set, which, when used to develop acceptance criteria will lead to unrealistically narrow criteria. Remember that for a 95% confidence interval, 1 out of every 20 observations likely will still fall outside the limits. While professional judgement is important in evaluating data to be used to develop acceptance criteria, specific results are not discarded simply because they do not meet one's expectations. Rather, a statistical test for outlier values is employed (see Section 11.3).

In-house QC limits must be examined for reasonableness. Poor recoveries should not be legitimized due to the incorrect choice of methods or spiking levels. In-house limits are important when considering the objectives of specific analyses. For example, recovery limits that include allowance for a relatively high positive bias (e.g., 70-170%) may be appropriate for determining that an analyte is not present in a sample. However, they would be less appropriate for the analysis of samples near but below a regulatory limit, because of the potential high bias.

It may be useful to compare QC limits generated in the laboratory to the performance data that may be listed in specific determinative methods. However, be aware that performance data generated from multiple laboratory data tend to be significantly wider than those generated from single laboratory data. In addition, comparisons between in-house limits and those from other sources should generally focus more on the accuracy (recovery) limits of single analyses rather than the precision limits. For example, a mean recovery closer to 100% is generally preferred, even if the $\pm$3 standard deviation range is slightly wider, because those limits indicate that the result is likely closer to the "true value". In contrast, the precision range provides an indication of the results that might be expected from repeated analyses of the same sample.

11.2.2 Standard Deviation and Control Limits

Historical data that the laboratory generates are used to calculate in-house control limits for matrix spike recoveries, surrogate recoveries and laboratory control sample recoveries. The development of in-house control limits and the use of control charts or similar procedures to track laboratory performance are important.

Accuracy is estimated from the recovery of spike analytes from the matrix of interest. For each matrix spike sample, calculate the percent recovery of each matrix spike compound added to the sample, as described in Section 12.1.4.3 (Data Reduction, Verification and Reporting).

For each collected sample, calculate the percent recovery of each surrogate, as follows:

$$\text{Recovery (\%)} = \frac{\text{Conc. (or amt.) found}}{} \times 100$$

Conc. (or amt.) added

Calculate the average percent recovery (p) and the standard deviation (s) for each of the matrix spike compounds after analysis of 20-30 matrix spike sample of the same matrix. Calculate the average percent recovery (p) and the standard deviation (s) for each of the surrogates after analysis of 20-30 collected sample of the same matrix, in a similar fashion.

Calculate upper and lower control limit for each matrix spike or surrogate compound, as follows:

Upper control limit = p + 3s
Lower control limit = p - 3s

Calculate warning limits as:

Upper control limit = p +2s
Lower control limit = p - 2s

In general, the laboratory utilizes method or laboratory defined warning and control limits for reporting data (i.e., statutory control limits). Those statutory limits may be modified utilizing statistical information collected over time. The precision and recovery data are used for the diagnosis of analytical problems. For laboratory parameters, calculated statistical control limits are used as criteria to accept or reject data only if they are more stringent than the criteria in Table 5.1.

The formulae used for the calculation of standard deviation, mean, upper and lower control and warning limits are shown below. (Reference chapter 6 of "*Handbook for Analytical Quality Control in Water and Wastewater Laboratories" - EPA 600/4-79-019, March 1979*).

a. Standard deviations are calculated based on the formula:

$$Sp = \sqrt{[\sum_{i=1}^{n} P_i^2 - (\sum_{i=1}^{n} P_i)^2 / n] / n-1}$$

Where:
Sp = standard deviation of the population
n = total number of points in the population
P_i = the value for each point

b. The mean is calculated as the average of all points:

$$\overline{P} = \frac{\sum_{i=1}^{n} Pi}{n}$$

c. For recovery, the upper and lower control limits are based on a 99% confidence level.

$UCL = P + t_{(0.99)}Sp$
$LCL = P - t_{(0.99)}Sp$

d. The upper and lower warning limits for recovery are based on a 95% confidence level.

$UWL = P + t_{(0.95)}Sp$
$LWL = P - t_{(0.95)}Sp$

Where $t_{(0.99)}$ and $t_{(0.95)}$ are Student's t factors for 99% and 95% confidence, respectively.

Because levels of statistical confidence vary with sample size, a fixed level of statistical confidence is employed that approximates 2 and 3 standard deviations. Those control limits are based on requirements specified in various EPA methods and in EPA's 'Handbook for Analytical Quality Control in Water and Wastewater Laboratories'. The statistical program utilizes a Student's t table, setting warning limits at 95% confidence and control limits at 99% confidence. Those Student's t factors correspond approximately to 2 and 3 standard deviations for 7 collected data points. The advantage of using Student's t factors is that control limits are based on known confidence limits regardless of the number of data points in the population.

e. For precision on duplicate samples, the upper warning and control limits are based on a 95% and 99%confidence level, respectively.

$UWL = D_3P$
$UCL = D_4P$

Where D_3 and D_4 are Shewhart factors representing 95% and 99% confidence limits for pairs of duplicates[1,2] and P is the mean for the population of precision values (as %RPD measurements).

11.3 Statistical Outlier Tests

It is important to exclude extreme measurements from a data set to eliminate bias in statistical evaluations such as control limit calculation. Extreme or atypical values are often referred to as outliers because of their location outside the normal distribution for a particular data set. When data follow a Gaussian distribution, certain statistical assumptions can be made about the data:

- about 68% of the measurements will be within one standard deviation of the mean;
- about 95% of the measurements will be within two standard deviations of the mean; and
- about 99% of all measurements will be within three standard deviations of the mean.

Outliers may be rejected outright only when they are caused by a known or demonstrated physical reason, such as sample spillage, contamination, mechanical failure or improper calibration. Data points, which appear to deviate from the expected sample distribution for no known physical reason, must be verified as outliers using statistical criteria.

11.3.1 Z Score

Z-scores can be calculated for large sample sizes (greater than 30 data points), and thus are useful to determine if a value should be excluded from a calculation of control limits. A Z-score of greater than 4 is an indication that the data point in question is an outlier. The Z-score is calculated as follows:

$$Z = \frac{|X - X_{bar}|}{S}$$

Where:
Z = Z-score

X= the measurement in question
X_{bar} = the mean of the measurements
S = the standard deviation of the measurement

Look up the critical value of Z in Table 11-1 below, where N is the number of values in the data set. If the calculated Z value is greater than the tabulated value, then the P value is <0.05. This means that there is less than a 5% chance that you'd encounter an outlier.

11.3.2 Grubbs' T test

The Grubbs' T test is an objective test for determining whether a point is an outlier in a smaller data set (less than 20 data points). The Grubbs' T value is calculated as follows:

$$T = \frac{|X_q - X_{bar}|}{S}$$

Where:
T = Grubbs' T value
X_q = the measurement in question (the data point furthest from the mean)
X_{bar} = the mean of the measurements
S= the standard deviation of the measurement

The result of the calculation is compared against the value of T from Table 11-1, using the appropriate number of measurements and the acceptable rejection factor (the 5% rejection factor is presented here). If the Grubbs' T value is greater than the value of T from the table, the data point in question is a statistical outlier, and should be rejected from the data set.

Table 11-1: Critical values for Grubb's T

Number of Data Points	Critical Value
7	1.94
8	2.03
9	2.11
10	2.18
12	2.29
14	2.37
15	2.41
16	2.44
18	2.50
20	2.56

The Grubbs' test detects one outlier at a time. This outlier is expunged from the data set and the test is iterated until no outliers are detected. However, multiple iterations change the probabilities of detection, and the test should not be used for sample sets of 6 or less since it frequently tags most of the points as outliers.

11.4 Method Detection Limits (MDL) and Practical Quantitation Limits (PQL)

The MDL defined below is adapted from 40 CFR Part 136, Appendix B, Revision 1.11 et seq. Similarly, the PQL is defined on the basis of this MDL study.

11.4.1 Scope and Application

The MDL is defined as the minimum concentration of an analyte that can be measured by the method with 99% confidence of its presence in the sample matrix. This procedure is designed for applicability to a wide variety of sample types ranging from reagent water spiked with the analyte, to wastewater containing analyte, to sand or other solid matrices containing the analyte. The MDL for an analytical procedure may vary as a function of sample type. The procedure requires a complete, specific, and well-defined analytical method. It is essential that all sample-processing steps of the analytical method be included in the determination of the MDL. The MDL obtained by this procedure is used to judge the significance of a single measurement of a future sample. The MDL procedure was designed for applicability to a broad variety of physical and chemical methods, and should be performed in both aqueous and non-aqueous matrices (where samples are analyzed in both matrix types). MDL's must be determined each time there is a significant change in the test method or instrument type. A MDL study is not required for any component for which spiking solutions or quality control samples are not available, such as BOD_5, $CBOD_5$, TS, TSS, TDS, coliform, chlorophyll *a*, turbidity and color.

11.4.2 Procedure

Make an estimate of the detection limit using one of the following:

- The concentration value that corresponds to an instrument signal/noise in the range of 2.5 to 5.
- The concentration equivalent of three times the standard deviation of replicate instrumental measurements of the analyte in reagent water.
- That region of the standard curve where there is a significant change sensitivity, i.e., a break in the slope of the standard curve.
- Instrumental limitations.
- It is recognized that the experience of the analyst is important to this process. However, the analyst must include the above considerations in the initial estimate of the detection limit.

Prepare a matrix (i.e., reagent water) that is as free of analyte as possible. Reagent or interference free water is defined as a water sample in which analyte and interferant concentrations are not detected at the MDL of each analyte of interest. Interferences are defined as systematic errors in the measured analytical signal of an established procedure caused by the presence of interfering species (interferant). The interferant concentration is presupposed to be normally distributed in a representative sample of a given matrix.

11.4.2.1 Matrix choice

a) If the MDL is to be determined in reagent water, prepare a laboratory standard at a concentration which is at least equal to or in the same concentration range as the estimated detection limit (recommend between 1 and 5 times the estimated detection limit).

b) If the MDL is to be determined in another sample matrix, analyze recommended range of one to five times the estimated detection limit. (Note: Clean sand may also be spiked to determine the MDL for solids).

1) If the measured level of analyte is less than the estimated detection limit, add a known amount of analyte to bring the level of analyte between one and five times the estimated detection limit.

2) If the measured level of analyte is greater than five times the estimated detection limit, there are two options.

i) Obtain another sample with a lower level of analyte in the same matrix if possible.

ii) This sample may be used as is for determining the MDL if the spike level does not exceed 10 times the calculated MDL of the analyte in reagent water. The variance of the analytical method changes as the analyte concentration increases from the MDL; hence the MDL determined under these circumstances may not truly reflect method variance at lower analyte concentrations.

11.4.2.2 Analysis

a) Take a minimum of seven aliquots of the sample to be used to calculate the MDL and process each through the entire analytical method. Make all computations according to the defined method with final results in the method reporting units. If a blank measurement is required to calculate the measured level of analyte, obtain a separate blank measurement for each sample aliquot analyzed. Where allowed by the methods, the average blank measurement is subtracted from the respective sample measurements.

b) It may be economically and technically desirable to evaluate the estimated MDL before proceeding with 11.4.2.2a. This will: (1) Prevent repeating this entire procedure when the costs of analyses are high and (2) insure that the procedure is being conducted at the correct concentration. It is quite possible that an inflated MDL will be calculated from data obtained at many times the real MDL even though the level of analyte is less than five times the calculated MDL. To insure that the estimate for the MDL is a good estimate, it is necessary to determine that a lower concentration of analyte will not result in a significantly lower MDL. The two aliquots of the sample to be used to calculate the MDL and process each through the entire method, including blank measurements as described above in 11.4.2.2a.

1) If these measurements indicate the sample is in desirable range for determination of the MDL, take five additional sample aliquots and proceed. Use all seven measurements for calculation of the MDL.

2) If these measurements indicate the sample is not in correct range, re-estimate the MDL, obtain new sample as in 11.4.2.1 and repeat either 11.4.2.2a or 11.4.2.2b.

Calculate the standard deviation (s) of the replicate measurements.

Compute the MDL, as follows:

$MDL = t_{(n-1, 1-\mu=0.99)} (s)$

Where:

MDL = the method detection limit
$T_{(n-1, \mu-1=0.99)}$ = the Student's t value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom (see Table 11-2).
S = standard deviation of the replicate analyses.

Table 11-2: Students' t-Values at the 99% Confidence Level

Number of replicates	Degrees of freedom (n-1)	$T_{(n-1, 0.99)}$
7	6	3.143
8	7	2.998
9	8	2.896
10	9	2.821
11	10	2.764
12	11	2.718
13	12	2.681
14	13	2.650
15	14	2.624
16	15	2.602
17	16	2.583
18	17	2.567
19	18	2.552
20	19	2.539

The MDL is recalculated/verified on at least an annual basis or anytime any major changes have been made to the analytical system. A processed blank sample is analyzed with each sample set.

The PQL is considered the lowest level of concentration that can be reliably achieved within specified limit of precision and accuracy during routine laboratory operating conditions. This laboratory sets the PQL at 3 to 5 times the MDL depending on the method of analysis and the analyte, unless otherwise specified.

11.5 MDL Reporting

The analytical method used must be specifically identified by number and method title. The date of the study, instrument ID and the name of the analyst(s) performing the analysis must be included. If the analytical method permits options that affect the MDL, these conditions must be specified with the MDL value (i.e., sample preparation methods, columns, and detectors). The sample matrix, date of calibration and the standard (ID# and concentration) used must be documented. The MDL for each analyte must be expressed in the appropriate method reporting units. Report the mean analyte level with the MDL. If a laboratory standard or a sample that contained a known amount analyte was used for this determination, also report the mean recovery. If the level of analyte in the sample was below the determined MDL or exceeded 10 times the

MDL of the analyte in reagent water, do not report a value of the MDL. An example format for documenting each MDL can be found in Figure 11-1.

11.6 Blind QC Check Sample Analysis

The laboratory is a participant in EPA's Performance Evaluation Study. Results of these tests will be summarized and included in the laboratory's QA report.

Figure 11-1: Example MDL Reporting Format

Method Detection Limit (MDL) Study

Laboratory Name:		**Analytical Method:**	
Analyst(s) Name(s):		**SOP#:**	
Date:		**Instrument:**	
Sample Prep. Method:		**Column:**	
SOP#:		**Detector:**	
Matrix:		**Cleanup/Modification:**	

Analyte	Spike conc.	Units	1	2	3	4	5	6	7	Mean Recovery %	Average Recovery X	Standard Deviation s	MDL	PQL

MDL = t (n-1, 1-a = 0.99) (s)
t = Student's t values appropriate for 99% confidence level. Table of Students' t values can be found in 40 CFR Part 136, Appendix C.
PQL = 3 to 5 times the calculated MDL.

Comments: _____

_____ _____ _____
Chemist's/Technician's Name Signature Date

_____ _____ _____
Branch Manager's Name Signature Date

_____ _____ _____
Lead Chemist's Name Signature Date

_____ _____ _____
Quality Assurance Officer's Name Signature Date

12.0 Data Reduction, Verification and Reporting

In order to provide complete, accurate and verifiable results, all analytical data generated by the DWQ Laboratory Section is recorded, reviewed, reported and archived according to Laboratory policy. Analytical areas have slightly different data reduction, validation and reporting protocols depending on the means by which the data are generated and specific method requirements. The general procedures involved in the process of data reduction, validation and reporting are explained in this section. Detailed procedures are outlined in laboratory operational or analytical SOPs.

12.1 Data Reduction

Data reduction includes all activities that convert analytical values into reportable sample concentrations of the target analyte(s). These activities may involve mathematical calculations, compound identification and summary statistics. The final results may be obtained in two ways:

1. Direct readings from the instrument; or
2. Calculations based on instrument output, readings or responses.

The Laboratory Section's goal is to minimize the steps needed to transform raw data into reportable results and maximize on the number of analytical results generated by automated systems. The more automated the data reduction process, the less likely data transcription and calculations errors are to occur.

12.1.1 Manual data reduction

Manual data reduction refers to those activities in which analytical output is converted to sample concentrations by calculations performed manually or by validated computer applications.

During the manual data reduction process, analysts will:

- Assure that all data are correctly transcribed into worksheets, forms or computer application;
- If the analytical instrument used generates hardcopy reports (e.g., strip charts, tabulated reports, etc.), the analyst will keep such raw data as part of the analysis records;
- Select the appropriate, method-specified formulae for calculating results. The formulae used are written in the standard operating procedures;
- Proofread computer-generated reports to ensure that the raw data manually entered into the computer application was entered correctly.
- Record appropriate and accurate information concerning sample identification, operating conditions, etc.

The Laboratory Section retains documentation of all computer applications used for this purpose, including the mathematical formulae used to calculate sample concentrations. If such information is not available, or can not be obtained from the code, the application is validated by comparing the results of the application with the results of manual calculations. A record of this verification is maintained in the analytical unit.

All raw data output (i.e., strip charts, tabular printouts, etc.) must be identified with the following information (where applicable):

- Date of analysis
- Sample ID numbers
- Analyst or operator
- Type of analysis
- Instrument operating conditions
- Detector
- Column

- Instrument configuration

12.1.2 Computer data reduction

Computer data reduction refers to those activities in which analytical acquisition and initial calculations are performed automatically by validated computer applications.

When computer data reduction is performed, the analysts will (as is appropriate to the method used):

- Ensure that all variables required for final calculations (sample amount, dilution factor, extract volume, percent solids, surrogate amount, etc.) are entered accurately;
- Properly interpret the computer output in terms of properly identified components, positive or negative identifications and appropriate confirmatory measures;
- Record appropriate and accurate information concerning sample identification, operating conditions, etc.;
- Calculate surrogate recoveries and verify that internal standard responses are acceptable;
- Verify that target compounds analyzed by chromatographic methods are within the appropriate retention time or relative retention time windows and that additional confirmation is initiated as needed.

Raw data files are assigned a unique filename by the analyst performing the analyses. In some instances, the computer performs the filename assignment using rules that ensure that filenames will not be repeated (i.e., a queue number). In such cases, a cross-reference index or log is maintained to identify the computer data files with sample ID numbers. Additional information that should be entered into the data file records are date of analysis, analysis type and analyst initials. Cross-referenced auxiliary records may be required to identify instrument operating conditions. Many analytical instruments are interfaced with computers or integrators that automatically evaluate, identify and calculate final values. The results are printed in combinations of graphic (e.g., chromatograms) and tabular forms. As with manual data reduction, the Section must be aware and should have on file a record of the mathematical formulae or algorithms that are being used by the computer. If the information is not available, the organization shall maintain records, which demonstrate that the software is providing the expected results.

Where there are cases in which the results from spiked samples suggest interferences, attempts are made to remove the interferences, or alternate analytical procedures are used. If the interference problem cannot be resolved, the data is flagged and an explanation included on a SAR form.

12.1.3 General data reduction responsibilities

Additional data reduction responsibilities include:

- Ensuring that samples are analyzed only when the instrument is calibrated according to the method;
- Ensuring that QC results are calculated correctly, within criteria, and if not, initiating corrective actions;
- Identifying QC results for review by the responsible person(s);
- Documenting sample preparation and analysis, and the conditions under which they were performed, in appropriate logbooks or on appropriate benchsheets;
- Ensuring that the laboratory sample ID is directly traceable to the field sample and is correctly transcribed into all associated analytical records;
- For computer-controlled data acquisition and data reduction, the analysts are responsible for entering all the parameters needed for final result calculation correctly;
- For manual data reduction, the analysts are responsible for performing the calculations according to the method requirements;

- If the result is transcribed, the analysts are responsible for ensuring that the entry is entered correctly;
- The analysts are responsible for alerting a Supervisor about any problems that the analyst believes may affect the quality of the data.

Every instrument and/or method within the six analytical areas and two regional laboratories has a slightly different data reduction process depending on the way in which data are generated and the required data transformations. Most sample concentration results are read directly from instrumentation without further reduction or calculations. Dilution factors are applied upon the dilution of samples having concentrations above the calibration range. In many cases, these are input into the instrument computer and correct results are calculated automatically. In other cases, a manual calculation may be performed (this may be done by hand or by entering the raw data result into an Excel spreadsheet programmed to perform the additional data manipulations). The Laboratory Section calculates results according to the guidance provided in the methods cited in Section 5. Exceptions would be clearly noted in the raw data and on final reports.

Soil/sediment concentration results for all laboratory sections are calculated on a dry weight basis, prior to reporting, by dividing the instrument result by the fractional dry weight. Section 12.1.4 lists equations used in computer-controlled instrumentation for data reduction as well as equations used for the manual calculation of reportable concentration results.

The laboratory raw data containing the instrument-generated reports, manually calculated results, and all supporting preparation, calibration, and analytical data are retained at the individual work stations until reports are issued unless additional handling or data packaging is required. Laboratory SOPs include equations used to calculate results, the method of calculation, benchsheets used to record pertinent data for each analytical method and a description of the data reduction process. All data processed either manually or electronically is verified by a second analyst.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, significant figures, etc. If components of interest are detected in any quality control blank (e.g. method blanks, digestion blanks, etc.), the blank concentration must be reported. The blank concentration shall not be subtracted from any associated sample data. Blank correction will be applied only when required by the method/per manufacturer's indication; otherwise, it should not be performed.

It is the Laboratory Section's policy to report automated peak integration results; however, manual integration is allowed if peaks are not properly integrated by the software. Improper integration includes:

- Integration of the wrong peak,
- Not finding the peak at all,
- Improper division of coeluded peaks, and
- Improper drawing of baseline under a peak.

Manual integration is performed along the baseline or above noise level. Calculations are independently verified by appropriate laboratory staff. Manual integrations must never be used solely to meet QC criteria or as a substitute for corrective actions on the analytical system. Corrective action with regard to the instrumentation or computer software must be taken if manual integrations become common for an analysis or instrument that normally uses automated peak integration. Manual integration must be clearly identified and documented on the data report by flagging the affected analytes. The analyst must initial and date the corrected data.

When data are reported from dual columns (as in gas chromatography), the default procedure is to report the highest result between the primary and confirmation columns if the relative percent

difference (%RPD) is <40%. If the %RPD exceeds 40%, the analyst evaluates the data for the presence of matrix interferences and reports the result that is most appropriate for that sample and flags the results to note the discrepancy.

12.1.4 Formulae and Calculations

The final results of each test shall be calculated by the formula specified in the analytical method that is being used. If the formulas outlined in this section are not used, the correct formula can be found in the appropriate method SOP.

12.1.4.1 The analyte concentration in a sample analyzed using external standard calibration can be determined by:

$$\text{Concentration (ppb)} = \frac{(A_s)(V_t)(D)}{(\text{avgCF})(V_i)(S)}$$

where:
A_s is the area of the peak for the analyte in the sample
V_t is the total volume of the extract in µl (for purge and trap analysis V_t=1)
D is the dilution factor (if no dilution is performed, D=1)
avgCF is the mean calibration factor from the initial calibration in area/ng
V_i is the volume of the extract injected in µl (for purge and trap analysis V_i = 1)
S is the sample volume or mass (in mL or g) extracted or purged

12.1.4.2 The analyte concentration in a sample analyzed using internal standard calibration can be determined by:

$$\text{Concentration (ppb)} = \frac{(A_s)(C_{is})(V_t)(D)}{(A_{is})(\text{avgRF})(S)}$$

where:
A_s is the area of the peak for the analyte in the sample
C_{is} is the concentration of the internal standard
V_t is the total volume of the extract in mL (for purge and trap analysis V_t=1)
D is the dilution factor (if not dilution is performed D= 1)
A_{is} is the area of the internal standard
avgRF is the mean response factor form the initial calibration
S is the sample volume or mass (in L or kg) extracted or purged

12.1.4.3 Calculated values for spiked samples, duplicate analyses, and reference standards are compared with quality control limits to determine data validity. Recovery of any spiked analyte (including surrogate compounds) is calculated as:

$$\%\text{Recovery} = \frac{C_s - C_u}{C_n} \times 100$$

where:
C_s is the measured concentration of the analyte or surrogate
C_u is the concentration of the unspiked sample (for LCS and surrogate recoveries C_u = 0)
C_n is the true value or known concentration of the analyte or surrogate.

12.1.4.4 The precision of duplicate analyses is determined from the relative percent difference (RPD) calculated by:

$$RPD(\%) = 2\frac{|R_1 - R_2|}{R_1 + R_2} \times 100$$

where:
R_1 is the measured concentration of one replicate
R_2 is the measured concentration of the second replicate

12.1.4.5 Relative Standard Deviation (RSD) is computed from the standard deviation and mean recovery when the standard deviation is derived from multiple recovery results:

$$RSD(\%) = \frac{\text{Standard Deviation}}{\text{Mean Recovery}} \times 100$$

12.1.4.6 Sample and QC result calculations are reduced as follows:

A. Results from analyzed sample extracts or digestates are processed manually, by the analytical instruments' PC-based data systems or by laboratory chromatography software, based on the method protocols discussed in Sections 5 and 9. These raw sample results are manually calculated or manually/electronically downloaded from the analytical instrument to the appropriate computer application.

B. Sample results and QC results are linked together by date of analysis and assigned lab numbers, so sample prep and analysis batches are always identified with their associated QC. Using pertinent sample prep/analysis data (e.g., amount of sample digested or extracted, final digestate or extract volume, dilution factors, spiking level/solution used, etc.), calculations are either performed manually or by an appropriate computer application. Examples of typical water and sediment calculations performed follow:

Concentration in ug/L (for water samples) =

$$\frac{\text{Final extract or digestate conc. (ug/mL)} \times \text{Final extract or digestate volume (mL)}}{\text{Initial Sample Volume Extracted or Digested (L)}}$$

Concentration in ug/kg (for sediment samples) =

$$\frac{\text{Final extract or digestate conc. (ug/mL)} \times \text{Final extract or digestate volume (mL)}}{\text{Initial Sample Weight Extracted or Digested (kg)} \times \text{Dry Weight Correction Factor}}$$

C. The resulting sample and associated QC results are reviewed by the chemist, then if deemed acceptable, are uploaded to the DWQ STAR LIMS database or for organic analyses, typed onto a STAR report page. Current acceptance criteria (warning and control limits) for each QC element are stored within an Excel spreadsheet or posted in the analytical unit. If QC results are outside of the current control limits, data is flagged with appropriate qualification, comments or codes. The analysis data is fully reviewed to determine if sample contamination or matrix problems exist. The associated sample batch may then be re-submitted for re-digestion/re-extraction and/or re-analysis. If there is still a problem with the quality of the data, in-depth investigation into the method in question is conducted until the problem is resolved. If the problem cannot be resolved immediately, the data may be rejected or reported with qualification.

12.1.5 Corrections

Entries in records shall not be obliterated by methods such as erasures, liquid paper, overwritten files or markings. All corrections to record-keeping errors shall be made by one line marked through the error. The individual making the correction shall sign (or initial) and date the correction. These criteria shall also apply to electronically maintained records.

12.1.6 Significant Figures

Every measurement has a degree of uncertainty associated with it. The uncertainty derives from the limitations of the measuring device and from the skill with which it is used. The accuracy of a measurement is expressed by the number of significant digits (or significant figures) written when the measurement is reported. All digits in a reported result are expected to be known definitely, except for the last digit, which may be in doubt (i.e., has an uncertainty of ± 1 unit). Such a number is said to contain only significant figures.

12.1.6.1 Significant Figure Rules

There are several rules for determining the number of significant digits (or significant figures) in a measurement. In general significant figures are determined starting with the leftmost digit.

1. Non-zero digits are always significant.
2. All zeros between other significant digits are significant.
3. The number of significant figures is determined starting with the leftmost non-zero digit. The leftmost non-zero digit is sometimes called the most significant digit or the most significant figure. For example, in the number 0.004205 the '4' is the most significant figure. The left-hand '0's are not significant. The zero between the '2' and the '5' is significant.
4. The rightmost digit of a decimal number is the least significant digit or least significant figure. Another way to look at the least significant figure is to consider it to be the rightmost digit when the number is written in scientific notation. Least significant figures are still significant. In the number 0.004205 (which may be written as 4.205×10^{-3}), the '5' is the least significant figure. In the number 43.120 (which may be written as 4.3210×10^{1}), the '0' is the least significant figure.
5. If no decimal point is present, the rightmost non-zero digit is the least significant figure. In the number 5800, the least significant figure is '8'.

12.1.6.2 Uncertainty in Calculations

Measured quantities are often used in calculations. The precision of the calculation is limited by the precision of the measurements on which it is based.

Addition and Subtraction
When measured quantities are used in addition or subtraction, the uncertainty is determined by the absolute uncertainty in the least precise measurement (not by the number of significant figures). Sometimes this is considered to be the number of digits after the decimal point.

Example:
32.01 grams
5.325 grams
12 grams

Added together, you will get 49.335 grams, but the sum should be reported as '49 grams'.

Multiplication and Division
When experimental quantities are multiplied or divided, the number of significant figures in the result is the same as that in the quantity with the smallest number of significant figures. If, for example, a density calculation is made in which 25.624 grams is divided by 25 mL, the density should be reported as 1.0 g/mL, not as 1.0000 g/mL or 1.000 g/mL.

When doing several calculations, carry out all of the calculations to at least one more significant figure than you need. Round off the final result.

12.1.6.3 Losing Significant Figures

Sometimes significant figures are 'lost' while performing calculations. For example, if the mass of a filter is found to be 53.110 g, add residue to the filter and find the mass of the filter plus residue to be 53.987 g, the mass of the residue is 53.987-53.110 g = 0.877 g The final value only has three significant figures, even though each mass measurement contained 5 significant figures.

12.1.6.4 Exact Numbers

Sometimes numbers used in a calculation are exact rather than approximate. This is true when using defined quantities, including many conversion factors, and when using pure numbers. Pure or defined numbers do not affect the accuracy of a calculation. These may be thought of as having an infinite number of significant figures. Pure numbers are easy to spot, because they have no units. Defined values or conversion factors, like measured values, may have units.

Example:
To calculate the average of three measured titration volumes: 30.1 ml, 25.2 ml, 31.3 ml; calculate as follows: (30.1 + 25.2 + 31.3)/3 = 86.6/3 = 28.87 = 28.9 ml. There are three significant figures in the volumes; even though you are dividing the sum by a single digit, the three significant figures should be retained in the calculation.

12.1.7 Rounding

Whenever data is reduced using computer applications, the rounding rules used are those provided with the operating software. The final result should be rounded off to an appropriate number of significant figures (typically 2 significant figures). When manual calculations are performed, the following rounding rules are followed:

- If the digit to be dropped is less than 5, do not change the last digit to be retained (e.g., 2.23 rounds off to 2.2).
- If the digit to be dropped is greater than 5, increase the last digit to be retained by one (e.g., 2.26 rounds to 2.3).
- If the digit to be dropped is equal to 5, increase the last digit to be retained by one if it is odd (e.g., 2.35 rounds to 2.4, or do not change the last digit to be retained if it is even (e.g., 2.45 rounds to 2.4).

As a general rule, the results should be converted to the reporting units presented in Section 12.1.8. Other reporting conventions (i.e., wet weight instead of dry weight) should be clearly identified on the final reports with appropriate justification.

12.1.8 Reporting Units

The reporting units listed below are used for results unless otherwise requested by the client. Solid matrices are reported as dry weight unless otherwise requested.

Parameter	Water	Soil
Metals (except as noted below) Ca, Mg, Na, K	µg/L mg/L	mg/Kg
Purgeable Organic Compounds (except as noted below) TPH - GRO	µg/L mg/L	µg/Kg mg/Kg
Extractable Organic Compounds (except as noted below) TPH - DRO	µg/L mg/L	µg/Kg mg/Kg
Inorganic/Microbiology Parameters (except as noted below) Specific Conductance Turbidity Coliform, MF Coliform, MPN Color, PtCo Color, ADMI Boron Total Phenol Hexavalent Chromium Chlorophyll *a*	mg/L µmhos/cm @ 25°C NTU Colony/100 ml MPN/100 ml Color units (c.u.) Color units (c.u.) µg/L µg/L µg/L µg/L	mg/Kg

12.2 Data Verification

Data verification or review is the routine laboratory process through which proper quantification, recording, transcription, and calculations are confirmed. It also confirms that the data is reasonable and complete. The process should be such that errors are minimized and that corrective action steps are taken and documented when errors are detected. The objective of data verification is to provide results of verifiable and acceptable quality whose validity is not jeopardized. The data verification process ensures that:

- The correct samples are reported;
- There were not systematic errors in calculating final results;
- Samples were analyzed within calibration and the required holding times;
- The QC elements monitored were within known acceptance limits.

Each analyst and/or technician is responsible for determining that the results of each analytical determination have all associated QC measurements (completeness) and that the acceptance criteria are met and documented according to protocol (correctness). The analyst and/or technician is responsible for checking calculations, completing sample preparation, calibration, analysis, standard and instrument logs. Each analyst, peer reviewer or supervisor is responsible for reviewing this work for completion and correctness prior to authorizing the individual results for release. This includes checking for appropriate flagging of final results. Any discrepancy or inconsistency will initiate a recheck of data or reanalysis of the sample(s).

The data verification process includes four steps: initial, secondary, and final review and release authorization.

12.2.1 Initial Review

Raw data is converted to reportable data and transcribed from benchsheets or instrument printouts onto standardized laboratory parameter spreadsheets by the analyst performing the test. The analyst performs the initial review of the data and data result entry. The analyst is responsible for verifying the correctness of the data entered into the Laboratory DWQ STAR LIMS system. This initial review includes, but is not limited to, verifying that quality control indicators meet criteria, calibration criteria are met, appropriate detection limits were used, data was reduced correctly and that any corrective action was documented properly. The primary reviewer is responsible for verifying any documentation associated with the data, completing all records associated with the process, and completing sample anomaly reports as required. The analyst is responsible for assembling a data package containing all relevant raw data needed for data interpretation. This may include: benchsheets, instrument printouts such as quantitation reports, integrator peak area/height and retention time reports, chromatograms, and diagnostic reports. The analyst must perform primary review on 100% of the data generated.

12.2.2 Secondary Review

A party other than the analyst generating the data (e.g., a peer within the same analytical area) is responsible for a secondary review of the data. This step is intended as a verification of the primary review. Secondary review focuses on laboratory data entries and calculations for errors and mistakes, calibration criteria, quality control indicators, compound identification, results expression, reporting limits, holding times, sample and standard preparation logs, data transcription and documentation. All data are verified. If problems exist during this review, the data is returned to the primary analyst and a 100% review is done and corrective action is performed as appropriate. Once the data is checked and deemed acceptable for reporting, the secondary reviewer dates and initials the quality control section on bench worksheets or on the cover page of computer-generated reports and submits the data to the supervisor for final review.

Specific checks required of the secondary reviewer are summarized in Figures 12.1 and 12.2.

12.2.3 Final Review

Final review must be performed prior to committing the data results to the DWQ STAR LIMS database by an individual familiar with it, but not involved in the original data reduction process (e.g., supervisor and/or branch manager). The process includes, but is not limited to, verifying that chemical relationships are evaluated, sample ID numbers are correct, tests have been performed within the appropriate holding times, all precision and accuracy requirements are addressed, data transcription and data entry were performed correctly, narratives are present, flags are appropriate, SARs are attached and project specific requirements are met.

Data found to be of doubtful quality by the analyst, through internal audits or arising from customer concerns, must be reviewed by a member of laboratory management or the QA/QC Coordinator using the procedures outlined in Section 13.

After verification of the data is complete, a macro is initiated by a Processing Assistant to check for completeness. When all results for a sample have been entered into the database, the results are printed from the DWQ STAR LIMS system into a final report. The hard copy report is then checked for data entry errors by a second Processing Assistant. The report is then sent to the branch managers or supervisors for release authorization.

12.2.4 Release Authorization

This review ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that chemical

relationships are evaluated, sample ID numbers are correct, tests have been performed within the appropriate holding times, the results are relevant to historical values, project-specific requirements have been met, and the chain of custody was maintained. This action authorizes transmittal of the final report to the client.

Figure 12.3 is a flow chart of the analytical data review and reporting process.

12.3 Reporting

Each supervisor is responsible for authorizing the individual analysis results for release. After all the sample results are authorized, the Processing Assistant uses the DWQ STAR LIMS to generate final reports in electronic and hard copy format with the appended organics report (when applicable), and any associated anomaly reports which detail the reason data was qualified. The completed report package is sent to the Branch Managers for release authorization.

The branch managers or supervisors certify the hard copy reports by reviewing, dating and initialing. One report is retained with the original fieldsheets in the laboratory. The other report is mailed with copies of the fieldsheets to the client. All final sample results are archived in the DWQ STAR LIMS database and can be retrieved in the future if necessary.

12.3.1 Data Qualifier Codes

Data qualifier codes are used on reports as needed to inform the client of any additional information that might aid in the interpretation of the data. The data flagging system incorporates data qualifiers which are similar to flags specified in the Contract Laboratory Program protocols, and STORET, as well as additional flags used to help explain batch specific events. The results may be qualified as follows:

A Value reported is the mean (average) of two or more determinations. This code is to be used if the results of two or more discrete and separate samples are averaged. These samples shall have been processed and analyzed independently (e.g. field duplicates, different dilutions of the same sample).

B Results based upon colony counts outside the acceptable range and should be used with caution. This code applies to microbiological tests and specifically to membrane filter (MF) colony counts. It is to be used if less than 100% sample was analyzed and the colony count is generated from a plate in which the number of coliform colonies exceeds the ideal ranges indicated by the method. These ideal ranges are defined in the method as:

Fecal coliform bacteria: 20-60 colonies
Total coliform bacteria: 20-80 colonies

1. Countable membranes with less than 20 colonies. Reported value is estimated or is a total of the counts on all filters reported per 100 ml.

2. Counts from all filters were zero. The value reported is based on the number of colonies per 100 ml that would have been reported if there had been one colony on the filter representing the largest filtration volume (reported as a less than "<" value).

3. Countable membranes with more than 60 or 80 colonies. The value reported is calculated using the count from the smallest volume filtered and reported as a greater than ">" value.

4. Filters have counts of both >60 or 80 and <20. Reported value is a total of the counts from all countable filters reported per 100 ml.

5. Too many colonies were present; too numerous to count (TNTC), the numeric value represents the maximum number of counts typically accepted on a filter membrane (60 for fecal and 80 for total), multiplied by 100 and then divided by the smallest filtration volume analyzed. This number is reported as a greater than value.

6. Estimated Value. Blank contamination evident.

Note: A "B" value shall be accompanied by justification for its use denoted by the numbers listed above (ex. B1, B2, etc.)

C Total residual chlorine was present in sample upon receipt in the laboratory; value not accurate (cyanide, phenol, NH_3, TKN, coliform, organics)

G A single quality control failure occurred during biochemical oxygen demand (BOD) analysis. The sample results should be used with caution.

1. The dissolved oxygen (DO) depletion of the dilution water blank exceeded 0.2 mg/L.

2. The bacterial seed controls did not meet the requirement of a DO depletion of at least 2.0 mg/L and/or a DO residual of at least 1.0 mg/L.

3. No sample dilution met the requirement of a DO depletion of at least 2.0 mg/L and/or a DO residual of at least 1.0 mg/L.

4. Evidence of toxicity was present. This is generally characterized by a significant increase in the BOD value as the sample concentration decreases.

5. The glucose/glutamic acid standard exceeded the range of 198 ± 30.5 mg/L.

6. The calculated seed correction exceeded the range of 0.6 to 1.0 mg/L.

7. Less than 1 mg/L DO remained for all dilutions set. The reported value is an estimated greater than value and is calculated for the dilution using the least amount of sample.

8. Oxygen usage is less than 2 mg/L for all dilutions set. The reported value is an estimated less than value and is calculated for the dilution using the most amount of sample.

9. The DO depletion of the dilution water blank produced a negative value.

Note: A "G" value shall be accompanied by justification for its use denoted by the numbers listed above (ex. G1, G2, etc.)

J Estimated value; value may not be accurate. This code is to be used in the following instances:

1. Surrogate recovery limits have been exceeded;

2. The reported value failed to meet the established quality control criteria for either precision or accuracy;

3. The sample matrix interfered with the ability to make any accurate determination; or

4. The data is questionable because of improper laboratory or field protocols (e.g. composite sample was collected instead of grab, plastic instead of glass container, etc.).

5. Temperature limits exceeded (samples frozen or >6° C) during transport, non-reportable for NPDES compliance monitoring.

6. The laboratory analysis was from an unpreserved or improperly chemically preserved sample. The data may not be accurate.

M Sample and duplicate results are "out of control". The sample is non-homogenous (e.g., VOA soil). The reported value is the lower value of duplicate analysis of a sample.

N Presumptive evidence of presence of material; **estimated** value. This code is to be used if:

1. The component has been tentatively identified based on mass spectral library search;
2. There is an indication that the analyte is present, but quality control requirements for confirmation were not met (i.e., presence of analyte was not confirmed by alternate procedures).
3. This code shall be used if the level is too low to permit accurate quantification, but the estimated concentration is less than the laboratory practical quantitation limit and greater than the laboratory method detection limit. *This code is not routinely used for most analyses.*

Q Holding time exceeded. These codes shall be used if the value is derived from a sample that was received, prepared and/or analyzed after the approved holding time restrictions for sample preparation and analysis.

1. Holding time exceeded prior to receipt by lab.
2. Holding time exceeded following receipt by lab.

S Not enough sample provided to prepare and/or analyze a method-required matrix spike (MS) and/or duplicate (MSD).

U Indicates that the analyte was analyzed for but not detected above the reported practical quantitation limit*. The number value reported with the "U" qualifier is equal to the laboratory's practical quantitation limit*.

X Sample not analyzed for this constituent

1.Sample not screened for this compound.
2.Sampled, but analysis lost or not performed-field error
3.Sampled, but analysis lost or not performed-lab error

Note: an "X" value shall be accompanied by justification for its use by the numbers listed.

V Indicates the analyte was detected in both the sample and the associated method blank. Note: The value in the blank shall not be subtracted from the associated samples.

Z The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

P Elevated PQL* due to matrix interference and/or sample dilution.

Y Elevated PQL* due to insufficient sample size.

The decision to qualify a result on these factors is at the discretion of the authorizing supervisor and must comply with Laboratory Section Standard Operating Procedures.

12.3.2 Report Format and Contents

Data is transmitted to laboratory data users in two ways: paper reports for each sample (i.e., lab number) and by electronic read-only access. Final reports for test data are issued after all internal review has been completed. Electronic transfer of data is an option available to laboratory data users that have access to the laboratory network.

Analytical results are issued in a format that mimics the sample submission fieldsheets in the case of inorganics. Since organic parameters are multi-analyte, a separate report is attached to this report. The final reports are printed, reviewed and signed by a Branch Manager or their designee. Persons designated to sign reports include the Section Chief, Branch Managers, Unit Supervisors, and the QA/QC Officer.

An example report can be found in Figure 12.2. At a minimum, the following information must be included on all reports:

- Name of laboratory;
- Unique identification of the report (sample ID#) and of each page and the total number of pages;
- Name of the person or entity to report the results to;
- Date received;
- Date reported;
- Sample priority;
- Sample results with units of measurement;
- Relevant SCUR/SAR forms;
- Authorization signature/initials and date.

12.3.3 Corrected Reports

Occasionally a report must be re-issued due to the addition of a test, or the correction of an error. When the report is re-issued, a notation of "REVISED REPORT" is to be placed on the page of the report along with a brief explanation of the correction and authorization initials and date. If it is not practical to include this information directly on the corrected page, a "text" flag can be placed in the result column of the report and a case narrative containing the explanation can be included with the report.

Additionally, a SAR report is required whenever data is changed after authorization. This allows assessment of why the data review process failed to detect an error prior to authorization and release of data and assures that corrective actions are implemented, when possible to prevent future occurrence.

12.4 Data Storage

All data is maintained in such a manner that the records are easily retrievable by authorized personnel. These records may be in electronic or hard copy form. Records may be retained either as original records or as true copies such as photocopies, microfilm, microfiche, or other accurate reproductions of the original records.

12.4.1 Hard copy records storage

After the samples are completed, the hard copy raw and supporting data are stored and filed numerically, alphabetically, or chronologically by date or batch as appropriate for the type of record. The data are maintained in a secured area in the analytical unit in which the data were generated for approximately 1 year. Hard copies of the final reports with associated fieldsheets, COC forms and anomaly reports are filed chronologically in the front office of the Central Laboratory where they are maintained for approximately 3 years. Hard copy records are then transferred to storage boxes that are labeled with the month(s) and year(s) in which the records were generated and a brief content description. Each box is given a unique number and assigned an archive code. This code is entered into an archive log that includes a full description of the contents of each box. The archived boxes are stored on-site for approximately 5 years. Data storage areas are protected against fire, theft, environmental deterioration and vermin. Data storage areas are regularly inspected as part of the Internal Audit program.

Sample data relating to known litigation samples and subsamples will be stored in a locked file cabinet or other secure area maintained by the Section Chief. An archive access log is maintained to document entry into this cabinet.

After the in-house storage period is up, records are processed and transferred to the State Records Center (SRC), 215 North Blount Street, Raleigh, NC. In accordance with the North Carolina Administrative Code, entry and access to the SRC building is limited to persons on official business. Access to stored records is restricted to the creating agency's staff. Persons other than an agency's staff must contact the appropriate agency and receive written permission prior to using records in the SRC. Procedures and forms required by the Center are identified in the State Records Center Handbook.

Currently, paper records are stored in their original form for a total of 10 years. After 10 years, the records are destroyed. Alternatively, records may be processed for microfilming.

These records include:

- Correspondence between laboratory and client;
- All fieldsheets and documentation on the sampling event;
- All field and laboratory analytical records including supporting calibration, raw data, data reduction calculations, quality control information and all data output records (chromatograms, strip charts and other instrument response readout records); Original raw analytical data. This also includes, but it not limited to logbooks, QC samples and analytical samples, MDLs, control limits, standard preparation, method reference and data review records.
- All field and laboratory custody records including shipping receipts, sample transmittal forms, and sample disposal records;
- All notebooks, data forms, and logs pertaining to laboratory operations including sample receipt and log in;
- All records and reports of maintenance, calibration and inspection of equipment and instrumentation;
- All records concerning receipt, preparation and use of calibration standards;
- All statistical calculations used in data reduction and in determination of quality control limits;
- Quality Assurance records including, but not limited to, archived responses, PT sample results and raw data, internal and external audit findings and employee training records.
- Copies of final reports.

Retrieval of archived records (electronic or on paper) is done by referring to the archived records which contain the requester's name, agency, phone number, address, the archive code numbers, date, and the contents. Access to archived information is documented and requester's must complete a Records Retrieval form.

Earlier revisions of SOPs and the Quality Assurance Manual are also archived. The document's "date" indicates the time the policy or procedure was first adopted. Subsequent revision dates indicate when the next revision was adopted.

12.4.2 Electronic records storage

All in-lab data generated by computer systems are printed and archived as hard copies. When the capability exists, data is stored to tape, CD or on hard disc. The tapes, CDs or discs are labeled and stored at the individual workstations and serve as backup copies of the lab's raw data files. Currently, only GC/MS data for organics is backed up and electronically stored on a regular basis (weekly) to CDs. Chromatograms and data files are given a unique alphanumeric identification by the chemists initiating the analyses in each unit where appropriate. These file identification numbers reflect either the date the sequence was initiated, the order in which the samples were analyzed and/or the sample identification and log numbers given by the client and listed on the DWQ STAR LIMS.

The records must be protected from environmental degradation; stored under secure conditions to discourage tampering or vandalism; and must be cross-indexed by laboratory ID number or some other common identifier for easy retrieval.

The DWQ STAR LIMS data resides on the main Laboratory Section server. The server is programmed to backup daily. These daily tapes are overwritten every two weeks. A full back up of this server occurs monthly. The monthly tapes are managed by the LAN specialist and are stored in the room in which the server is located in a fireproof cabinet.

Records, which are stored only on electronic media, must be maintained and supported in the laboratory by all hardware and software necessary for immediate data retrieval and review.

12.4.3 Analytical notebooks/logbooks

Laboratory notebooks used to document pertinent information are stored within each analytical unit. Information contained in notebooks may include sample processing steps such as extractions and digestion records, instrument maintenance and routine checks, and standard and reagent preparations. Notebooks are not destroyed. A master log is kept of all notebooks (e.g., standard logbooks and instrument logbooks) that are issued. At a minimum, this log includes:

- Notebook Number - Each notebook is issued a unique number that is determined sequentially.
- Used for - Purpose and department of notebook.
- Replaces notebook number - Place the number of the notebook that the issued notebook will replace if applicable.
- Date of issue - This is the date that the notebook is released.
- Issued to - The analytical unit the notebook is released to.

Guidelines for Logbook use are as follows:

- Use permanent dark ink. No pencil entries are to be made.
- Corrections - use a single line to cross out documentation error. Date and initial the correction.
- Blank pages or space between the last entry and the bottom of the page must be "Z'd" through, initialed and dated.
- Data must be entered directly and consecutively into the notebook. It is not to be placed onto scratch paper and entered later.
- Entries added to previously signed pages must be dated, initialed and witnessed (if appropriate) below the new material.
- Sign and date each page upon completion.
- When pages are added to the notebook, they must be signed and dated across both the added page and the notebook page.

All notebooks are archived when they are complete and no longer in use.

Figure 12.1 Organics

In the organic areas, the following information is verified when applicable to the method being reviewed.

- Check dates (e.g., extraction, calibration, analysis) and verify that holding times are met.
- All criteria for calibration, instrument tuning, internal standard areas, retention times, surrogate recoveries and analytical quality control results are checked.
- Check all method quality control data (e.g., blanks, spikes, duplicates, etc.) to assure the correct type and amount of checks are performed and results are within control limits.
- Compounds identified on the quantitation report were confirmed and agree with results reported on data sheets.
- All calculations such as total volatile hydrocarbons, soil concentrations, percent recoveries and dilutions are checked.
- All irregularities are properly documented and if necessary data flagged when pre-established control limits are not met.

Figure 12.2 Inorganics and Microbiology

In the inorganic and microbiological analytical areas, the second analysts check the following items prior to results being entered into the data management system.

- Check dates (extraction, digestion, calibration, incubation, analysis) and verify that holding times are met.
- All calibration criteria are met.
- Check all method quality control data (e.g., blanks, QCS, spikes, etc.) to assure the correct type and amount of checks are performed and results are within control limits.
- Check all calculation or data entry into calculation programs designed to calculate final results. Calculated results are checked against data bench worksheets for transcription errors.
- Check to be sure any irregularity is documented and; if necessary, data flagged when pre-established control limits are not met.
- Check reasonableness of data relationships (e.g., ammonia nitrogen results should not exceed total Kjeldahl nitrogen results).

Figure 12.2 Example report.

DIVISION OF WATER QUALITY

Chemistry Laboratory Report / Water Quality

COUNTY : MECKLENBURG
RIVER BASIN: CTB34

REPORT TO : MRO Regional Office

Other :
COLLECTOR(S) : BLOVE

PRIORITY
[x] AMBIENT [ ] QA
[ ] COMPLIANCE [ ] CHAIN OF CUSTODY
[ ] EMERGENCY

VISIT ID
V121155

[W] SAMPLE TYPE
[X] STREAM [ ] EFFLUENT
[ ] LAKE [ ] INFLUENT
[ ] ESTUARY

Lab Number :
Date Received:
Time Received:
Received By :

Data Released :
Date Reported:

Estimated BOD Range:

Station Location: SUGAR CR KATNC51 AT FINEVILLE

Seed: Chlorinated:

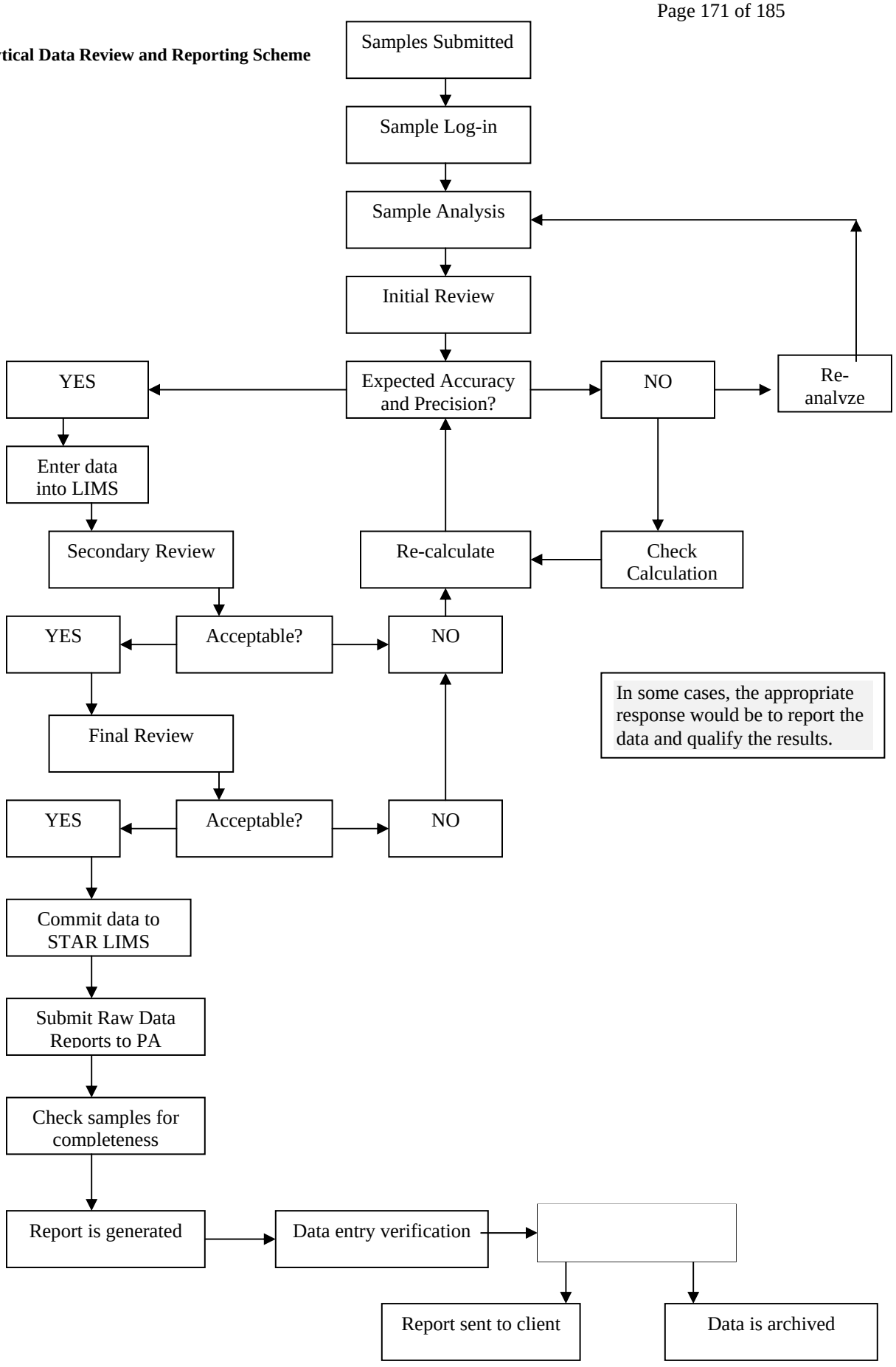
Remarks:

Station #	Date Begin (yy/mm/dd)	Date End (yy/mm/dd)	Time Begin	Time End	Depth - D/M/D/B/D/B/M	Value Type - A/H/L	Composite - T/S/B
C9050000	20030102		1030		0.1		

	BOD 310		mg/L		Chloride 940	mg/L	x	N-Bas N610	0.02	mg/L		Li-Lithium 1132
	COD High 340		mg/L		Chl a Tri 32217	ug/L	x	TKN an N625	0.50	mg/L		Mg-Magnesium 927
	COD Low 335		mg/L		Chl a Cor 32209	ug/L	x	NO2 plus NO3 as N630	2.9	mg/L		Mn-Manganese 1055
x	Coliform MF Fecal 31616	150 QL	/100ml		Pheophytin a 32213	ug/L	x	P, Total as P665	0.28	mg/L		Na-Sodium 929
	Coliform MF Total 31504		/100ml		Color: True 80	cu		PO4 as P 70507		mg/L		Arsenic Total 1002
	Coliform tube Fecal 31615		/100ml		Color: (pH) 83 pH=	cu		P, Dissolved as P666		mg/L		Se-Selenium 1147
	Coliform Fecal Strep 31673		/100ml		Color: pH 7.6 82	cu		K-Potassium		mg/L		Hg-Mercury 71900
	Residue Total 500		mg/L		Cyanide 720	mg/L		Cd-Cadmium 1027		ug/L		Ba, Barium
	Volatile 505		mg/L		Fluoride 951	mg/L		Cr-Chromium Total 1034		ug/L		Organochlorine Pesticides
	Fixed 510		mg/L		Formaldehyde 71880	mg/L		Cu-Copper 1042		ug/L		Organophosphorus Pesticides
	Residue Suspended 530		mg/L		Grease and Oils 556	mg/L		N-Nickel 1067		ug/L		
	Volatile 535		mg/L		Hardness Total 900	mg/L		Pb-Lead 1051		ug/L		Acid Herbicides
	Fixed 540		mg/L		Specific Cond 95	umhos/cm2		Zn-Zinc 1092		ug/L		
	pH 403		units		MBAS 38260	mg/L		V-Vanadium		ug/L		Base/Neutral & Acid Extractable Org
	Acidity to pH 4.5 436		mg/L		Phenols 32730	ug/L		Ag-Silver 1077		ug/L		TPH Diesel Range
	Acidity to pH 8.3 435		mg/L		Sulfate 945	mg/L		Al-Aluminum 1105		ug/L		
	Alkalinity to pH 8.3 415		mg/L		Sulfide 745	mg/L		Be-Beryllium 1012		ug/L		Purgeable Organics (VOA bottle req
	Alkalinity to pH 4.5 410		mg/L		Boron			Ca-Calcium 916		mg/L		TPH Gasoline Range
	TOC 680		mg/L		Tannin & Lignin	ug/L		Co-Cobalt 1037		ug/L		TPH BTEX Gasoline Range
x	Turbidity 76	16	NTU		Hexavalent Chromium	ug/L		Fe-Iron 1045		ug/L		Phytoplankton
	Coliform Total Tube		"/100mls		Bicarbonate	mg/L						
					Carbonate	mg/L						

COMMENTS:

Figure 12.3 - Analytical Data Review and Reporting Scheme



13.0 Corrective Actions

Quality control elements are used to monitor and assess the validity of sampling and analysis activities. Formal corrective actions will be initiated if data are determined to be of questionable validity or if QC elements are not within required limits. When QC deficiencies or nonconformance situations exist, corrective action procedures provide a systematic approach to assess and restore laboratory analytical system integrity. For routine problems, the analyst corrects the problem and documents the process on the raw data in the analytical run log or on the bench worksheet and a formal corrective action report is not required. Any laboratory employee that becomes aware of a problem related to one or more samples which cannot be immediately resolved, is responsible for initiating a corrective action investigation.

Quality control elements generally monitored by the DWQ Laboratory Section are listed in Section 5 (QA Targets for Precision, Accuracy and MDLs/PQLs), Section 9 (Calibration Procedures and Frequency) and Section 11 (Quality Control Checks and Routines to Assess Precision and Accuracy and Calculation of Method Detection Limits). Other method-specific QC elements are also monitored during routine operations. Table 13.1 identifies the QC elements routinely monitored by the Laboratory Section, and lists the most appropriate corrective actions that should be taken if criteria are not met. Analytical SOPs detail algorithms for parameter-specific corrective action procedures.

Corrective actions are initiated based on either the internal QC checks, data validation or performance audits. Outside sources such as performance evaluation studies, split samples, as well as recommendations by EPA, will also initiate corrective actions.

13.1 Procedures for Reporting Exceptions

Significant deviations from standard policies or practices of the laboratory are reported to the client and documented with the analytical reports. Any samples that are prepared or analyzed beyond accepted holding times have a qualifier code reported with the data alerting the client to the fact that tests were conducted after the sample had expired. Similarly, the failure of any quality control checks is commented with the data via qualifier codes, directing the client to the Sample Anomaly Report for details of failures. All other significant observations that do not conform with accepted practices or policies are documented and reported along with analytical results.

13.1.1 Sample Anomaly Report (SAR) Form

Corrective actions necessary to obtain acceptable results are implemented and documented. Corrective action at the bench level is documented on the raw data or through the use of a SAR form (Figure 13.1). The action is approved by the Supervisor, Branch Manager, and QA Officer and a copy is placed in each applicable analytical batch folder.

A Sample Anomaly Report documents laboratory quality control and quality assurance issues that warrant further investigation. These forms are logged into a SCUR/SAR database with the original retained in the sample report files and copies provided to the client and any applicable analytical data folders.

13.1.2 Sample Condition Upon Receipt (SCUR) Form

The Sample Condition Upon Receipt (SCUR) form (Figure 13.2) is used by sample receipt personnel to document a nonconformance found during log-in. These are logged into a SCUR/SAR database with the original retained in the sample report files and copies provided to the client and any applicable analytical data folders. Section 7.0 describes how this form is used.

If there is a critical problem that requires immediate action in consultation with the client (e.g., samples received after holding time expired, insufficient sample volume), the client if notified immediately and the corrective action designed in consultation with the client is documented on the form.

13.1.3 Audit Reports and PT Results Reports

An additional type of corrective action documentation is a formally presented report of findings and resolutions for internal and external audits and PT results. These reports are filed in the QA Office with the audit and are distributed to parties interested in the audit findings.

13.2 Quality Control Batch Problems

A measurement system may be out of control when QC samples fall outside of the limits described in Section 5 (QA Targets for Precision, Accuracy and MDLs/PQLs), Section 9 (Calibration Procedures and Frequency) and Section 11 (Quality Control Checks and Routines to Assess Precision and Accuracy and Calculation of Method Detection Limits).

An entire batch of samples may require corrective action if these quality control criteria are not met. Supervisors and/or the analyst will decide if re-analysis, re-extraction, etc. is necessary. Re-analysis would be noted in the folder with both sets of results included and clearly identified. The supervisor reviews both sets of data to determine if the problem has been resolved.

The EPA recommends the following guidelines for assessing acceptable data. If any data is determined to be out of control, one or all of the following should be followed:

- Review the method with the analyst.
- Re-analyze the sample batch and evaluate the new results.
- Recalibrate the instrument with freshly prepared reagents and reanalyze the samples.
- Re-extract and/or re-analyze the samples per method.
- Evaluate the data and sample behavior and investigate any possible chemical interferences.
- Check instrument for possible maintenance requirements.
- Seek additional help from other analysts or provide additional training for laboratory personnel.
- Perform a system audit to evaluate corrective action measurements.

13.3 Sample Collection Problems

Samples may have to be re-collected if review of the data related to the sample collection, preservation, storage and custody indicate that representative, compliant samples were not obtained. The findings and corrective action procedures are documented on the appropriate SCUR or SAR form.

13.4 Systematic Problems

Those problems of a procedural/system nature generally require the supervisor's and/or branch manager's involvement. Examples might include previously reported data that has been affected by a situation requiring correction or if corrective action will impact project schedule or budget. If previous data is affected, the laboratory management staff is responsible for determining the significance of the problem and notifying the customer, of any event that casts significant doubt on the validity of the data. This notification must be documented.

13.5 Departures from Documented Policies or Procedures

Due to the frequently unique nature of environmental samples, sometimes departures may be needed from documented policies and procedures. When the analyst encounters such a situation, the problem is presented to his/her supervisor for advice. The supervisor may elect to discuss it with the branch manager or QA/QC Coordinator or may contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst so notes it on the raw data and/or on a SAR form. This information can then be supplied to the client in the form of a footnote or on a Sample Anomaly Report.

13.6 External Corrective Actions

Any actions deemed necessary by external regulatory or certifying agencies such as EPA would be taken. These actions are most likely to arise from a system or performance audit, or from data review conducted by the agency.

13.7 Complaint Handling

Addressing complaints is a normal function of conducting business and a valuable tool to improve service to and relationships with clients. The Laboratory Section's goal is expeditious resolution of complaints.

The Laboratory Section is committed to resolving complaints and implementing suggestions for improvement. All informal complaints, suggestions or requests for information are directed to the appropriate staff for resolution. If immediate resolution cannot be attained, the matter is passed through the chain-of-command, ultimately to the Section Chief who may investigate and direct the resolution. Formal written complaints submitted to the Section are responded to in writing after investigation and resolution. Copies of responses are kept for reference.

13.8 Immediate vs. Long Term Corrective Action

Immediate corrective actions are necessary to correct or repair non-conforming equipment and systems. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis.

Long-term corrective actions are necessary to eliminate causes of nonconformance. The need for such actions will probably be identified by audits. Examples of this type of action include:

- Staff training in technical skills or in implementing the quality assurance program.
- Rescheduling of laboratory routine to ensure analyses are performed within hold times.
- Identifying vendors to supply reagents of sufficient purity.
- Revision of quality assurance system or replacement of personnel.

Corrective action may also be initiated by various auditing authorities when deemed necessary. For either immediate or long-term corrective actions, steps comprising a closed-loop corrective action system are as follows:

- Define the problem.
- Assign the responsibility for investigating the problem.
- Investigate and determine the cause of the problem.
- Determine a corrective action plan to eliminate the problem.
- Assign and accept responsibility for implementing the corrective action.
- Establish effectiveness of the corrective action and implement the correction.
- Verify that the corrective action has eliminated the problem.

Table 13.1 Guide to Corrective Actions for QC Elements Monitored by the Laboratory Section.

QC Activity	Acceptance Criteria	Recommended Corrective Action
Initial Calibration	See method or Section 9	♦ Reanalyze standards. ♦ Review standard preparation logs for calculation/dilution errors or expired sources. ♦ Prepare fresh calibration standards and analyze new calibration curve. ♦ Evaluate instrument operation and perform preventive maintenance if needed.
Initial Calibration Verification Standard	See method or Section 9	♦ Reanalyze standard. ♦ Take corrective action for initial calibration.
Continuing Calibration Verification Standard	See method or Section 9	♦ Reanalyze standard. ♦ Review standard preparation logs for calculation/dilution errors or expired sources. ♦ Prepare fresh calibration standard and analyze. ♦ Take similar corrective action as for initial calibration.
Interference Check Standard (ICP only)	See method	♦ Reanalyze standard. ♦ Review standard preparation logs for calculation/dilution errors or expired sources. ♦ Prepare fresh standard and analyze. ♦ Evaluate instrument operation and perform preventive maintenance if needed.
MS tuning standard (GC/MS only)	See method	♦ Re-tune instrument using FC-43 (PFTBA). ♦ Reanalyze tune calibration standard (BFB/DFTPP). ♦ Review standard preparation logs for calculation/dilution errors or expired sources. ♦ Evaluate instrument operation and perform preventive maintenance if needed.
Method Blanks	Less than ½ the PQL with exceptions noted in analytical SOPs.	♦ Reanalyze the method blank. ♦ Determine the source of contamination (reagents, storage and analysis environment, equipment, improper cleaning of labware, reagent water, etc.). ♦ Re-prepare/re-analyze all associated samples. Note: Re-analysis may not be necessary if no samples in the batch contain the analyte(s) of interest detected in the method blank.
Matrix Spikes	See Section 5 or method	♦ Reanalyze. ♦ Review results for calculation errors. ♦ Review other QC samples in the analysis batch. Perform corrective actions for these QC samples. ♦ Analyze a LCS prepared in the same analytical batch as the suspect matrix spike. If the LCS meets criteria, report exception as due to possible matrix effect. ♦ If the LCS fails criteria, review standard preparation logs for calculation/dilution errors or expired solutions. ♦ Analyze the matrix spiking solution to confirm that is was prepared correctly. ♦ Re-prepare/re-analyze all associated samples.
Duplicates/matrix spike duplicates	See Section 5 or method	♦ Reanalyze. ♦ Review results for calculation errors. ♦ Review other QC samples in the analysis batch. Perform corrective actions for these QC samples. ♦ Analyze a LCS prepared in the same analytical batch. If the LCS meets criteria, report exception as due to possible matrix effect. ♦ Review sample preparation protocols to ensure that samples are homogenized before preparation/analysis. ♦ Re-prepare/re-analyze all associated samples.

QC Activity	Acceptance Criteria	Recommended Corrective Action
Laboratory Control Sample	See Section 5	♦ Reanalyze. ♦ Review results for calculation errors. ♦ Review standard preparation logs for calculation/dilution errors or expired solutions. ♦ Review other QC samples in the analysis batch. If other QC samples in batch meet criteria, re-evaluate the need for corrective action. ♦ If the failed LCS is combined with failed matrix spikes or duplicates for the same spiked parameters, re-prepare/re-analyze all associated samples.
Surrogates	See method or analytical SOP	♦ Reanalyze. ♦ Evaluate the analytical results for unusual matrix effects (presence of chromatographic humps, etc.). ♦ Review results for calculation errors. ♦ Review standard preparation logs for calculation/dilution errors or expired solutions. ♦ Re-prepare/re-analyze. ♦ Review QC samples in the analysis batch. If other QC samples in batch meet criteria, additional corrective action may not be necessary.
Internal Standards	See method	♦ Follow method guidelines.
Trip blanks (VOA only)	Less than PQL	♦ Check related method blank for contamination.
Titrating Solutions	See method or analytical SOP	♦ Review results for calculation errors. ♦ Review standard preparation logs for calculation/dilution errors or expired solutions. ♦ Reanalyze all samples form last acceptable titration solution check.
Microbiology + and - controls for media	Should be + and -, respectively	♦ Reject medium.
Sample results	Calibration	♦ If the calibration fails for a target and the corresponding target is not detected, the results may be reported as <PQL if the PQL standard is analyzed and detected.
	Spike criteria limits	♦ If a limited list MS or LCS is high biased and no targets are detected above the PQL, results may be reported as <PQL. When a full compound spike is utilized, and the MS or LCS result is high biased, and the corresponding target is not detected, the result for the corresponding target may be reported as <PQL, regardless of the other targets.
	Surrogate criteria limits	♦ If surrogate recovery is high biased and no target is detected, the results are reported as <PQL.

Figure 13.1 Sample Anomaly Report (SAR) Form

NC DENR/DWQ Chemistry Laboratory
Sample Anomaly Report (SAR)

Report to: _____

Lab Number: _____ Sample ID: _____

Station Location: _____ County: _____ Region: _____

Sample Type: _____ Priority: _____ Collector: _____

Date collected: _____/_____/_____ Date received: _____/_____/_____ Date analyzed: _____/_____/_____

Affected Parameter(s): _____

Analytical Area (check one):

❑ WCH ❑ METALS ❑ VOA ❑ SVOA
❑ NUT ❑ MICRO ❑ PEST

The following anomalies occurred (**check all that apply**):

❑ **Samples**
❑ Improper container used
  ❑ VOA vials with headspace
  ❑ Sulfide samples with headspace
❑ Samples not received, but listed on fieldsheet
❑ Samples received, but not listed on fieldsheet
❑ Mislabeled as to tests, preservatives, etc.
❑ Holding time expired
  ❑ Prior to receipt in lab
  ❑ After receipt in lab
❑ Insufficient quantity for analysis
❑ Sample exhibits gross non-homogeneity
❑ Sample not chemically preserved properly
  ❑ pH out of range (record pH): _____
  ❑ Improper chemical
❑ Residual chlorine present in sample
❑ Color interference
❑ Heavy emulsion formed during extraction
❑ Sample bottle broken in lab - no reportable results

❑ **Quality Control**
❑ Instrument failure – no reportable results
❑ Analyst error – no reportable results
❑ Surrogates
❑ None added
❑ Recovery outside acceptance limits
❑ Spike recovery
❑ None added
❑ Recovery outside acceptance limits
❑ Failed to meet criteria for precision
❑ Internal standards
❑ Blank contamination
❑ QC data reported outside of controls (i.e. QCS, LCS)
❑ Incorrect procedure used
❑ SOP intentionally modified with QA and Branch Head approval
❑ Invalid instrument calibration
❑ Elevated detection limits due to:
❑ Insufficient sample volume

❑ **Other** (specify):_____
_____
_____

Comments: _____
_____

Corrective Action:

❑ Samples were rejected by DWQ Lab. Authorized by: _____ Date: _____/_____/_____
❑ Accepted and analyzed after notifying the collector or contact person and determining that another sample could not be secured.
❑ Sample(s) on hold until: _____
❑ Sample reported with qualification. Data qualification code used: _____
❑ Other (explain): _____
_____
Notification Required (circle one)? Yes No
Person Contacted: _____ Date: _____/_____/_____

Form completed by: _____ Date: _____/_____/_____

Lead Chemist Review (initial):

❑ BIOCHEM _____ ❑ PEST _____ ❑ SVOA _____
❑ METALS _____ ❑ VOA _____

Branch Head Review (initial): _____
QA/QC Review (initial): _____ Logged into database by (initial): _____

Figure 13.2 Sample Condition Upon Receipt (SCUR) Report

NC DENR/DWQ Chemistry Laboratory
Report to: _____ **Sample Condition Upon Receipt Anomaly Report (SCUR)**

The condition of these samples were not acceptable because (**check all that apply**):

- ❑ **Coolers**
- ❑ Samples were not received on wet ice
- ❑ No temperature blank submitted (see comments):
  - ❑ Sample T° reading : _____°C
  - ❑ Cooler T° reading: _____°C
- ❑ Temperature >6°C. T° reading: _____°C
- ❑ Samples frozen

- ❑ **Containers**
  - ❑ Leaking
  - ❑ Broken
  - ❑ Without labels
  - ❑ VOA vials with headspace
  - ❑ Sulfide samples with headspace

- ❑ **Container Labels**
  - ❑ Not the same ID/info. as on COC
  - ❑ Not the same ID/info. as on fieldsheet
  - ❑ Incomplete. Missing the following:
    - ❑ Station #/Sample ID
    - ❑ Collection date
    - ❑ Collector
    - ❑ Analysis
    - ❑ Preservative
    - ❑ Other: _____
  - ❑ Markings smeared or illegible
  - ❑ Torn

- ❑ **Samples (affected samples are described below)**
  - ❑ Samples not received, but listed on fieldsheet
  - ❑ Samples received, but not listed on fieldsheet
  - ❑ Samples not received, but listed on COC
  - ❑ Samples received, but not listed on COC
  - ❑ Mislabeled as to tests, preservatives, etc.
  - ❑ Holding time expired
  - ❑ Improper container used
  - ❑ Insufficient quantity for analysis

- ❑ **Chain of Custody**
  - ❑ No custody seals
  - ❑ Custody seals not intact
  - ❑ Not relinquished.
    - ❑ No date/time relinquished
    - ❑ No signature
  - ❑ Incomplete information

- ❑ **Documentation**
- ❑ Fieldsheet wet/illegible
- ❑ Fieldsheet incomplete: _____
- ❑ Records not written in indelible ink
- ❑ Sample(s) submitted without fieldsheet

- ❑ **Other (specify)**: _____
  _____
  _____

Comments: _____
_____

Corrective Action:

- ❑ Samples were rejected by DWQ Lab. Authorized by: _____
- ❑ Accepted and analyzed per collector's request after notifying the collector.
- ❑ Accepted and analyzed after notifying the client and determining that another sample could not be secured.
- ❑ Sample(s) on hold until: _____
- ❑ Sample(s) accepted and analyzed. No notification required.
- ❑ Other (explain): _____

Person Contacted: _____ Date: _____/_____/_____

Form completed by: _____Date: _____/_____/_____

Lead Chemist Review (initial):
❑ BIOCHEM _____ ❑ VOA _____ ❑ PEST_____
❑ MET _____ ❑ SVOA _____

Branch Head Review (initial): _____
QA/QC Review (initial): _____ Logged into database by (initial): _____

14.0 Performance and Systems Audits

Internal and external audits are conducted regularly within the DWQ Laboratory Section to ensure that the guidance provided in this document and in other related documents is followed. Internal audits are performed by the QA department, which is responsible for all QA/QC functions in the laboratory, and/or members of the professional laboratory staff that do not normally work in the section or analytical unit being audited. External audits are conducted by persons who are not direct employees of the DWQ Laboratory Section (generally EPA Region 4) to provide an independent and unbiased review of laboratory operations.

There are two types of audits: systems audits and performance audits.

1) Systems audits involve an in-depth review and evaluation of some or all components of the analytical laboratory to determine the proper application of guidelines listed in the Quality Management Plan (QMP) and Quality Assurance Manual (QAM).
2) Performance audits require the analysis of blind samples or other samples whose values are not known to the analytical areas. These results are used to evaluate the accuracy of the laboratory analytical system.

14.1 Systems audits

Systems audits may be initiated either internally or externally.

14.1.1 Internal audits

It is the responsibility of the QA/QC Coordinator to plan and organize audits as required by a predetermined schedule and as requested by management. Such audits shall be carried out by the QA/QC Coordinator and/or trained and qualified personnel who are, wherever resources permit, independent of the activity being audited. Personnel shall not audit their own activities except when it can be demonstrated that an effective audit will be carried out. System audits evaluate procedures and documentation in the laboratory. Additional audits may be necessary throughout the year to address specific project requirements, problem troubleshooting or issues that arise from other audits.

The QA/QC Coordinator conducts several systems audit during each calendar year. During these audits, one or more components of the laboratory will be reviewed to determine if that part is functioning in compliance with the Laboratory Section Quality Management Plan, the Laboratory Section Quality Assurance Manual, the approved standard operating procedures and approved methodology. An audit report will include a list of deficiencies that must be addressed in order to correct or improve the laboratory operations.

(1) Selected systems will be audited every three months with a goal of auditing all systems once per year.

(2) The QA/QC Coordinator will conduct the audits.

(3) The audit will consist of the submittal of blind samples and/or the random selection of previously reported samples, tracking of these samples through the system, evaluation of sample results, and a follow-up laboratory audit.

(4) System components to be audited will include, but are not limited to:

(i) All documentation associated with sample and data handling, to include linkage mechanism employed between all records for tracking documentation for any sample data result.

(ii) Use of established, approved procedures as outlined in this Quality Assurance Manual.

(iii) Personnel training records.

(iv) Proper execution of established procedures.

(v) Anomaly reports and follow-up to corrective actions from previous audits, external audits, performance testing (PT) samples or blind samples.

(vi) Sample and data handling activities include:

[a] All sample log-in, routing and disposal.

[b] Sample preparations

[c] Method calibrations

[d] Sample analyses

[e] Data reduction, validation and reporting

[f] Preventive maintenance and repair procedures

[g] Standard and reagent preparation, documentation and storage

[h] Sample and waste disposal

[i] Container and labware decontamination

[j] QC management practices and assessment of analytical precision, accuracy and sensitivity

(5) Deficiency lists and associated corrective action orders will be formally communicated to responsible staff.

14.1.2 External audits

External audits are performed when certifying agencies or clients submit sample for analysis and/or conduct on-site inspections. It is the Laboratory Section's policy to cooperate fully with certifying agencies. It is also our policy to comply fully with system audits conducted by regulatory agencies and clients. Currently, these include:

(1) EPA, Region IV; for selected systems, on an 18-36 month basis, depending on budget constraints

(2) USGS; selected systems; per-project basis

14.2 Performance Audits

The laboratory is involved in external performance audits conducted annually through the analysis of PT samples provided by this lab and a third party.

14.2.1 Blind Sample Audits

The QA/QC Coordinator conducts internal performance evaluations using commercially prepared samples as blind samples. The results of these audits will be documented and reported to the supervisors, branch managers and Section Chief so that any necessary adjustments can be made.

Blind sample audits may be initiated upon observed or suspected problem with specific system and split sample analyses by specific, per-project agreement with regulatory and commercial laboratories. Reports of results, deficiencies and corrective actions are communicated as with internal system audits, with the addition of reports to affected external organizations.

Blind sample audits are performed by submitting QC samples to the analyst. The true values are only made known after the test is complete. Blind sample audits are carried out by the QA/QC Coordinator, clients and certifying agencies as necessary to assure the laboratory is capable of achieving success with a blind QC sample.

14.2.2 Performance Testing samples

The lab participates in an annual Section-wide internal Performance Testing (PT) program to evaluate methods that are not commonly included in the PT studies.

Holding time begins when the vial is opened. Full volume PTs follow normal hold time procedures and storage requirements unless the vendor-supplied directions instruct otherwise. Login will obtain the documentation provided with the PTs and fieldsheets will be reviewed by the QA/QC Coordinator or other designated staff prior to delivery to the analytical work areas.

Vials will be prepared as required in the instruction set provided with the samples. After preparation to full volume, the sample may be spiked, digested, concentrated, etc., as would be done for any normal sample requiring similar analysis. PT samples will not undergo multiple preparations, multiple runs, multiple methods (unless being used to evaluate multiple methods), multiple dilutions, unless this is what would be done to a normal client sample. No special reviews shall be performed by operation and QA, unless this is what would be done to a normal client sample. To the degree that special report forms or login procedures are required by the PT supplier, it is reasonable that the laboratory would apply special review procedures, as would be done for any client requesting unusual reporting or login processes. Special QC samples can be included in the analytical run if this is what would be done with normal client samples under similar circumstances.

It is, however, recognized that PT samples are often not representative of "real world" samples either in their form (e.g., vials) or content (e.g., multiple target analyte hits) and as such, present the laboratory with special challenges. It is the policy of DWQ that PT samples be treated as typical samples in the normal production process whenever this is possible. Further, where PT samples present special or unique problems in the normal production process they need to be treated differently, as would any special or unique request submitted by any client.

14.3 Quality Systems Management Review

The QA/QC Coordinator conducts an annual review of its quality systems to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. Managers may be included in this process.

This review uses information generated during the preceding year to assess the "big picture" by ensuring that routine quality actions taken and reviewed on a quarterly basis are not components of larger systematic concerns. The quarterly review (Section 15) should keep the quality systems current and effective, therefore the annual review is a formal senior management process to review specific existing documentation.

Significant issues from the following documentation are summarized by the QA/QC Coordinator prior to the review meeting:

- Matters arising from the previous annual review.
- Prior Monthly Quality Assurance Reports.
- Review of report reissue request.
- Minutes form prior meetings.
- Internal and External audits.

Consider:

- Adequacy of staff, equipment and facility resources.
- Future plans for resources and testing capability and capacity.

14.4 Corrective Actions

All deficiencies found during audits are reported to the Section Chief. Audit information is also provided through a monthly report. The Section Chief and QA/QC Coordinator agree upon a time frame for correction. The lab's response and corrective action procedures are evaluated by the QA/QC Coordinator and when acceptable, are attached to each audit and filed. If issues arise that may require method suspension or restriction, the procedures in Section 13 are followed.

External audits often require written reports that include proof of correction. The QA coordinates this written response. Written responses to PT's may be required. The response must address the reason for any "unacceptable" or "check for error" result. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

14.5 Report Audits

Routine report audits are the responsibility of the laboratory Quality Assurance Officer. The QA Officer performs an independent systems review of reports generated by the laboratory. The reviewer is not expected to pursue the correctness of every reference in the file contents, but concentrates on the internal consistency of the data package. Areas for review include COC, correspondence with the analytical request, batch QC status, completeness of any corrective action statements, 5% of calculations, format, holding time, sensibility and completeness of the file contents. A list of reports reviewed is maintained in an audit file.

15.0 Quality Assurance Reports

Quality assurance reports to laboratory management are required to keep them informed about how the laboratory QA program is progressing. Items in which performance is not satisfactory are addressed and a plan for corrective action prepared and implemented.

15.1 Internal Reports

A quarterly QA report is prepared by the QA/QC Coordinator. This information is circulated to the Section Chief, Branch Managers and unit supervisors. An example format with the minimum required topics for reporting is illustrated in Figure 15.1.

Reports of internal laboratory audits and all performance audits are addressed to the Section Chief, who in turn distributes them to the management staff for corrective action, as needed. Results of external laboratory audits are routed to the management staff through the Section Chief for corrective action, if required. The QA/QC Coordinator ensures that corrective actions are implemented.

15.2 External Reports

The QA/QC Coordinator will prepare external QA reports for specific projects, agencies or clients that may require it. These will be addressed to the client or data user at the frequency and in the format mandated by the specific project requirements.

Figure 15.1 Monthly QA Report to Management Format

QA MONTHLY REPORT TO MANAGEMENT

LABORATORY:
ANALYTICAL UNIT:
PERIOD COVERED:
PREPARED BY:

TO: Steve Tedder, Section Chief
CC: Branch Manager(s)

KEY ISSUES:

1.
2.
3.

A. SOPs
- The following SOPS were finalized (include updated SOP summary with report):
- The following SOPs are in QA for review:
- The following SOPs are due to QA:

B. CORRECTIVE ACTION REPORTS (SARs/SCURs)
- Total number of SARs:
- Total number of SCURs:
- Number of unresolved SARs:
- Highlights:

C. MDLs/IDOCs
- MDLs completed:
- MDLs due:
- IDOCs completed:

D. AUDITS
- INTERNAL AUDITS (The following internal audits were performed - include method and general)
- EXTERNAL AUDITS (Include source, date, highlights, date corrective action package is due, progress on corrective actions)

E. PE SAMPLES
- The following PE samples are now in-house (due dates):
- The following PE results have been received (results presented as a percentage by Unit, discuss corrective action)

F. TRAINING
- Training record issues

G. MISCELLANEOUS

H. NEXT MONTH (Items planned for next month)

16.0 Selected References

1. "Definition and Procedure for the Determination of the Method Detection Limit- Revision 1.11", 40 CFR Part 136, Appendix B.

2. Handbook for Analytical Quality Control in Water and Wastewater, EPA 600/4-79-019, March 1979.

3. Methods for Chemical Analysis of Water and Wastes, USEPA Office of Research and Development, Rev. 3/83. Cincinnati, OH, 3/83; EPA 600/4-79-020.

4. Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods, SW-846; 3rd edition (9/86), with Final Updates I (7/92), II (9/94), IIA (9/93) and IIB (1/95); USEPA Office of Solid Waste and emergency Response, Washington, D.C.

5. Method for the Determination of Organic Compounds in Drinking Water, Supplement I, EPA 600/4-90/020, July 1990.

6. Standard Methods for the Examination of Water and Wastewater (designated SM), 18th Edition, American Public Health Association, Washington, DC, 1992.

7. Standard Methods for the Examination of Water and Wastewater (designated SM), 19th Edition, American Public Health Association, Washington, DC, 1995.

8. Standard Methods for the Examination of Water and Wastewater (designated SM), 20th Edition, American Public Health Association, Washington, DC, 1998.

9. Code of Federal Regulations, Title 40, Part 136, U.S. Government printing office, Washington, D.C., July 1993.

10. Chemical Hygiene Plan, North Carolina Division of Water Quality, Laboratory Section - Central Laboratory, October 1, 1997.

11. Chemical Hygiene Plan, North Carolina Division of Water Quality, Laboratory Section - Washington Regional Office, April 2003.

12. Chemical Hygiene Plan, North Carolina Division of Water Quality, Laboratory Section - Asheville Regional Office, March 5, 2001.

ATTACHMENT 3:
NC SURFACE WATER QUALITY STANDARDS

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Summary Table of Surface Water Standards
http://portal.ncdenr.org/web/wq/ps/csu/swstandards

Disclaimer: This table is intended to provide summary information only. It does not substitute for any written regulation, nor is it a regulation itself. **May 1, 2007**

The following standards, criteria, or toxic concentrations are adopted per 15A NCAC 2B. See last page for appropriate use information.

Pollutant	CAS	Freshwater Aquatic Life	Saltwater Aquatic Life	Water Supply (WS)[1]	Human Health (HH)[2]	Trout Waters (Tr)	High Quality Waters (HQW)	Swamp Waters (Sw)	Synonyms & Other Info	Carcinogen
		ug/l (unless noted)	ug/l (unless noted)	ug/l (unless noted)	ug/l (unless noted)	ug/l (unless noted)	ug/l (unless noted)			
Aldrin	309-00-2	0.002	0.003	0.05 ng/L	0.05 ng/L					y
Arsenic	7440-38-2	50	50	10	10					y
Barium	7440-39-3			1.0 mg/L						n
Bacterial Indicators	*see enterococcus and fecal coliform*									NA
Benzene	71-43-2			1.19	51					y
Beryllium	7440-41-7	6.5								n
Cadmium	7440-43-9	2 (N)	5 (N)			0.4 (N)				n
Carbon Tetrachloride	56-23-5			0.254	1.6				Benzinoform; Carbon Chloride	y
Chlordane	57-74-9	0.004	0.004	0.8 ng/L	0.8 ng/L					y
Chloride	16887-00-6	230 mg/L (AL)		250 mg/L						n
Chlorine (TRC)	7782-50-5	17								n
Chlorinated Benzenes				488						y
Chlorinated Phenols				1.0 (N)						NA
Chlorophyll -a, corrected		40(N)	40(N)			15(N)				NA
Chromium		50	20							NA
Copper	7440-50-8	7 (AL)	3 (AL)							n
Cyanide	57-12-5	5 (N)	1							n
D, 2,4-	94-75-7			100					2,4-Dichlorophenoxy acetic acid	n
DDT, 4,4'-	50-29-3	0.001	0.001	0.2 ng/L	0.2 ng/L				4,4'-Dichlorodiphenyltrichloroethane	y
Demeton	8065-48-3	0.1	0.1							n
Dieldrin	60-57-1	0.002	0.002	0.05 ng/L	0.05 ng/L					y
Dioxin (2,3,7,8-TCDD)	1746-01-6			0.000005 ng/L	0.000005 ng/L				2,3,7,8-Tetrachlorodibenzo-p-dioxin	y
Dissolved Gases		110% sat (N)	110% sat (N)							NA

Summary Table of Surface Water Standards
http://portal.ncdenr.org/web/wq/ps/csu/swstandards

The following standards, criteria, or toxic concentrations are adopted per 15A NCAC 2B. See last page for appropriate use information.

Pollutant	CAS	Freshwater Aquatic Life	Saltwater Aquatic Life	Water Supply (WS)[1]	Human Health (HH)[2]	Trout Waters (Tr)	High Quality Waters (HQW)	Swamp Waters (Sw)	Synonyms & Other Info	Carcinogen
		ug/l (unless noted)	ug/l (unless noted)	ug/l (unless noted)	ug/l (unless noted)	ug/l (unless noted)	ug/l (unless noted)			
Dissolved Oxygen		not less than 5.0 mg/L (N)	not less than 5.0 mg/L (N)			not less than 6.0 mg/L (N)	not less than 6.0 mg/L (E)	(N)		NA
Endosulfan	115-29-7	0.05	0.009						Same values apply to Endosulfan Sulfate, alpha-Endosulfan, and beta-Endosulfan	n
Endrin	72-20-8	0.002	0.002							n
Enterococcus					geomean of 35 organisms/100 mL (applicable to class SA, SB, and SC Saltwaters) (N)					NA
Fecal Coliform (MFTCC/100mL)[3]					geomean of 200 organisms/100 mL in Class C Freshwaters (N); and a geomean of 14 organisms/100 mL in class SA Saltwaters (N)					NA
Fluoride		1.8 mg/L								NA
Guthion	86-50-0	0.01	0.01							NA
Hardness, Total				100 mg/L Calcium Carbonate						NA
Heptachlor	76-44-8	0.004	0.004	0.08 ng/L	0.08 ng/L					y
Hexachlorobutadiene	87-68-3			0.44	18				HCBD	y
Iron	7439-89-6	1.0 mg/L (AL)								n
Lead	7439-92-1	25 (N)	25 (N)							n
Lindane	58-89-9	0.01	0.004						gamma-BHC, g-HCH	c
Manganese	7439-96-5			200						n
MBAS[4]				500 (N)					Methylene-blue-active substances (see note)	NA

Summary Table of Surface Water Standards
http://portal.ncdenr.org/web/wq/ps/csu/swstandards

The following standards, criteria, or toxic concentrations are adopted per 15A NCAC 2B. See last page for appropriate use information.

Pollutant	CAS	Freshwater Aquatic Life	Saltwater Aquatic Life	Water Supply (WS)[1]	Human Health (HH)[2]	Trout Waters (Tr)	High Quality Waters (HQW)	Swamp Waters (Sw)	Synonyms & Other Info	Carcinogen
		ug/l (unless noted)	ug/l (unless noted)	ug/l (unless noted)	ug/l (unless noted)	ug/l (unless noted)	ug/l (unless noted)			
Mercury	7439-97-6	0.012	0.025							n
Methoxychlor	72-43-5	0.03	0.03							n
Mirex	2385-85-5	0.001	0.001							c
Nickel	7440-02-0	88 (N)	8.3 (N)	25						n
Nitrate (as N)	14797-55-8			10.0 mg/L					Total nitrogen may be regulated in NSW waters. See 2B .0200s for further info	n
Oil and Grease		(N)	(N)							NA
Parathion	56-38-2	0.013	0.178							n
PCB, total		0.001 (N)	0.001 (N)		0.064 ng/L (N)				polychlorinated biphenyls / (Total of all identified PCBs)	y
pH		6.0-9.0 (N)	6.8-8.5 (N)					(N)	Freshwater and Saltwater Aquatic Life Standards are listed as acceptable pH ranges	NA
Phenolic Compounds		(N)	(N)		(N)				(phenolic compounds: no fish flesh tainting)	NA
Polynuclear aromatic hydrocarbons (PAH's)[5]				0.0028 Total PAH's	0.0311 Total PAH's					y
Radioactive Substances		(N)	(N)		(N)					NA
Salinity			(N)							NA
Selenium	7782-49-2	5	71							n
Sewage		(N)	(N)	(N)						NA
Silver	7440-22-4	0.06 (AL)	0.1 (AL)							n
Silvex	93-72-1			10					2,4,5-TP; 2,4,5-Trichlorophenoxypropionoic Acid	n
Solids, settleable		(N)	(N)						also includes floating solids and sludge deposits	NA
Solids, total dissolved				500 mg/L						NA
Solids, total suspended		(N)				HQW=10 mg/L (E)	20 mg/L (E)			NA
Sulfates				250 mg/L						n
Temperature		(N)	(N)		(N)					NA
Tetrachloroethane, 1,1,2,2-	79-34-5			0.17	4				acetosol; acetylene tetrachloride	y
Tetrachloroethylene (PERC)	127-18-4			0.7	3.3				PERC; PCE; perchloroethylene	y

Summary Table of Surface Water Standards
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The following standards, criteria, or toxic concentrations are adopted per 15A NCAC 2B. See last page for appropriate use information.

Pollutant	CAS	Freshwater Aquatic Life	Saltwater Aquatic Life	Water Supply (WS)[1]	Human Health (HH)[2]	Trout Waters (Tr)	High Quality Waters (HQW)	Swamp Waters (Sw)	Synonyms & Other Info	Carcinogen
		ug/l (unless noted)	ug/l (unless noted)	ug/l (unless noted)	ug/l (unless noted)	ug/l (unless noted)	ug/l (unless noted)			
Toluene	108-88-3	11				0.36			methyl benzene; phenyl methane	n
Toxaphene	8001-35-2	0.2 ng/L	0.2 ng/L							y
Trialkyltin		0.07	0.007							n
Tributyltin (TBT)	56573-85-4	0.07	0.007							n
Trichloroethylene	79-01-6			2.5	30				TCE	y
Turbidity		50/25 NTU (N)	25 NTU (N)			10 NTU (N)				NA
Vinyl Chloride	75-01-4			0.025	2.4				chloroethylene	y
Zinc	7440-66-6	50 (AL)	86 (AL)							n

Footnotes, Codes, and Additional Information with Reference to Classifications and Standards

*To determine the appropriate standard, use the most stringent of all applicable columns.For Class C, use the most stringent of freshwater (or, if applicable, saltwater) column and the Human Health column.

* For a WS water, use the most stringent of Freshwater, WS & Human Health. Trout Waters & High Quality Waters likewise must adhere to the most stingent of all applicable standards

* All metal criteria are as total recoverable metals.

(AL) Action Level Standard - See 2B .0211 for additional information

(N) = Narrative standard See 2B .0211 and for WS: .0212, 0214,.0215, .0216 and.0218

(E) For effluent limits only. See 2B .0224

(NTU) Nephelometric Turbidity Units

(HQW) High Quality Waters - see 02B .0101 and .0201

(Sw) Swamp Waters - as defined by 02B .0101

(Tr) Trout Waters - as defined by 02B .0101 and 0301

1 WS standards are applicable to *all* Water Supply Classifications. WS standards are based on the consumption of fish and water. See 2B .0208 for equation.

2 Human Health Standards are based on the consumption of fish only unless dermal contact studies are available. See 2B .0208 for applicable equations.

3 MFTCC/100 mL = Membrane Filter Total Coliform Count per 100 mL of sample

4 MBAS: additional narrative language is located in 02B .0212, .0214, .0215, .0216, .0218

5 PAH=Applies to total PAHs present and includes the following: benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(k)fluoranthene, chrysene, dibenz(a,h)anthracene and indeno(1,2,3-cd)pyrene

Carcinogenicity Color Key:	
Known to cause cancer in humans (y)	**Blue**
Not known to cause cancer in humans (n)	**Green**
Possible human carcinogen (c)	**Yellow**
Carcinogenicity not assessed or Does Not Apply (NA)	**No Color**

Unit Conversions: 1.0 mg/L = 1000.0 ug/L = 1000000.0 ng/L
ng/L = 0.001 ug/L = 0.000001 mg/L

1.0

ATTACHMENT 4:
NC DWR ALMP STATION INFORMATION

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All stations are sampled for:

Temperature	Dissolved oxygen
Specific conductivity	pH
Secchi depth	Chlorophyll a
Phytoplankton	Nutrients
Turbidity	TSS
Total Solids	

Additional grab sampling is conducted at some stations on lakes classified as Water Supplies (WS-I through V) and Primary Recreation (B). Those stations are italicized. The additional samples are for chlorides, metals, and fecal coliform. Other parameters may be added as necessary.

NC DWR ALMP STATION INFORMATION								
Station Number	Location	Latitude	Longitude	County	Station Classification	ADDITIONAL SAMPLES BASED ON CLASSIFICATION		
						Chloride	Metals	Fecal coliform
BROAD RIVER BASIN								
BRD001C	LAKE LURE AT LURE COVE AT LAKE LURE NC	35.43468	-82.21496	RUTHERFORD	B Tr			
BRD001D1	LAKE LURE AT CENTER OF LAKE AT LAKE LURE NC	35.43371	-82.19558	RUTHERFORD	B Tr			
BRD001F	LAKE LURE AT SUNSET COVE AT LAKE LURE NC	35.43189	-82.19032	RUTHERFORD	B Tr			
BRD005Q	LAKE SUMMIT DS SR 1852 NR TUXEDO NC	35.23040	-82.40119	HENDERSON	C Tr			
BRD005R	LAKE SUMMIT AT MIDPOINT NR TUXEDO NC	35.22581	-82.41581	HENDERSON	C Tr			
BRD005T	LAKE SUMMIT AT DAM NR TUXEDO NC	35.22060	-82.42634	HENDERSON	C Tr			
BRD007J	LAKE ADGER AT MOUTH OF PANTHER LR NR SUNNY VIEW NC	35.33439	-82.22705	POLK	C			

NC DWR ALMP STATION INFORMATION								
Station Number	Location	Latitude	Longitude	County	Station Classification	ADDITIONAL SAMPLES BASED ON CLASSIFICATION		
						Chloride	Metals	Fecal coliform
BRD007L	LAKE ADGER AT MOUTH OF JACKSON COVE NR SUNNY VIEW NC	35.33794	-82.20303	POLK	C			
BRD007P	LAKE ADGER AT DAM NR SUNNY VIEW NC	35.33638	-82.18776	POLK	C			
BRD056C	KINGS MOUNTAIN RESERVOIR IN BUFFALO CR ARM NR STUBBS NC	35.30597	-81.46191	CLEVELAND	WS-III CA			
BRD056E	KINGS MOUNTAIN RESERVOIR IN WHITEOAK CR ARM NR STONY POINT	35.30769	-81.44880	CLEVELAND	WS-III CA			
BRD056G	KINGS MOUNTAIN RESERVOIR AT MIDPOINT NR STONY POINT	35.29375	-81.45052	CLEVELAND	WS-III CA			
BRD056J	KINGS MOUNTAIN RESERVOIR AT DAM NR OAKGROVE NC	35.27816	-81.45541	CLEVELAND	WS-III CA	X	X	
CHOWAN RIVER BASIN								
CHO0153A	MERCHANTS MILL POND AT EAST END	36.42490	-76.67684	GATES	C NSW			
CHO0154A	MERCHANTS MILL POND AT WEST END	36.42663	-76.68867	GATES	C NSW			
CAPE FEAR RIVER BASIN								
CPF0021A	LAKE HUNT AT UPPER END	36.33967	-79.72815	ROCKINGHAM	WS-III B CA			
CPF0022A	LAKE HUNT AT MID POINT	36.33085	-79.72688	ROCKINGHAM	WS-III B CA			
CPF0023A	LAKE HUNT AT DAM	36.32683	-79.72697	ROCKINGHAM	WS-III B CA	X	X	
CPF0025A	REIDSVILLE LAKE NR MONROETON NC	36.29704	-79.70488	ROCKINGHAM	WS-III NSW CA			
CPF002A1	REIDSVILLE LAKE NR FOUSHEE NC	36.28892	-79.68022	ROCKINGHAM	WS-III NSW CA	X	X	

NC DWR ALMP STATION INFORMATION								
Station Number	Location	Latitude	Longitude	County	Station Classification	ADDITIONAL SAMPLES BASED ON CLASSIFICATION		
						Chloride	Metals	Fecal coliform
CPF007A1A	LAKE BRANDT MIDWAY REEDY FORK ARM	36.17391	-79.85305	GUILFORD	WS-III NSW CA			
CPF007A4	LAKE BRANDT MIDWAY HORSEPEN CR ARM	36.16389	-79.84583	GUILFORD	WS-III NSW CA			
CPF007B	LAKE BRANDT AT DAM NR HILLSDALE NC	36.17123	-79.83965	GUILFORD	WS-III NSW CA	X	X	
CPF0251A	LAKE BURLINGTON BELOW TOMS CR	36.20014	-79.41279	ALAMANCE	WS-II HQW NSW CA			
CPF025A	LAKE BURLINGTON AT DAM AT UNION RIDGE	36.17911	-79.41108	ALAMANCE	WS-II HQW NSW CA	X	X	
CPF038F	LAKE MACKINTOSH AT NC HWY 61 NR WHITSETT NC	36.05225	-79.57031	ALAMANCE	WS-IV NSW CA			
CPF038G	LAKE MACKINTOSH AT NC 61 NR SEDALIA NC	36.03799	-79.57861	ALAMANCE	WS-IV NSW CA			
CPF038H	LAKE MACKINTOSH IN UPSTRAM END NR ALAMANCE NC	36.04502	-79.55837	ALAMANCE	WS-IV NSW CA			
CPF038J	LAKE MACKINTOSH DS SR 1149 NR ALAMANCE NC	36.03906	-79.52369	ALAMANCE	WS-IV NSW CA			
CPF038L	LAKE MACKINTOSH IN BEAVER CR ARM NR ALAMANCE NC	36.03313	-79.50705	ALAMANCE	WS-IV NSW CA			
CPF038N	LAKE MACKINTOSH AT DAM NR ALAMANCE NC	36.03892	-79.50370	ALAMANCE	WS-IV NSW CA	X	X	
CPF050A9	PITTSBORO TOWN LAKE AT UPPER END	35.71514	-79.18536	CHATHAM	WS-IV NSW	X	X	
CPF050B	PITTSBORO TOWN LAKE AT WATER INTAKE	35.71379	-79.18489	CHATHAM	WS-IV NSW	X	X	

NC DWR ALMP STATION INFORMATION								
Station Number	Location	Latitude	Longitude	County	Station Classification	ADDITIONAL SAMPLES BASED ON CLASSIFICATION		
						Chloride	Metals	Fecal coliform
CPF055C	JORDAN LAKE ABOVE STINKING CR NR PITTSBORO NC	35.69131	-79.07905	CHATHAM	WS-IV B NSW CA	X	X	
CPF055E	JORDAN LAKE ABOVE DAM NR MONCURE NC	35.65995	-79.07005	CHATHAM	WS-IV B NSW CA			
CPF081A1C	JORDAN LAKE AT MOUTH NEW HOPE CR	35.81622	-78.98683	CHATHAM	WS-IV B NSW CA			
CPF083B	MOUNTAIN ISLAND LAKE BELOW DUE POWER COMPANY	35.37023	-80.95589	GASTON	WS-IV B CA			
CPF086C	JORDAN LAKE AT MOUTH OF MORGAN CK NR FARRINGTON	35.82151	-78.99738	CHATHAM	WS-IV B NSW CA			
CPF086F	JORDAN LAKE NR FARRINGTON NC	35.79700	-79.01081	CHATHAM	WS-IV B NSW CA	X	X	
CPF087B	JORDAN LAKE AT FOLKNER CR NR FARRINGTON NC	35.78903	-79.02059	CHATHAM	WS-IV B NSW CA			
CPF087B3	JORDAN LAKE AT BOUY #9 NR MERRY OAKS NC	35.76522	-79.02596	CHATHAM	WS-IV B NSW CA	X	X	
CPF087D	JORDAN LAKE AT MOUTH OF White OAK CR NR SEAFORTH NC	35.73864	-79.02415	CHATHAM	WS-IV B NSW CA			
CPF08801A	JORDAN LAKE	35.73506	-79.02472	CHATHAM	WS-IV B NSW CA			
CPF0880A	JORDAN LAKE NR MOUTH OF BEAVER CR NR MERRY OAKS NC	35.69647	-79.04356	CHATHAM	WS-IV B NSW CA	X	X	
CPF0884A	JORDAN LAKE	35.68049	-79.06608	CHATHAM	WS-IV B NSW CA			
CPF089D3	OAK HOLLOW LAKE	36.02493	-79.99361	GUILFORD	WS-IV CA			

NC DWR ALMP STATION INFORMATION								
Station Number	Location	Latitude	Longitude	County	Station Classification	ADDITIONAL SAMPLES BASED ON CLASSIFICATION		
						Chloride	Metals	Fecal coliform
CPF089D4	OAK HOLLOW LAKE AT SR 1507 NR HIGH POINT	36.01472	-80.00284	GUILFORD	WS-IV CA			
CPF089D5	OAK HOLLOW LAKE AT DAM NR HIGH POINT	36.01236	-79.98828	GUILFORD	WS-IV CA	X	X	
CPF089E2	HIGH POINT LAKE DS OF SR 1507 NR HIGH POINT	36.00756	-79.94092	GUILFORD	WS-IV CA			
CPF089E4	HIGH POINT LAKE ABOVE DEEP RIVER	35.99615	-79.94499	GUILFORD	WS-IV CA	X	X	
CPFRD1	RANDLEMAN LAKE DNS SR 1129	35.93696	-79.88818	GUILFORD	WS-IV CA			
CPFRD2	RANDLEMAN LAKE UPS HWY 62	35.91943	-79.86259	GUILFORD	WS-IV CA			
CPFRD3	RANDLEMAN LAKE NEAR LEVEL CROSS	35.86287	-79.82857	RANDOLPH	WS-IV CA			
CPFRD4	RANDLEMAN LAKE AT WATER INTAKE	35.88540	-79.84209	RANDOLPH	WS-IV CA	X	X	
CPFRD5	RANDLEMAN LAKE IN MUDDY CREEK ARM NEAR GLENOLA	35.86624	-79.86446	RANDOLPH	WS-IV CA			
CPFRD6	RANDLEMAN LAKE IN MUDDY CREEK ARM BOB BRANCH	35.85663	-79.84458	RANDOLPH	WS-IV CA			
CPFRD7	RANDLEMAN LAKE DNS SR 1936	35.84207	-79.81259	RANDOLPH	WS-IV CA			
CPFRD8	RANDLEMAN LAKE NEAR DAM	35.83813	-79.81885	RANDOLPH	WS-IV CA			
CPFRD9	RANDLEMAN LAKE DNS SR 196	35.84557	-79.82942	RANDOLPH	WS-IV CA			
CPF113R	CARTHAGE CITY LAKE AT DAM NR CARTHAGE NC	35.33205	-79.40977	MOORE	WS-III CA	X	X	
CPF1201A	ROCKY RIVER RESERVOIR	35.79581	-79.47758	CHATHAM	WS-III CA	X	X	
CPF1201B	ROCKY RIVER RESERVOIR AT CTR NR CRUTFIELD XRD	35.80414	-79.48059	CHATHAM	WS-III CA			
CPF126A2	HARRIS LAKE NR SR 1914 NR DUNCAN NC	35.58975	-78.94163	CHATHAM	WS-V			
CPF126A4	HARRIS LAKE NR RR BRIDGE NR HOLLEMANS CROSSROAD	35.61148	-78.94592	CHATHAM	WS-V			
CPF126A6	HARRIS LAKE AT SR 1915 NR CORINTH NC	35.56857	-78.96669	CHATHAM	WS-V			

NC DWR ALMP STATION INFORMATION								
Station Number	Location	Latitude	Longitude	County	Station Classification	ADDITIONAL SAMPLES BASED ON CLASSIFICATION		
						Chloride	Metals	Fecal coliform
CPF135B	OLD TOWN RESERVOIR NR SOUTHERN PINES NC	35.21244	-79.40794	MOORE	WS-III			
CPF135D	OLD TOWN RESERVOIR AT DAM NR SOUTHERN PINES NC	35.21529	-79.40279	MOORE	WS-III	X	X	
CPF138A4	BONNIE DOONE LAKE AT DAM NR BONNIE DOONE NC	35.11013	-78.94388	CUMBERLAND	WS-IV B CA	X	X	X
CPF138A6	KORNBOW LAKE AT DAM NR BONNIE DOONE NC	35.10090	-78.92950	CUMBERLAND	WS-IV	X	X	
CPF138A8	MINTZ POND AT DAM NR BONNIE DOONE NC	35.08992	-78.92450	CUMBERLAND	WS-IV	X	X	
CPF138B	GLENVILLE LAKE AT DAM NR FAYETTEVILLE NC	35.06932	-78.89730	CUMBERLAND	WS-IV CA	X	X	X
CPF151	HOPE MILLS LAKE AT HOPE MILLS	34.97326	-78.94517	CUMBERLAND	B			X
CPF153C	SALTERS LAKE AT NORTH END NR RUSKIN	34.70810	-78.63200	BLADEN	C			
CPF153D	SALTERS LAKE AT SOUTH END NR YORICK	34.70130	-78.62838	BLADEN	C			
CPF1552A	JONES LAKE AT UPPER END NR YORICK NC	34.69092	-78.60418	BLADEN	B			
CPF1553A	JONES LAKE AT LOWER END NR YORICK NC	34.68458	-78.59977	BLADEN	B			
CPF155A	WHITE LAKE AT WHITE LAKE NC	34.63584	-78.49338	BLADEN	B			
CPF155B	WHITE LAKE NR WHITE LAKE NC	34.64346	-78.49799	BLADEN	B			
CPF155C	WHITE LAKE NR ELIZABETHTOWN NC	34.65102	-78.50244	BLADEN	B			
CPF155G	BLACK LAKE NR BEARD NC	34.67816	-78.41370	BLADEN	C Sw			
CPF155I	BLACK LAKE NR LAGOON	34.66139	-78.40190	BLADEN	C Sw			
CPF176D	SINGLETARY LAKE AT NORTH END	34.59748	-78.46718	BLADEN	B Sw			
CPF176E	SINGLETARY LAKE AT CENTER NR WHITE LAKE	34.59072	-78.46029	BLADEN	B Sw			
CPF176F	SINGLETARY LAKE ABOVE LAKE DRAIN	34.58920	-78.45132	BLADEN	B Sw			
CPF211B	GREENFIELD LAKE AT UPPER END AT WILMINGTON	34.20726	-77.93481	NEW HANOVER	C Sw			

NC DWR ALMP STATION INFORMATION								
Station Number	Location	Latitude	Longitude	County	Station Classification	ADDITIONAL SAMPLES BASED ON CLASSIFICATION		
						Chloride	Metals	Fecal coliform
CPF211C	GREENFIELD LAKE AT LOWER END AT WILMINGTON NC	34.21270	-77.94449	NEW HANOVER	C Sw			
CPFBSL2	BOILING SPRINGS LAKE IN UPS END	34.03012	-78.06004	BRUNSWICK	B Sw			
CPFBSL4	BOILING SPRINGS LAKE IN NORTH LAKE ARM	34.04191	-78.05124	BRUNSWICK	B Sw			
CPFBSL6	BOILINGS SPRINGS LAKE AT DAM	34.04709	-78.03865	BRUNSWICK	B Sw			
CPFCCR2	CANE CR RESERVOIR IN UPS END NR ORANGE GROVE NC	35.97000	-79.21254	ORANGE	WS-II HQW NSW CA			
CPFCCR4	CANE CR RESERVOIR UPS SR 1101 NR OAKS NC	35.95840	-79.22965	ORANGE	WS-II HQW NSW CA			
CPFCCR6	CANE CR RESERVOIR AT DAM NR OAKS NC	35.94955	-79.24155	ORANGE	WS-II HQW NSW CA	X	X	
CPFCL1	CABIN LAKE AT UPSTREAM END	34.99212	-77.79350	DUPLIN	B Sw			
CPFCL2	CABIN LAKE AT DOWNSTREAM OF UPPER END	34.98820	-77.79432	DUPLIN	B Sw			
CPFCL3	CABIN LAKE AT LOWER MIDDLE OF LAKE	34.98539	-77.79476	DUPLIN	B Sw			
CPFCL4	CABIN LAKE NEAR DAM	34.98149	-77.79618	DUPLIN	B Sw			
CPFGMR1	GRAHAM-MEBANE RESERVOIR IN QUAKER CR ARM	36.10637	-79.33003	ALAMANCE	WS-II HQW NSW CA			
CPFGMR2	GRAHAM-MEBANE RESERVOIR IN UPS OF BACK CR ARM	36.12732	-79.29782	ALAMANCE	WS-II HQW NSW CA			
CPFGMR3	GRAHAM-MEBANE RESERVOIR MIDLAKE IN BACK CR ARM	36.11557	-79.30870	ALAMANCE	WS-II HQW NSW CA			
CPFGMR4	GRAHAM-MEBANE RESERVOIR AT DAM NR HAW RIVER NC	36.09976	-79.32872	ALAMANCE	WS-II HQW NSW CA	X	X	
CPFGMROA	GRAHAM-MEBANE RESERVOIR AT UPS END NR MEBANE NC	36.12743	-79.31636	ALAMANCE	WS-II HQW NSW CA			

NC DWR ALMP STATION INFORMATION								
Station Number	Location	Latitude	Longitude	County	Station Classification	ADDITIONAL SAMPLES BASED ON CLASSIFICATION		
						Chloride	Metals	Fecal coliform
CPFLH2	LAKE HIGGINS UPS SR 2190 NR GREENSBORO NC	36.15707	-79.89234	GUILFORD	WS-III NSW CA			
CPFLH4	LAKE HIGGINS AT DAM NR GREENSBORO NC	36.16894	-79.88205	GUILFORD	WS-III NSW CA	X	X	
CPFLT4	LAKE TOWNSEND UPS SR 1001 NR GREENSBORO	36.17696	-79.79236	GUILFORD	WS-III NSW CA			
CPFLT6	LAKE TOWNSEND UPS SR 2523 NR GREENSBORO NC	36.17062	-79.77038	GUILFORD	WS-III NSW CA			
CPFLT8	LAKE TOWNSEND AT DAM NR GREENSBORO NC	36.18969	-79.73329	GUILFORD	WS-III NSW CA	X	X	
CPFSC1	SANDY CR RESERVOIR AT DAM NR RAMSEUR NC	35.74443	-79.6763	RANDOLPH	WS-III CA	X	X	
CPFSC2	SANDY CR RESERVOIR AT MID-LAKE NR RAMSEUR	35.75686	-79.67137	RANDOLPH	WS-III CA			
CPFSC3	SANDY CR RESERVOIR AT UPS END NR RAMSEUR NC	35.77335	-79.66412	RANDOLPH	WS-III CA			
CPFSCR2	STONY CR RESERVOIR AT US END NR CAROLINA NC	36.13954	-79.41158	ALAMANCE	WS-II HQW NSW CA			
CPFSCR4	STONY CR RESERVOIR AT DAM NR CAROLINA NC	36.12893	-79.40698	ALAMANCE	WS-II HQW NSW CA	X	X	
CPFUL4	UNIVERSITY LAKE IN NEVILLE CR ARM	35.89733	-79.10234	ORANGE	WS-II HQW NSW CA			
CPFUL6	UNIVERSITY LAKE AT DAM NR CHAPEL HILL NC	35.89647	-79.09322	ORANGE	WS-II HQW NSW CA	X	X	
CATAWBA RIVER BASIN								
CTB013B	LAKE JAMES A MARION NC	35.72159	-81.98075	BURKE	WS-V B			
CTB015A	LAKE JAMES NR NEBO NC	35.73940	-81.90547	BURKE	WS-V B			
CTB015C	LAKE JAMES NR BRIDGEWATER NC	35.74071	-81.85445	BURKE	WS-V B	X	X	

NC DWR ALMP STATION INFORMATION								
Station Number	Location	Latitude	Longitude	County	Station Classification	ADDITIONAL SAMPLES BASED ON CLASSIFICATION		
						Chloride	Metals	Fecal coliform
CTB023A1	LAKE JAMES AT LONGTOWN NC	35.78319	-81.86473	BURKE	WS-V B			
CTB023B	LAKE JAMES NR GLEN ALPINC NC	35.75956	-81.8463	BURKE	WS-V B			
CTB034A	LAKE RHODHISS AT SR 1501 NR DREXEL NC	35.78718	-81.62544	CALDWELL	WS-IV B CA			
CTB040A	LAKE RHODHISS AT SR 1001 NR BATON NC	35.78023	-81.52305	CALDWELL	WS-IV B CA			
CTB040B	LAKE RHODHISS NR RHODHISS NC	35.77346	-81.44087	CALDWELL	WS-IV B CA			
CTB048A	LAKE HICKORY AT US HWY 321 AT HICKORY NC	35.75924	-81.37576	CATAWBA	WS-IV B CA	X	X	
CTB056A	LAKE HICKORY AT NC HWY 127 NR HICORY NC	35.80263	-81.30454	CATAWBA	WS-IV, B CA			
CTB0581F	LOOKOUT SHOALS LAKE AT MOUTH OF ELK SHOALS CR	35.78549	-81.12322	CATAWBA	WS-IV, B CA			
CTB058C	LAKE HICKORY NR PROSPST STORE NC	35.80416	-81.26902	CATAWBA	WS-V B			
CTB058D	LAKE HICKORY NR MILLERSVILLE NC	35.81105	-81.22229	CATAWBA	WS-V B			
CTB058F	LOOKOUT SHOALS LAKE 1.5 MI US DAM	35.77577	-81.10846	CATAWBA	WS-IV, B CA			
CTB058G	LOOKOUT SHOALS LAKE AT DAM NR CATAWBA	35.75964	-81.09042	CATAWBA	WS-IV, B CA	X	X	
CTB079A	LAKE NORMAN AT SR 1004 NR MOORESVILLE NC	35.69504	-80.99123	CATAWBA	WS-IV B CA	X	X	
CTB082A	CORNELIUS CR ARM OF LAKE NORMAN	35.61659	-80.88543	IREDELL	WS-IV B CA	X	X	
CTB082AA	LAKE NORMAN AT HUNTERSVILLE WATER INTAKE	35.45420	-80.90200	MECKLENBURG	WS-IV B CA	X	X	
CTB082B	LAKE NORMAN AT DUKE POWER PINNACLE ACCESS	35.60555	-80.94380	CATAWBA	WS-IV B CA	X	X	
CTB082BB	LAKE NORMAN AT COWANS FORD DAM	35.43736	-80.95644	LINCOLN	WS-IV B CA	X	X	
CTB082M	LAKE NORMAN AT SR 1844 IN MOUNTAIN CR ARM.	35.56602	-80.99025	CATAWBA	WS-IV B CA	X	X	
CTB082Q	LAKE NORMAN AT DAVIDSON WATER INTAKE	35.50535	-80.91133	MECKLENBURG	WS-IV B CA	X	X	
CTB082R	LAKE NORMAN AT MOUTH OF REEDS CR ARM	35.48689	-80.94152	LINCOLN	WS-IV B CA	X	X	

NC DWR ALMP STATION INFORMATION								
Station Number	Location	Latitude	Longitude	County	Station Classification	ADDITIONAL SAMPLES BASED ON CLASSIFICATION		
						Chloride	Metals	Fecal coliform
CTB086A	MCDOWELLS CR AT MOUTH NR HUNTERSVILLE	35.37020	-80.94117	MECKLENBURG	WS-IV B CA			
CTB086B	MOUNTAIN ISLAND LAKE ABOVE GAR CR NR PAW CR	35.35426	-80.93773	GASTON	WS-IV B CA			
CTB086C	GAR CR AT MOUTH NR PAW CR	35.34915	-80.93388	MECKLENBURG	WS-IV B CA	X	X	
CTB087	MOUNTAIN ISLAND LAKE AT NC HWY 16 NR THRIFT	35.3497	-80.97296	GASTON	WS-IV B CA			
CTB087A	MOUNTAIN ISLAND LAKE ABOVE DAM NR MT HOLLY NC	35.33707	-80.98824	GASTON	WS-IV B CA	X	X	
CTB103	CATAWBA RIVER AT SOUTH BELMONT	35.21197	-81.00694	MECKLENBURG	WS-IV B CA			
CTB105B	LAKE WYLIE NR SHOPTON NC	35.16947	-81.00427	MECKLENBURG	WS-V B			
CTB174	SOUTH FORK CATAWBA RIVER AT SR 2524 NR SOUTH BELMONT	35.16690	-81.03843	GASTON	WS-V B			
CTB177	CATAWBA CR AT SR 2302 AT NC-SC STATE LINE	35.15150	-81.05839	MECKLENBURG	WS-V B			
CTB178	LAKE WYLIE AT NC HWY 49 NR OAK GROVE	35.10198	-81.03975	MECKLENBURG	WS-V B			
CTB198B5	LAKE WYLIE IN CROWDERS CR ARM NR CLOVER SC	35.11075	-81.08753	YORK	WS-V B			
CTB198C5	LAKE WYLIE IN ALLISON CR ARM NR CONCORD SC	35.04975	-81.09717	YORK	WS-V B			
CTB198D	LAKE WYLIE AT CATAWBA DAM	35.02338	-81.01361	YORK	WS-V B	X	X	
CTB1D2	ICARD DAM IMPDT NR DAM NR SR 1143	35.81734	-81.33934	ALEXANDER	WS-IV			
CTBBCL1	BESSEMER CITY LAKE NR SR 1404	35.29647	-81.29983	GASTON	WS-II HQW CA	X	X	
CTBLT1	LAKE TAHOMA NR CHESTNUT BRANCH NR NC HWY 80	35.72810	-82.08770	MCDOWELL	WS-II B Tr HQW			
CTBLT2	LAKE TAHOMA NR DAM NR NC HWY 80	35.72375	-82.08066	MCDOWELL	WS-II B Tr HQW	X	X	
CTBNCL1	NEWTON CITY LAKE AT DAM NR NEWTON NC	35.65547	-81.25089	CATAWBA	WS-III CA	X	X	
CTBO13C	LAKE JAMES AT MARION NC	35.74686	-81.95530	BURKE	WS-V B			

NC DWR ALMP STATION INFORMATION								
Station Number	Location	Latitude	Longitude	County	Station Classification	ADDITIONAL SAMPLES BASED ON CLASSIFICATION		
						Chloride	Metals	Fecal coliform
FRENCH BROAD RIVER BASIN								
FRB047A	LAKE JUNALUSKA AT MOUTH OF RICHLAND CR	35.52029	-82.97746	HAYWOOD	B			
FRB047B	LAKE JUNALUSKA AT CENTER NR LAKE JUNALUSKA NC	35.52712	-82.97267	HAYWOOD	B			
FRB047C	LAKE JUNALUSKA AT DAM AT LAKE JUNALUSKA NC	35.52705	-82.96460	HAYWOOD	B			
FRBACR2	ALLEN CR RESERVOIR AT UPS END NR HAZELWOOD NC	35.42011	-83.00950	HAYWOOD	WS-1 HQW			
FRBACR4	ALLEN CR RESERVOIR AT DAM NR HAZELWOOD NC	35.42369	-83.00939	HAYWOOD	WS-1 HQW	X	X	
FRBBTR1	BEETREE RESERVOIR AT DAM NR SWANNANOA NC	35.64261	-82.40092	BUNCOMBE	WS-1 HQW	X	X	
FRBBUR2	BURNETT RESERVOIR AT UPS END NR WALTERTOWN NC	35.67457	-82.33560	BUNCOMBE	WS-1 HQW			
FRBBUR4	BURNETT RESERVOIR	35.66398	-82.34333	BUNCOMBE	WS-1 HQW	X	X	
FRBLJ2	LAKE JULIAN DW HWY 280 NR SKYLAND NC	35.48291	-82.53300	BUNCOMBE	C			
FRBLJ4	LAKE JULIAND AT SOUTHERN RR BRIDGE NR SKLAND	35.47598	-82.53035	BUNCOMBE	C			
FRBLJ6	LAKE JULIAN NR DAM NR SKYLAND	35.47612	-82.54592	BUNCOMBE	C			
FRBWL2	WATERVILLE LAKE IN UPS END NR HEPCO NC	35.67285	-83.01352	HAYWOOD	C			
FRBWL4	WATERVILLE LAKE AT MOUTH OF WILKINS CR	35.68731	-83.03554	HAYWOOD	C			
FRBWL6	WATERVILLE LAKE IN CATALOOCHEE CR ARM	35.68831	-83.05593	HAYWOOD	C			
FRBWL8	WATERVILLE LAKE AT DAM NR HEPCO NC	35.69421	-83.04940	HAYWOOD	C			
HIWASSEE RIVER BASIN								
HIW000B	CHATUGE LAKE BELOW ARMSTRONG COVE	35.00298	-83.78905	CLAY	B			
HIW000D	CHATUGE LAKE AT SHOOTING CR ARM	35.02128	-83.76266	CLAY	B			
HIW000F	CHATUGE LAKE AT POWER INTAKE TOWER	35.01652	-83.79100	CLAY	B			

NC DWR ALMP STATION INFORMATION								
Station Number	Location	Latitude	Longitude	County	Station Classification	ADDITIONAL SAMPLES BASED ON CLASSIFICATION		
						Chloride	Metals	Fecal coliform
HIW009A	HIWASSEE LAKE	35.09751	-84.07491	CHEROKEE	B C			
HIW009B	HIWASSEE LAKE IN MOUTH NOTTLEY RIV	35.08500	-84.07078	CHEROKEE	B C			
HIW009D	HIWASSEE LAKE BELOW GRAPE CRK	35.11461	-84.12471	CHEROKEE	B C			
HIW009F	HIWASSEE LAKE BELOW BEARPAW CRK	35.12543	-84.16233	CHEROKEE	B C			
HIW009G	HIWASSEE LAKE BELOW CHAMBERS CRK	35.15712	-84.16280	CHEROKEE	B C			
HIW011A	APALACHIA LAKE AT ANDERSON CR	35.14776	-84.20301	CHEROKEE	B C			
HIW011C	APALACHIA LAKE BELOW NORTH SHOAL CK	35.17290	-84.24181	CHEROKEE	B C			
HIW012	APALACHIA LAKE AT DAM AT STATE LINE	35.16770	-84.29344	CHEROKEE	B C			
LUMBER RIVER BASIN								
LBR007A	LAKE RHODHISS	34.68983	-79.43228	CALDWELL	WS-IV B CA			
LBR017B	MAXTON POND AT CENTER AT MAXTON NC CLEAN LAKES	34.72106	-79.36742	ROBESON	C Sw			
LBR027D	PAGES LAKE	35.14080	-79.43249	MOORE	B			
LBR027E	PAGES LAKE	35.13560	-79.43001	MOORE	B			
LBR076A	LAKE WACCAMAW AT BIG CR AT LAKE WACCAMAW CLEAN LAKES	34.28864	-78.48204	COLUMBUS	B Sw ORW			
LBR076K	LAKE WACCAMAW AT CENTER NW END CLEAN LAKES	34.30516	-78.53097	COLUMBUS	B Sw ORW			
LBR076P	LAKE WACCAMAW AT CENTER LAKE WACCAMAW CLEAN LAKES	34.29078	-78.51171	COLUMBUS	B Sw ORW			
LBR091B	LAKE TABOR AT MID POINT	34.16367	-78.86069	COLUMBUS	B Sw			
LBR091C	LAKE TABOR AT LOWER END	34.16031	-78.85899	COLUMBUS	B Sw			
LITTLE TENNESSEE RIVER BASIN								

NC DWR ALMP STATION INFORMATION								
Station Number	Location	Latitude	Longitude	County	Station Classification	ADDITIONAL SAMPLES BASED ON CLASSIFICATION		
						Chloride	Metals	Fecal coliform
LTN006C	LAKE SEQUOYAH NR SR 106 HIGHLANDS	35.05723	-83.22141	MACON	WS-I B CA			
LTN008C	LAKE SEQUOYAH NR MIRROR LAKE NR HOLT KNOB	35.06226	-83.22065	MACON	WS-I B CA			
LTN008E	LAKE SEQUOYAH NR DAM NR US 64	35.06660	-83.22445	MACON	WS-I B CA	X	X	
LTN013B	NANTAHALA LAKE	35.16690	-83.66063	MACON	B Tr			
LTN013C	NANTAHALA LAKE	35.18084	-83.68317	MACON	B Tr			
LTN013D	NANTAHALA LAKE NR DAM NR AQUONE	35.19015	-83.65351	MACON	B Tr			
LTN013H	FONTANA LAKE BELOW HAZEL CR	35.44346	-83.77517	GRAHAM	WS-IV B CA			
LTN015A	WOLF CRK RES NR GRAYS RIDGE	35.23315	-82.99076	JACKSON	WS-III B Tr HQW			
LTN015A1	WOLF CRK RES NR DAM NR SR 281	35.22162	-82.99725	JACKSON	WS-III B Tr HQW			
LTN015B	BEAR CR LAKE	35.23302	-83.05041	JACKSON	WS-III B Tr			
LTN015D	BEAR CR LAKE	35.24142	-83.07095	JACKSON	WS-III B Tr	X	X	
LTN015F	CEDAR CLIFF LAKE NR SR 281 NR CEDAR CLIFF MTN	35.24260	-83.08141	JACKSON	WS-III B Tr			
LTN015H	CEDAR CLIFF LAKE NR DAM NR TUCKASEGEE	35.25400	-83.09900	JACKSON	WS-III B Tr			
LTN015L	THORPE RES NR HURRICANE CRK NR SR 107	35.16100	-83.12900	JACKSON	WS-III B Tr HQW			
LTN015N	THORPE RES NR GLENVILLE NR SR 107	35.17209	-83.14935	JACKSON	WS-III B Tr HQW			
LTN015P	THORPE RES NR MILL CRK NR SR 107	35.18596	-83.15407	JACKSON	WS-III B Tr HQW			
LTN015R	THORPE RES NR DAM NR SR 107	35.19541	-83.15704	JACKSON	WS-III B Tr HQW			

NC DWR ALMP STATION INFORMATION								
Station Number	Location	Latitude	Longitude	County	Station Classification	ADDITIONAL SAMPLES BASED ON CLASSIFICATION		
						Chloride	Metals	Fecal coliform
LTN031A	FONTANA LAKE AT TUCKASEGEE RIV AT BRYSON CITY	35.45304	-83.58301	SWAIN	B			
LTN031B	FONTANA LAKE BELOW TUCKASEGEE R	35.43513	-83.59081	GRAHAM	B			
LTN031D	FONTANA LAKE ABOVE STECOAH CRK	35.43838	-83.64445	GRAHAM	B			
LTN031J	FONTANA LAKE AT FONTANA DAM	35.45619	-83.80479	GRAHAM	WS-IV B CA			
LTN032B	LAKE CHEOAH AT NC 28 NR FONTANA VILLAGE	35.44720	-83.81704	SWAIN	C Tr			
LTN032D	LAKE CHEOAH AT POWER LINES NR POWER HOUSE	35.44822	-83.86594	SWAIN	C Tr			
LTN032F	LAKE CHEOAH NR DAM NR TAPOCO	35.44853	-83.93526	SWAIN	C Tr			
LTN037B	SANTEELAH LAKE 1MI UPS SNOWBIRD CK	35.34463	-83.83664	GRAHAM	B Tr			
LTN037D	SANTEELAH LAKE NR BROOKS GAP	35.35876	-83.86285	GRAHAM	B Tr			
LTN037E	SANTEELAH LAKE 100 YDS UPS DAM	35.37582	-83.87662	GRAHAM	B Tr			
LTN040	CALDERWOOD LAKE NR SLICKROCK CR	35.46339	-83.95694	DUPLIN	C Tr			
LTN041	CALDERWOOD LAKE NR DAM	35.49397	-83.97915	GRAHAM	C Tr			
NEUSE RIVER BASIN								
NEU0061G	LAKE MICHIE NR MOUTH OF DIAL CR NR BAHAMA NC	36.17124	-78.85848	DURHAM	WS-III NSW CA			
NEU0061J	LAKE MICHIE NR MOUTH OF DRY CR NR BAHAMA NC	36.16060	-78.8403	DURHAM	WS-III NSW CA			
NEU0061L	LAKE MICHIE NR SR 1622 NR BAHAMA NC	36.15808	-78.82624	DURHAM	WS-III NSW CA	X	X	
NEU006S	LITTLE RIVER RESERVOIR AT MOUNTAIN CR ARM	36.13487	-78.89028	DURHAM	WS-II NSW CA			

NC DWR ALMP STATION INFORMATION								
Station Number	Location	Latitude	Longitude	County	Station Classification	ADDITIONAL SAMPLES BASED ON CLASSIFICATION		
						Chloride	Metals	Fecal coliform
NEU006T	LITTLE RIVER RESERVOIR AT SR 1628 AT ORANGE FACTORY NC	36.12756	-78.87431	DURHAM	WS-II NSW CA			
NEU006U	LITTLE RIVER RESERVOIR NR DAM	36.11229	-78.87000	DURHAM	WS-II NSW CA	X	X	
NEU006X	LAKE MICHIE	36.19652	-78.77724	DURHAM	WS-III NSW CA			
NEU007	LAKE BUTNER NR SR 1122	36.18713	-78.76798	GRANVILLE	WS-II NSW CA			
NEU007B	LAKE BUTNER NR DAM	36.16902	-78.77220	GRANVILLE	WS-II NSW CA	X	X	
NEU00B	LAKE ORANGE IN WEST FORK	36.15452	-79.14825	ORANGE	WS-II NSW CA			
NEU00B4	LAKE ORANGE NR DAM NR KENNEDY	36.14805	-79.14975	ORANGE	WS-II NSW CA	X	X	
NEU00C	CORPORATION LAKE NR FAUCETTE MILL ROAD NR EFLAND NC	36.09320	-79.14268	ORANGE	WS-II NSW CA			
NEU00C1	CORPORATION LAKE NR DAM NR EFLAND NC	36.08487	-79.14157	ORANGE	WS-II NSW CA	X	X	
NEU00D	LAKE BEN JOHNSON AT DAM NR SR 1144	36.07111	-79.13089	ORANGE	WS-II NSW CA	X	X	
NEU010	FALLS LAKE AT SOUTHERN RR NR DURHAM NC	36.07757	-78.79304	GRANVILLE	WS-III NSW CA			
NEU013	FALLS LAKE AT I-85 NR NORTHSIDE NC	36.0702	-78.77947	DURHAM	WS-III NSW CA			
NEU013B	FALLS LAKE AT MARKER #16 NR REDWOOD NC	36.06685	-78.77337	DURHAM	WS-III NSW CA			

NC DWR ALMP STATION INFORMATION								
Station Number	Location	Latitude	Longitude	County	Station Classification	ADDITIONAL SAMPLES BASED ON CLASSIFICATION		
						Chloride	Metals	Fecal coliform
NEU0171B	FALLS LAKE AT MOUTH OF LITTLE LICK CR	36.01763	-78.73552	DURHAM	WS-III NSW CA			
NEU017A	LAKE ROGERS NR DAM NR CREEDMOOR NC	36.13084	-78.70383	GRANVILLE	WS-II NSW CA	X	X	
NEU018E	FALLS LAKE AT MOUTH OF LEDGE CR NR CREEDMOOR NC	36.01654	-78.70688	WAKE	WS-III NSW CA			
NEU019E	FALLS LAKE AT MOUTH OF BEAVERDAM CR NR MARKER #10	36.02241	-78.68501	WAKE	WS-III NSW CA			
NEU019L	FALLS LAKE AT CHANNEL MARKER #6 NR BAYLEAF NC	35.99963	-78.65168	WAKE	WS-III NSW CA			
NEU019P	FALLS LAKE AT NC HWY 98 NR BAYLEAF NC	35.97876	-78.63286	WAKE	WS-III NSW CA			
NEU020D	FALLS LAKE AT MARKER #1 NR BAYLEAF NC	35.95373	-78.58296	WAKE	WS-III NSW CA	X	X	
NEU035A7	REEDY CR LAKE NR DAM NR RALEIGH NC	35.83778	-78.74614	WAKE	B NSW			
NEU035G	BIG LAKE AT UPPER END AT UMSTEAD PARK	35.87865	-78.76930	WAKE	B NSW			X
NEU035H	BIG LAKE AT LOWER END AT UMSTEAD PARK	35.87258	-78.76596	WAKE	B NSW			X
NEU035J	SYCAMORE LAKE NR DAM NR RALEIGH NC	35.86331	-78.75272	WAKE	B NSW			X
NEU042C	LAKE JOHNSON AT UPPER END AT RALEIGH	35.76449	-78.72161	WAKE	B NSW			X
NEU055A	APEX RESERVOIR NR DAM NR APEX NC	35.74766	-78.81722	WAKE	WS-III NSW	X	X	
NEU055A01	LAKE WHEELER AT SR 1379 NR GARNER NC	35.70003	-78.72110	WAKE	WS-III NSW			
NEU055A02	LAKE WHEELER AT DAM NR GARNER NC	35.69227	-78.69625	WAKE	WS-III NSW	X	X	
NEU055A3	LAKE BENSON AT UPPER END NR GARNER NC	35.67192	-78.64280	WAKE	WS-III NSW CA			

NC DWR ALMP STATION INFORMATION								
Station Number	Location	Latitude	Longitude	County	Station Classification	ADDITIONAL SAMPLES BASED ON CLASSIFICATION		
						Chloride	Metals	Fecal coliform
NEU055A4	LAKE BENSON AT LOWER END NR GARNER NC	35.66568	-78.62462	WAKE	WS-III NSW CA	X	X	
NEU057C	BASS LAKE NR HOLLY SPRINGS NC	35.63805	-78.80725	WAKE	B NSW			X
NEU057C1	BASS LAKE NR HOLLY SPRINGS	35.64178	-78.80384	WAKE	B NSW			X
NEU067C	WENDELL LAKE AT UPPER END	35.73706	-78.36160	JOHNSTON	C NSW			
NEU067E	WENDELL LAKE AT DAM NR SR 1716	35.72816	-78.36025	JOHNSTON	C NSW			
NEU07113A	CLIFFS OF THE NEUSE LAKE NR SEVEN SPRINGS NC	35.23522	-77.88683	WAYNE	B NSW			X
NEU084B	BUCKHORN RESERVOIR UPS SR 2112 NR WILKERSON CROSSROADS	35.70726	-78.14524	WILSON	WSV NSW			
NEU084C	BUCKHORN RESERVOIR NR DAM NR WILKERSON CROSSROADS	35.69205	-78.12214	WILSON	WSV NSW	X	X	
NEU084D	WIGGINS MILL RESERVOIR AT CENTER NR WIGGINS MILL	35.69537	-77.95376	WILSON	WS-III NSW CA			
NEU084F	WIGGINS MILL RESERVOUR US DAM NR WIGGINS MILL NC	35.68848	-77.94912	WILSON	WS-III NSW CA	X	X	
NEU096B4	LAKE WILSON NR DAM NR DUNN COROSSROADS NC	35.78973	-77.92093	WILSON	WS-III NSW			
NEU096C	TOISNOT RESERVOIR NR SR 1326 NR WILSON NC	35.74896	-77.90525	WILSON	WS-III NSW CA			
NEU096E	TOISNOT RESERVOIR NR DAM NR NC 58	35.74476	-77.90246	WILSON	WS-III NSW CA	X	X	
NEU133D	LONG LAKE	34.88761	-76.99696	PAMLICO	C Sw NSW			
NEU133E	LONG LAKE AT WEST END NR HAVELOCK NC	34.88983	-77.00958	PAMLICO	C Sw NSW			
NEU133H	ELLIS LAKE AT LAKE CENTER	34.85035	-77.00184	PAMLICO	C Sw NSW			
NEUCL1	LAKE CRABTREE IN UPS END NR MORRISVILLE NC	35.82480	-78.81714	WAKE	B NSW			X

NC DWR ALMP STATION INFORMATION								
Station Number	Location	Latitude	Longitude	County	Station Classification	ADDITIONAL SAMPLES BASED ON CLASSIFICATION		
						Chloride	Metals	Fecal coliform
NEUCL2	LAKE CRABTREE UPS SR 3015 NR MORRISVILLE NC	35.84368	-78.80477	WAKE	B NSW			X
NEUCL3	LAKE CRABTREE AT DAM NR MORRISVILLE NC	35.83826	-78.78216	WAKE	B NSW			X
NEUOOB2	LAKE ORANGE IN EAST FORK	36.15690	-79.14272	ORANGE	WS-II NSW CA			
NEW RIVER BASIN								
NEW006E	ASU LAKE AT DAM NR BOONE NC	36.23912	-81.67036	WATAUGA	WS-II Tr HQW CA	X	X	
PASQUOTANK RIVER BASIN								
PAS0122A	LAKE MATTAMUSKEET NR LAKE LANDING NC	35.53137	-76.11081	HYDE	SC			
PAS0123A	LAKE MATTAMAUSKEET NR LAKE COMFORT	35.50182	-76.18211	HYDE	SC			
PAS0124A	LAKE MATTAMUSKEET NR FAIRFIELD NC	35.51052	-76.27309	HYDE	SC			
PAS012B	LAKE PHELPS AT WEST END NR ROPER NC	35.77056	-76.50348	WASHINGTON	B Sw ORW			
PAS012C	LAKE PHELPS AT CENTER NR CRESWELL NC	35.77071	-76.45490	WASHINGTON	B Sw ORW			
PAS012D	LAKE PHELPS AT NE END NR CRESWELL NC	35.76960	-76.41964	WASHINGTON	B Sw ORW			
ROANOKE RIVER BASIN								
ROA003A	HANGING ROCK STATE PARK LAKE CLEAN LAKES	36.39075	-80.26885	STOKES	B			X
ROA0092A	KERNERSVILLE LAKE	36.15220	-80.10203	FORSYTH	WS-IV CA	X	X	
ROA009E	BELEWS LAKE AT ROCKINGHAM ACCESS AREA CLEAN LAKE	36.29239	-80.03655	ROCKINGHAM	WS-IV B			
ROA009G	BELEWS LAKE AT MOUTH OF ASH BASIN NR WALNUT CVE	36.29270	-80.04899	ROCKINGHAM	WS-IV B			
ROA009H	BELEWS LAKE AT PINE HALL ACCESS AREA	36.31873	-80.02798	ROCKINGHAM	WS-IV B			

NC DWR ALMP STATION INFORMATION								
Station Number	Location	Latitude	Longitude	County	Station Classification	ADDITIONAL SAMPLES BASED ON CLASSIFICATION		
						Chloride	Metals	Fecal coliform
ROA009J	BELEWS LAKE AT MOUTH E BELEWS CRK NR PINE HALL CLN LK	36.26218	-80.04889	BUNCOMBE	WS-IV B			
ROA027G	FARMER LAKE AT UPS END NR YANCEYVILLE	36.36790	-79.39229	CASWELL	WS-II HQW CA			
ROA027J	FARMER LAKE UPS SR 1121 NR YANCEYVILLE	36.37869	-79.37827	CASWELL	WS-II HQW CA			
ROA027L	FARMER LAKE NR DAM NR YANCEYVILLE	36.38634	-79.36263	CASWELL	WS-II HQW CA	X	X	
ROA030C	HYCO LAKE AT MOUTH OF HYCO CRK NR CONCORD	36.48068	-79.09935	PERSON	WS-V B			
ROA030DA	LAKE ROXBORO NR SR 1716 NR RIDGEVILLE	36.31360	-79.15010	CASWELL	WS-II B HQW			
ROA030DC	LAKE ROXBORO NR HESTERS STORE	36.32827	-79.15484	CASWELL	WS-II B HQW			
ROA030DE	LAKE ROXBORO NR DAM NR FROGSBORO	36.34569	-79.15423	CASWELL	WS-II B HQW	X	X	
ROA030E	HYCO LAKE DNS NC 57 NR CONCORD	36.47004	-79.09317	PERSON	WS-V B			
ROA030F	HYCO LAKE AT POWER PLANT NR CEFF0	36.49593	-79.07193	PERSON	WS-V B			
ROA030G	HYCO LAKE AT MAIN DAM NR MCGHEES MILL	36.50500	-79.04700	PERSON	WS-V B			
ROA031C	LAKE ISAAC WALTON NR STORYS CRK NR HICKS VILLAGE	36.42289	-79.01519	PERSON	WS-II HQW CA	X	X	
ROA031E	LAKE ISAAC WALTON NR LICK BRANCH CRK NR OLIVE BR	36.42709	-79.01826	PERSON	WS-II HQW CA			
ROA031H	LAKE ISAAC WALTON NR DAM NR FIVE FORKS	36.43212	-79.01681	PERSON	WS-II HQW CA			
ROA0341A	MAYO LAKE	36.45734	-78.87787	PERSON	WS-V			
ROA0342A	MAYO LAKE	36.49677	-78.88218	PERSON	WS-V			
ROA0343A	MAYO LAKE AT DAM NR BETHEL HILL	36.53436	-78.87532	PERSON	WS-V			

NC DWR ALMP STATION INFORMATION								
Station Number	Location	Latitude	Longitude	County	Station Classification	ADDITIONAL SAMPLES BASED ON CLASSIFICATION		
						Chloride	Metals	Fecal coliform
ROA037A	KERR LAKE	36.40960	-78.39199	VANCE	B C			
ROA037E	KERR LAKE AT BUOY M NR HENDERSON	36.44154	-78.37742	VANCE	B C			
ROA037I	KERR LAKE AT BUOY I NR HENDERSON	36.49209	-78.36079	VANCE	B C			
ROA037IJ	KERR LAKE	36.53141	-78.33724	VANCE	B C			
ROA0382A	LAKE GASTON NR BRACEY VA	36.58816	-78.16428	MECKLENBURG	WS-V B	X	X	
ROA038A	LAKE GASTON NR FIVE FORKS	36.53791	-78.02660	WARREN	WS-V B	X	X	
ROA039	LAKE GASTON NR ELAMS	36.51834	-77.96241	WARREN	WS-V B	X	X	
ROA039B	LAKE GASTON NR SUMMIT	36.50321	-77.83114	WARREN	WS-IV B	X	X	
ROA039C	ROANOKE RAPIDS LAKE	36.48538	-77.76334	NORTHAMPTON	WS-IV B CA	X	X	
ROA039D	ROANOKE RAPIDS LAKE	36.48163	-77.72738	NORTHAMPTON	WS-IV B CA	X	X	
ROA039E	ROANOKE RAPIDS LAKE	36.48232	-77.68777	NORTHAMPTON	WS-IV B CA	X	X	
SAVANNAH RIVER BASIN								
SAV001C	CASHIERS LAKE AT US END NR CASHIERS NC	35.10786	-83.10028	JACKSON	B Tr ORW			
SAV001D	CASHIERS LAKE AT DAM NR CASHIERS	35.10552	-83.10099	JACKSON	B Tr ORW			
SAV002N	LAKE TOXAWAY AT US END NR LAKE TOXAWAY NC	35.14223	-82.95223	TRANSYLVANIA	B Tr			
SAV002P	LAKE TOXAWAY AT CENTER NR TOXAWAY FALLS NC	35.13612	-82.94306	TRANSYLVANIA	B Tr			
SAV002R	LAKE TOXAWAY IN DEEP FORD ARM NR TOXAWAY FALLS	35.12369	-82.94387	TRANSYLVANIA	B Tr			
SAV002T	LAKE TOXAWAY AT DAM NR TOXAWAY FALLS NC	35.12545	-82.93371	TRANSYLVANIA	B Tr			
TAR-PAMLICO RIVER BASIN								
TAR001C	LAKE DEVIN	36.30556	-78.63029	GRANVILLE	WS-II NSW CA			

NC DWR ALMP STATION INFORMATION								
Station Number	Location	Latitude	Longitude	County	Station Classification	ADDITIONAL SAMPLES BASED ON CLASSIFICATION		
						Chloride	Metals	Fecal coliform
TAR001E	LAKE DEVIN	36.30112	-78.62166	GRANVILLE	WS-II NSW CA	X	X	
TAR015E	TAR RIVER RESERVOIR UPS SR 1603 NR WINSTEAD XRDS	35.85415	-77.90785	NASH	WS-IV B NSW CA			
TAR015G	TAR RIVER RESERVOIR UPS SR 1745 NR LANGLEY XRDS	35.88317	-77.89375	NASH	WS-IV B NSW CA			
TAR017C	TAR RIVER RESERVOIR UPS SR 1603 NR LANGLEY XRDS	35.88607	-77.91351	NASH	WS-IV B NSW CA			
TAR017F	TAR RIVER RESERVOIR AT DAM NR LANGLEY XRDS	35.89827	-77.88572	NASH	WS-IV B NSW CA	X	X	
WHITE OAK RIVER BASIN								
WOK026D	CATFISH NR CROATAN NC	34.93023	-77.09430	CRAVEN	C			
WOK026E	CATFISH LAKE	34.93271	-77.11126	JONES	C			
WOK026G	GREAT LAKE AT UPPER END NR LONG LAKE	34.87475	-77.04118	CRAVEN	C			
WOK026H	GREAT LAKE	34.86336	-77.03702	CRAVEN	C			
YADKIN-PEE DEE RIVER BASIN								
YAD007A	W KERR SCOTT LAKE DNS STONY CRK	36.11644	-81.28644	WILKES	WS-IV B Tr			
YAD008	W KERR SCOTT LAKE NR WILKESBORO	36.12102	-81.26505	WILKES	WS-IV B Tr			
YAD008A	W KERR SCOTT LAKE UPS DAM	36.13004	-81.23406	WILKES	WS-IV B Tr			
YAD068E	LASATER LAKE AT US END NR CLEMMONS NC	36.02859	-80.41114	FORSYTH	WS-IV			
YAD068F	LASATER LAKE AT DAM NR CLEMMONS NC	36.02621	-80.41931	FORSYTH	WS-IV	X	X	
YAD077A	SALEM LAKE NR UPPER END	36.10192	-80.17016	FORSYTH	WS-III CA			
YAD077B1	SALEM LAKE IN LOWREY CRK ARM UPS I40	36.10600	-80.18500	FORSYTH	WS-III CA			

NC DWR ALMP STATION INFORMATION								
Station Number	Location	Latitude	Longitude	County	Station Classification	ADDITIONAL SAMPLES BASED ON CLASSIFICATION		
						Chloride	Metals	Fecal coliform
YAD077C	SALEM LAKE AT DAM I	36.09500	-80.19100	FORSYTH	WS-III CA	X	X	
YAD077D	WINSTON LAKE AT WINSTON SALEM	36.1145	-80.20179	FORSYTH	C			
YAD121R	LAKE WRIGHT AT DAM NR FIVE POINTS	35.58566	-80.63455	ROWAN	WS-II HQW CA	X	X	
YAD122B	LAKE CORRIHER AT US END NR LANDIS	35.56318	-80.61549	ROWAN	WS-IV CA			
YAD122D	LAKE CORRIHER AT DAM NR CHINA GROVE	35.56476	-80.60947	ROWAN	WS-IV CA	X	X	
YAD1391A	HIGH ROCK LAKE	35.72026	-80.33843	ROWAN	WS-V		X	
YAD152A	HIGH ROCK LAKE	35.65436	-80.30535	ROWAN	WS-IV B		X	
YAD152C	HIGH ROCK LAKE NR ROCKWELL CLEAN LAKES	35.64600	-80.29700	ROWAN	WS-IV B		X	
YAD156A	HIGH ROCK LAKE	35.62666	-80.29236	ROWAN	WS-IV B		X	
YAD160B	LAKE THOM A LEX AT BRUSHY FORK CLEAN LAKES	35.89400	-80.18100	DAVIDSON	WS-III CA			
YAD1611A	LAKE THOM A LEX AT LOWER END CLEAN LAKES	35.87709	-80.19241	DAVIDSON	WS-III CA	X	X	
YAD169A	HIGH ROCK LAKE	35.64477	-80.25404	DAVIDSON	WS-V, B		X	
YAD169B	HIGH ROCK LAKE UPS PANTHER CRK CLEAN LAKES	35.62476	-80.25859	ROWAN	WS-IV B		X	
YAD169E	HIGH ROCK LAKE AT MTH FLAT SWP CRK CLEAN LAKES	35.61133	-80.23454	DAVIDSON	WS-IV B CA			
YAD169F	HIGH ROCK LAKE NR HIGH ROCK CLEAN LAKES	35.60595	-80.23656	ROWAN	WS-IV B CA	X	X	
YAD172C	TUCKERTOWN LAKE AT BALD MTN	35.54603	-80.20063	ROWAN	WS-IV B CA			
YAD1780A	TUCKERTOWN LAKE ABOVE DAM	35.49371	-80.17868	ROWAN	WS-IV B CA	X	X	
YAD178B	BADIN LAKE DNS GARR CRK NR ISENHOUR	35.46480	-80.12444	MONTGOMERY	WS-IV B CA			
YAD178E	BADIN LAKE DNS BEAVERDAM CRK NR UWHARRIE IN 82072	35.46994	-80.08749	MONTGOMERY	WS-IV B CA			

NC DWR ALMP STATION INFORMATION								
Station Number	Location	Latitude	Longitude	County	Station Classification	ADDITIONAL SAMPLES BASED ON CLASSIFICATION		
						Chloride	Metals	Fecal coliform
YAD178F	BADIN LAKE UPS DAM NR PALMERVILLE	35.43345	-80.09692	MONTGOMERY	WS-IV B CA			
YAD178F1	BADIN LAKE AT BADIN	35.41713	-80.10964	MONTGOMERY	WS-IV B CA	X	X	
YAD178F3	FALLS LAKE AT UPPER END NR BADIN NC	35.40619	-80.09371	STANLY	WS-IV B			X
YAD178F5	FALLS LAKE AT LOWER END NR BADIN NC	35.39498	-80.08270	STANLY	WS-IV B	X	X	X
YAD179B	LAKE REESE AT UPS END NR MOTLETA	35.72303	-79.97787	RANDOLPH	WS-III CA			
YAD179D	LAKE REESE AT MIDLAKE NR MOTLETA	35.70337	-79.97257	RANDOLPH	WS-III CA			
YAD179F	LAKE REESE AT DAM NR FARMER	35.68174	-79.97021	RANDOLPH	WS-III CA	X	X	
YAD181E	MCCRARY LAKE AT DAM NR ASHEBORO	35.71553	-79.85769	RANDOLPH	WS-II HQW CA	X	X	
YAD181G	BUNCH LAKE AT DAM NR ASHEBORO	35.72133	-79.86015	RANDOLPH	WS-II HQW CA	X	X	
YAD181J	BACK CR LAKE	35.74267	-79.85848	RANDOLPH	WS-II HQW CA			
YAD181K	BACK CRK LAKE NR MID LAKE NR ASHEBORO	35.73752	-79.86916	RANDOLPH	WS-II HQW CA			
YAD181L	BACK CRK LAKE AT DAM NR ASHEBORO	35.73638	-79.87770	RANDOLPH	WS-II HQW CA	X	X	
YAD185A	LAKE TILLERY	35.35734	-80.06599	STANLY	WS-IV B CA			
YAD189	LAKE TILLERY	35.30480	-80.08218	STANLY	WS-IV B CA			
YAD189B	LAKE TILLERY	35.24924	-80.09588	STANLY	WS-IV B CA			
YAD189C	LAKE TILLERY 2 MI UPS DAM NR NORWOOD	35.23013	-80.08655	STANLY	WS-IV B CA	X	X	
YAD207A	KANNAPOLIS LAKE UPS SR 1104 NR ENOCHVILLE NC	35.52405	-80.64314	ROWAN	WS-III CA			
YAD207C	KANNAPOLIS LAKE NR DAM NR KANNAPOLIS NC	35.51333	-80.6481	ROWAN	WS-III CA	X	X	

NC DWR ALMP STATION INFORMATION								
Station Number	Location	Latitude	Longitude	County	Station Classification	ADDITIONAL SAMPLES BASED ON CLASSIFICATION		
						Chloride	Metals	Fecal coliform
YAD215R	LAKE FISHER UPS SR 1308 NR KANNAPOLIS NC	35.50995	-80.57005	CABARRUS	WS-IV CA			
YAD215T	LAKE FISHER BETWEEN SR 2180 AND SR 2000 NR KANNAPOLIS NC	35.49691	-80.57372	CABARRUS	WS-IV CA			
YAD216A	LAKE FISHER AT DAM	35.48678	-80.57834	CABARRUS	WS-IV CA	X	X	
YAD216C	LAKE CONCORD IN NORTH FORK NR HEILMANS MILL NC	35.48347	-80.58906	CABARRUS	WS-IV CA			
YAD216E	LKE CONCORD IN NW FORK NR KANNAPOLIS NC	35.48135	-80.59224	CABARRUS	WS-IV CA			
YAD216G	LAKE CONCORD AT DAM NR HEILMANS MILL NC	35.47836	-80.58595	CABARRUS	WS-IV CA	X	X	
YAD225C	LONG LAKE AT UPPER END NR ALBEMARLE NC	35.35688	-80.23549	STANLY	C			
YAD232C	LAKE LEE AT SR 2102 NR MONROE NC	34.96045	-80.52288	UNION	WS-IV CA			
YAD232D	LAKE MONROE NR MONROE NC	34.93071	80.52652	UNION	WS-IV CA			
YAD232F	LAKE MONROE AT DAM NR MONROE NC	34.94097	-80.51864	UNION	WS-IV CA	X	X	
YAD232H	LAKE LEE IN BUCK CR ARM NR MONROE NC	34.95435	-80.51150	UNION	WS-IV CA			
YAD233	LAKE LEE AT DAM NR MONROE NC	34.96516	-80.51134	UNION	WS-IV CA	X	X	
YAD235D	LAKE STEWART IN CHINKAPIN CR ARM NR MONROE NC	35.04075	-80.48511	UNION	WS-III CA			
YAD235F	LAKE STEWART MID LAKE NR FOWLER CROSSROADS NC	35.02791	-80.49260	UNION	WS-III CA			
YAD236	LAKE STEWART AT DAM NR FOWLER CROSSROADS NC	35.03569	-80.47899	UNION	WS-III CA	X	X	
YADCCR01	CODDLE CREEK RESERVOIR (LAKE HOWELL) NEAR DAM	35.43935	-80.70387	CABARRUS	WS-II HQW	X	X	
YADCCR02	CODDLE CREEK RESERVOIR (LAKE HOWELL) IN CENTER	35.45709	-80.71238	CABBARUS	WS-II HQW			

NC DWR ALMP STATION INFORMATION								
Station Number	Location	Latitude	Longitude	County	Station Classification	ADDITIONAL SAMPLES BASED ON CLASSIFICATION		
						Chloride	Metals	Fecal coliform
YADCCR03	CODDLE CREEK RESERVOIR (LAKE HOWELL) IN UPSTREAM	35.47635	-80.71384	CABBARUS	WS-II HQW			
YAD255D	LONG LAKE AT LOWER END	35.35067	-80.22764	STANLY	C			
YAD260B	BLEWETT FALLS LAKE UPS DAM 0.5MI	34.99181	-79.88764	RICHMOND	WS-IV B CA	X	X	
YAD262E	ROBERDEL LAKE UPS SR 1436 NR ROBERDEL NC	34.97488	-79.73866	RICHMOND	WS-III CA			
YAD263	ROBERDEL LAKE AT DAM NR ROBERDEL NC	34.97136	-79.74368	RICHMOND	WS-III CA	X	X	
YAD265C	ROCKINGHAM CITY LAKE AT DAM NR ROCKINGHAM NC	34.93779	-79.73935	RICHMOND	WS-III CA	X	X	
YAD275H	WADESBORO CITY POND AT DAM	34.91878	-80.08819	ANSON	WS-II HQW CA			
YAD275J	WADESBORO CITY POND AT US END	34.92360	-80.08135	ANSON	WS-II HQW CA	X	X	
YAD280C	WATER LAKE AT UPS END NR MOUTH OF MARKS CR	34.90597	-79.66269	RICHMOND	WS-II HQW CA			
YAD280E	WATER LAKE AT DAM NR HAMLET NC	34.89993	-79.66991	RICHMOND	WS-II HQW CA	X	X	
YAD282A	CITY LAKE AT UPPER END AT HAMLET NC	34.88604	-79.68908	RICHMOND	C			
YAD283	CITY LAKE AT HAMLET NC	34.88406	-79.69107	RICHMOND	C			

ATTACHMENT 5:

NCDENR/DWR CHEMISTRY LABORATORY DATA QUALIFIER CODES

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Symbol	Definition
A	Value reported is the mean (average) of two or more determinations. This code is to be used if the results of two or more discrete and separate samples are averaged. These samples shall have been processed and analyzed independently (e.g., field duplicates, different dilutions of the same sample). This code is not required for BOD or coliform reporting since averaging multiple dilutions for these parameters is fundamental to those methods.
B	Results based upon colony counts outside the acceptable range and should be used with caution. This code applies to microbiological tests and specifically to **membrane filter (MF)** colony counts. It is to be used if less than 100% sample was analyzed and the colony count is generated from a plate in which the number of colonies exceeds the ideal ranges indicated by the method. These ideal ranges are defined in the method as: *Fecal coliform or Enterococcus bacteria: 20-60 colonies* *Total coliform bacteria: 20-80 colonies* 1. Countable membranes with less than 20 colonies. Reported value is estimated or is a total of the counts on all filters reported per 100 ml. 2. Counts from all filters were zero. The value reported is based on the number of colonies per 100 ml that would have been reported if there had been one colony on the filter representing the largest filtration volume (reported as a less than "<" value). 3. Countable membranes with more than 60 or 80 colonies. The value reported is calculated using the count from the smallest volume filtered and reported as a greater than ">" value. 4. Filters have counts of both >60 or 80 and <20. Reported value is estimated or is a total of the counts on all filters reported per 100 ml. 5. Too many colonies were present; too numerous to count (TNTC). TNTC is generally defined as >150 colonies. The numeric value represents the maximum number of counts typically accepted on a filter membrane (60 for fecal or enterococcus and 80 for total), multiplied by 100 and then divided by the smallest filtration volume analyzed. This number is reported as a greater than value. 6. Estimated Value. Blank contamination evident. 7. Many non-coliform or non-enterococcus colonies or interfering non-coliform or non-enterococcus growth present. In this competitive situation, the reported value may under-represent actual density. Note: A "B" value shall be accompanied by justification for its use denoted by the numbers listed above (e.g., B1, B2, etc.). Note: A "J2" should be used for spiking failures.
BB	This code applies to **most probable number (MPN)** microbiological tests. 1. No wells or tubes gave a positive reaction. Value based upon the appropriate MPN Index and reported as a less than "<" value. 2. All wells or tubes gave positive reactions. Value based upon the MPN Index and reported as a greater than ">" value. Note: A "BB" value shall be accompanied by justification for its use denoted by the numbers listed above (e.g., BB1, BB2, etc.).
C	Total residual chlorine was present in sample upon receipt in the laboratory; value is **estimated**. Generally applies to cyanide, phenol, NH3, TKN, coliform, and organics.
G	A single quality control failure occurred during biochemical oxygen demand (BOD) analysis. The sample results should be used with caution. 1. The dissolved oxygen (DO) depletion of the dilution water blank exceeded 0.2 mg/L. 2. The bacterial seed controls did not meet the requirement of a DO depletion of at least 2.0 mg/L and/or a DO residual of at least 1.0 mg/L. 3. No sample dilution met the requirement of a DO depletion of at least 2.0 mg/L and/or a DO residual of at least 1.0 mg/L. 4. Evidence of toxicity was present. This is generally characterized by a significant increase in the BOD value as the sample concentration decreases. The reported value is calculated from the highest dilution representing the maximum loading potential and should be considered an **estimated** value. 5. The glucose/ glutamic acid standard exceeded the range of 198 ± 30.5 mg/L. 6. The calculated seed correction exceeded the range of 0.6 to 1.0 mg/L. 7. Less than 1 mg/L DO remained for all dilutions set. The reported value is an **estimated** greater than value and is calculated for the dilution using the least amount of sample. 8. Oxygen usage is less than 2 mg/L for all dilutions set. The reported value is an **estimated** less than value and is calculated for the dilution using the most amount of sample. 9. The DO depletion of the dilution water blank produced a negative value. Note: A "G" value shall be accompanied by justification for its use denoted by the numbers listed above (e.g., G1, G2, etc.).
J	**Estimated** value; value may not be accurate. This code is to be used in the following instances: 1. Surrogate recovery limits have been exceeded. 2. The reported value failed to meet the established quality control criteria for either precision or accuracy. 3. The sample matrix interfered with the ability to make any accurate determination. 4. The data is questionable because of improper laboratory or field protocols (e.g., composite sample was collected instead of grab, plastic instead of glass container, etc.). 5. Temperature limits exceeded (samples frozen or >6°C) during transport or not verifiable (e.g., no temperature blank provided): non-reportable for NPDES compliance monitoring.

J	6. The laboratory analysis was from an unpreserved or improperly chemically preserved sample. The data may not be accurate. 7. This qualifier is used to identify analyte concentration exceeding the upper calibration range of the analytical instrument/method. The reported value should be considered estimated. 8. Temperature limits exceeded (samples frozen or >6°C) during storage, the data may not be accurate. 9. The reported value is determined by a **one-point estimation** rather than against a regression equation. The estimated concentration is less than the laboratory practical quantitation limit and greater than the laboratory method detection limit. 10. Unidentified peak; estimated value. 11. The reported value is determined by a **one-point estimation** rather than against a regression equation. The estimated concentration is less than the laboratory practical quantitation limit and greater than the instrument noise level. *This code is used when an MDL has not been established for the analyte in question.* 12. The calibration verification did not meet the calibration acceptance criterion for **field parameters**. Note: A "J" value shall be accompanied by justification for its use denoted by the numbers listed above (e.g., J1, J2, etc.). A "J" value shall not be used if another code applies (e.g., N, V, M).
M	Sample and duplicate results are "out of control". The sample is non-homogenous (e.g., VOA soil). The reported value is the lower value of duplicate analyses of a sample.
N	Presumptive evidence of presence of material; **estimated** value. This code is to be used if: 1. The component has been tentatively identified based on mass spectral library search. 2. There is an indication that the analyte is present, but quality control requirements for confirmation were not met (i.e., presence of analyte was not confirmed by alternate procedures). 3. This code shall be used if the level is too low to permit accurate quantification, but the **estimated** concentration is less than the laboratory practical quantitation limit and greater than the laboratory method detection limit. *This code is not routinely used for most analyses.* 4. This code shall be used if the level is too low to permit accurate quantification, but the **estimated** concentration is less than the laboratory practical quantitation limit and greater than the instrument noise level. *This code is used when an MDL has not been established for the analyte in question.* 5. The component has been tentatively identified based on a retention time standard.
Q	Holding time exceeded. These codes shall be used if the value is derived from a sample that was received, prepared and/or analyzed after the approved holding time restrictions for sample preparation and analysis. The value does not meet NPDES requirements. 1. Holding time exceeded prior to receipt by lab. 2. Holding time exceeded following receipt by lab.
P	Elevated PQL* due to matrix interference and/or sample dilution.
S	Not enough sample provided to prepare and/or analyze a method-required matrix spike (MS) and/or matrix spike duplicate (MSD).
U	Indicates that the analyte was analyzed for but not detected above the reported practical quantitation limit*. The number value reported with the "U" qualifier is equal to the laboratory's practical quantitation limit*.
X	Sample not analyzed for this constituent. This code is to be used if: 1. Sample not screened for this compound. 2. Sampled, but analysis lost or not performed-field error. 3. Sampled, but analysis lost or not performed-lab error. Note: an "X" value shall be accompanied by justification for its use by the numbers listed.
V	Indicates the analyte was detected in both the sample and the associated method blank. Note: The value in the blank shall not be subtracted from the associated samples.
Y	Elevated PQL* due to insufficient sample size.
Z	The sample analysis/results are not reported due to: 1. Inability to analyze the sample. 2. Questions concerning data reliability. The presence or absence of the analyte cannot be verified.
***PQL**	The Practical Quantitation Limit (PQL) is defined and proposed as "the lowest level achievable among laboratories within specified limits during routine laboratory operation". The PQL is about three to five times the calculated Method Detection Limit (MDL) and represents a practical and routinely achievable detection limit with a relatively good certainty that any reported value is reliable".
3/10/2011	

QAPP Annual Staff Review Confirmation

QA Document Reviewed: NC DWR Ambient Lakes Monitoring Program (ALMP) QAPP, Version 2.0 (EPA

Program(s): Ambient Lakes Monitoring Program (ALMP) **Supervisor: Jason Green**

Calendar/Sampling Year(s): 2014

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