

Nashoba Analytical, LLC
M-MA1118

Quality Assurance/Quality Control Manual
2022

Table of Contents

| | |
|--|----|
| Table of Contents | 2 |
| Revisions | 3 |
| Introduction | 4 |
| Organization Chart | 5 |
| Personnel | 6 |
| Laboratory Ethics | 11 |
| Data Quality Objectives | 13 |
| Analytical Procedures | 14 |
| Laboratory Equipment-Instrumentation-Calibration-Maintenance | 20 |
| Calibration | 20 |
| Maintenance | 20 |
| QC Checks and Frequency | 25 |
| Instrument Performance Check | 25 |
| Quality Control Sample | 25 |
| Reagent Blanks | 25 |
| Matrix Spikes | 25 |
| Duplicate Analysis | 25 |
| Initial Demonstration of Capability | 26 |
| Method Detection Limits | 26 |
| Low Level Capability | 26 |
| Interlaboratory QC/Proficiency Testing Samples | 26 |
| QC Control Charts | 27 |
| Trend Analysis/Corrective Action | 27 |
| QA Reporting Procedures | 28 |
| Data Reduction, Validation, Reporting and Maintenance | 29 |
| Significant Figures | 29 |
| Rounding Off | 29 |
| Units | 29 |
| Raw Data | 29 |
| Data Validation | 29 |
| Final Data/Reports | 29 |
| Data Corrections | 30 |
| Record Maintenance | 30 |
| Glassware | 31 |
| Chemicals/Reagents | 31 |
| Safety Plan | 32 |
| Hazardous Samples/Waste Disposal | 34 |
| Appendix i-Preservation-Hold Times-Bottles | 35 |
| Appendix ii-Instrument Maintenance Schedule | 39 |
| Appendix iii - Water Sampling Instructions | 41 |
| Appendix iiiii – Safety Training Check List | 42 |

Revisions

| <u>Revision Number</u> | <u>Date</u> | <u>Page</u> | <u>Description</u> |
|------------------------|-------------|--|--|
| 00 | 8/06 | All | Initial Publication |
| 01 | 10/06 | All | Response to deviations |
| 02 | 11/06 | 10,12,21 Appendix iii Appendix iiiii | Response to on-site inspection |
| 03 | 4/07 | Appendix i | Preservation & Hold Times from method update rule |
| 04 | 4/08 | None | Annual Review |
| 05 | 4/09 | Appendix ii | Instrument Maintenance Annual Review |
| 06 | 10/10 | None | Annual review |
| 07 | 05/11 | None | Annual Review |
| 08 | 05/12 | None | Annual Review |
| 09 | 04/13 | 5 | Personnel Annual Review |
| 10 | 04/14 | 5 | Personnel Annual Review |
| 11 | 04/15 | None | Annual Review |
| 12 | 04/16 | None | Annual Review |
| 13 | 03/17 | 5,9 | Annual Review, Personnel Change, Added Ethics |
| 14 | 06/2017 | 10,14,18,28 | Response to deviations |
| 15 | 11/18 | 5,9 | Personnel Update |
| 16 | 4/19 | 5,8,9 | Annual Review, Personnel |
| 17 | 4/20 | None | Annual Review |
| 18 | 4/21 | 5 | Annual Review, Personnel |
| 19 | 9/21 | 5,8,29 | Personnel, Reporting |
| 20 | 11/21 | 5,8 | Personnel |
| 21 | 02/22 | 5,8 | Personnel |

Introduction

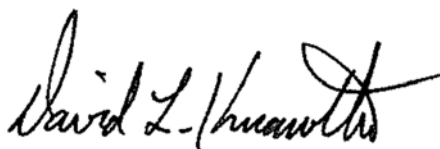
Nashoba Analytical, LLC is a privately-owned laboratory dedicated to the production of water quality data on chemical and microbiological parameters of public health significance. This includes but is not limited to the analysis of ground waters and surface waters for the purpose of compliance monitoring for public water supply systems, general potability testing for private individuals, monitoring of industrial effluents and storm water run off, and recreational waters to include bathing beaches and swimming pools. Some samples will be to investigate the occurrence, distribution, and trends of indicator analytes or organisms in surface and ground waters and relate these to environmental and water-quality factors.

Methodologies employed include those published in Standard Methods for the Examination of Water and Wastewater, 22nd Edition, methods published by the Environmental Protection Agency as well as methods published in the Code of Federal Regulations. We offer multiple method choices so that clients may choose the one that best fits their needs or application.

Quality-assurance and quality-control (QA/QC) practices for the operation of Nashoba Analytical, LLC are described in this manual. The Laboratory Director and staff are responsible for implementing and thoroughly executing all QA/QC procedures. This process begins at sample log-in with appropriate chain of custody documents, sample log in and tracking, and report generation. Paramount to this effort is the commitment of management and staff to correctly follow all SOP's and analytical quality-control procedures.

This manual will be reviewed at least annually, and any changes incorporated at that time.

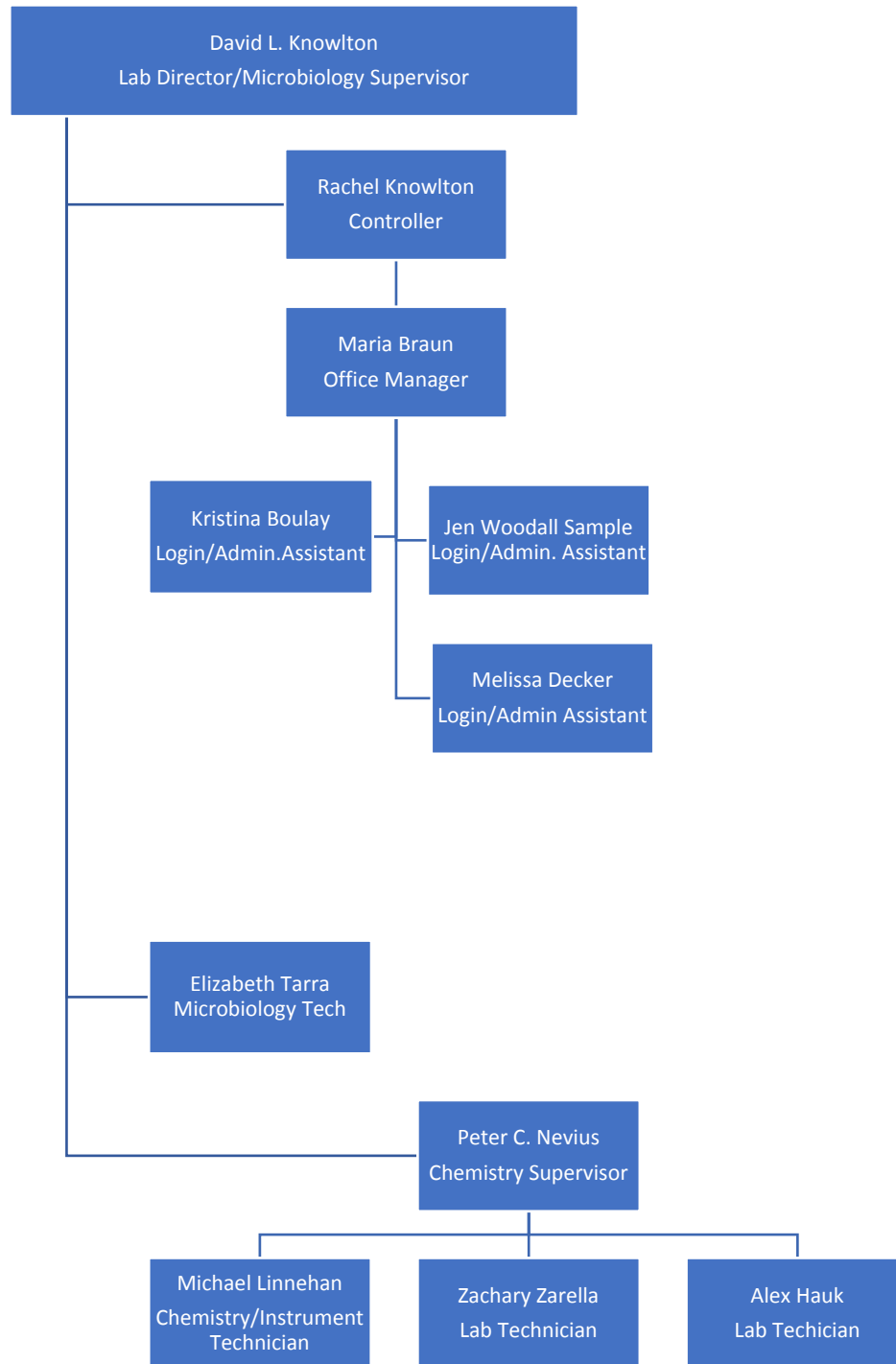
This manual has been reviewed and approved by David L. Knowlton, Lab Director and Manager, Nashoba Analytical, LLC



2/22/2022

Signature/Date

Organization Chart



Personnel

David L. Knowlton, Lab Director

-Graduate of Iowa State University, Ames, IA with a BS in Bacteriology and a minor in Chemistry, 1980.

-March 2006-Present

Lab Director/Supervisor and owner of Nashoba Analytical, LLC.

Experienced in the operation of all equipment and methodologies employed by the laboratory. Responsible for all aspects of the daily operation of the laboratory including health and safety, all QA/QC functions, hiring, training and review of employees, sample analysis and report generation.

-Sep 1990 - Oct 2005

Started at Thorstensen Laboratory as Microbiologist. This position developed as the company grew and additional employees were brought on, leading into the Laboratory Supervisor position. Responsibilities at Thorstensen Laboratory included:

- Overseeing all microbiological laboratory analyses
- Performing organic, inorganic, and wet chemistry analyses
- Training of laboratory technicians
- QA/QC inspections, writing of SOP's
- Scheduling of maintenance, and general operating performance

-From 1986-1990-Microbiologist for the Gillette Company, Andover MA
Responsibilities included microbiological analysis of personal care products for the quality control department. In addition, performed analytical testing on raw materials and finished products. Served as laboratory supervisor for one year covering a three shift operation with ten technicians.

Peter C. Nevius-Chemistry Supervisor

Graduate of University of Massachusetts, Amherst, MA with a BS in Environmental Science. 1996

Hire Date: July 24, 2006

Responsibilities at Nashoba Analytical, LLC will include analysis of samples for chemical parameters per established SOP's. This will include data validation and associated responsibilities as necessary to comply with this QA/QC manual and also any

state or federal regulations. Will perform associated laboratory duties as assigned by the lab director which will include maintenance of QA/QC data records and control charts.

May 1998-July 2006 Thorstensen Laboratory, Inc. Westford MA

Held position as Chemist performing analyses on potable and non-potable waters, and soil samples utilizing wet chemistry methods, IC, ICP, GFAA, FTIR, HPLC, and GC methods. Performed microbiological testing which included Total Coliform, HPC, and Fecal Coliform analysis. Responsible for maintaining QA/QC data relative to all parameters analyzed at the laboratory.

July 1996-April 1998

Held position as Biologist/Chemist at AquaTox Laboratory, Summerville SC. Performed whole effluent toxicity studies, BOD, solids, and associated waste water analyses. Responsible for field sampling and analysis. Maintained QA/QC data in keeping with state certification requirements.

June 1994-Jan 1996

T.R. Wilbury Laboratories, Marblehead MA

Laboratory Technician/Biologist

Performed toxicity studies, culture maintenance and general laboratory duties.

Rachel Knowlton, Controller

Aug 2007- Present

Responsibilities include all financial aspects of the business, including AR/AP and HR

Graduate of Bentley College, Waltham, MA with B.S. in Accounting, 1986.

Feb 2003 to August 2007- Financial Controller, Conquest Business Media, Beverly, MA.

Michael Linnehan - Chemistry Technician

BS Physics. Concentration in Environmental Science-UMass-Amherst- Sep 2009

July 2013 to present

Responsibilities at Nashoba Analytical, LLC will include analysis of samples for chemical parameters per established SOP's. This will include data validation and associated responsibilities as necessary to comply with this QA/QC manual and also any state or federal regulations. Will perform associated laboratory duties as assigned by the chemistry supervisor which will include maintenance of QA/QC data records and control charts.

Mathnasium, North Beverly MA-Math Instructor- Jan 2013-Jul 2013

Sam Ash Music, Tampa FL, Sales Associate Jun 2012-Oct 2012

UMass Boston, T.A. Environmental Science and Geology Jan 2010-May 2012

Maria Braun – Office Manager

Oct 2013 - present

Responsibilities will include clerical/reception duties and sample login per established SOP. Telephone coverage, generating reports and invoices, general filing, mailings, and other duties as assigned by the Controller.

June 2002 – Oct 2013 - Office Manager- Microbac Laboratories-Worcester MA

Oct 2000 – Jun 2002 – Staffing Consultant, John Leonard Employment Services, Inc.

Zachary Zarella-Lab Technician

June 2021-Present

B.S. Environmental Science, Fitchburg State University May 2021

Responsibilities at Nashoba Analytical, LLC will include analysis of samples for chemical parameters per established SOP's. This will include data validation and associated responsibilities as necessary to comply with this QA/QC manual and any state or federal regulations. Will perform associated laboratory duties as assigned by the chemistry supervisor which will include maintenance of QA/QC data records and control charts.

Alex Hauk-Lab Technician

January 3, 2022 - Present

University of New Haven: Fall 2018-December 2020 graduation TBD-Forensic Science

Responsibilities at Nashoba Analytical, LLC will include analysis of samples for chemical parameters per established SOP's. This will include data validation and associated responsibilities as necessary to comply with this QA/QC manual and any state or federal regulations. Will perform associated laboratory duties as assigned by the chemistry supervisor which will include maintenance of QA/QC data records and control charts.

Elizabeth Tarra-Microbiology Technician

Starts 11/29/21

Worcester State University
B.S. Biology, Minor Chemistry

March 2021-Present
Astellas Institute for Regenerative Medicine, Westborough MA
QC Microbiologist II

Sep 2018-Mar 2021
Bristol Meyers Squibb, Devens MA
QC Microbiologist

Dec 2017-Aug 2018
AbbVie Inc, Worcester MA
Contract Biologist

Responsibilities include analysis of microbiology samples per established SOP's, including data validation and associated responsibilities as necessary to comply with this QA/QC manual and also any state or federal regulations. Performs associated laboratory duties as assigned by the Microbiology Supervisor.

Jennifer Woodall-Sample Login/Admin Assistant

March 2018-Present
Responsibilities include sample log-in according to established acceptance criteria, create order, label bottles and accept payment if applicable. Provide telephone coverage, receive calls and assist clients with report questions. Maintain sample bottle inventory. Perform data entry and monitor subcontract shipments.

May 2014-Mar 2018: US Packing, Leominster MA Admin Assistant the Project Mgr.
Jan 2014-Mar 2014: Marathon Staffing- Temp Receptionist
Oct 2010-Aug 2013: Luvak Inc. Boylston MA, Log-In Administrator

Kristina Boulay-Sample Login/Admin Assistant

Feb 2021-Present

Responsibilities include sample log-in according to established acceptance criteria, create order, label bottles and accept payment if applicable. Provide telephone coverage, receive calls and assist clients with report questions. Generate reports and email to clients.

Apr 2019-May 2020

Sales/Office Assistant Trelleborg Pipe Seals-Milford NH

May 2015-Jan 2018

Host- The Sea Grill Restaurant-NY,NY

Melissa Decker--Sample Login/Admin Assistant

Jan 3, 2022-Present

Responsibilities include sample log-in according to established acceptance criteria, create order, label bottles and accept payment if applicable. Provide telephone coverage, receive calls and assist clients with report questions. Generate reports and email to clients

2020-2021 Nanny/Caregiver

2008-2020 Inventory Manager Adventix, LLC

2005-2008 Copy Technician/Manager OPL Inc Fulfillment

Laboratory Ethics

INTRODUCTION

A majority of the data produced at Nashoba Analytical, LLC has the potential for coming under the scrutiny of the legal system. It is imperative that the activities of the laboratory are of the highest ethical standards.

All employees will be required to participate in annual ethics training and review. The annual program must include procedures to ensure data integrity, recognition and prevention of improper laboratory practices, the promotion of objectivity and impartiality in the generation and reporting of data and procedures for confidential reporting of data integrity concerns to a supervisor or lab director. The training for each employee is to be documented and kept on file noting the date and content of review.

To assure that all data reported is reliable, accurate, and defensible in a court of law, all Standard Operating Procedures and contract specific requirements will be complied with in all circumstances.

All employees of Nashoba Analytical, LLC are considered ethical upon hiring and the checking of references and backgrounds.

It is the responsibility of Nashoba Analytical, LLC to protect employees from undue external and internal stresses that may lead to compromises in ethical conduct.

It is the responsibility of the laboratory personnel to avoid incidences where the impartiality and/or integrity of the laboratory and the services provided may come into question.

LABORATORY PROCEDURES

It is the policy of Nashoba Analytical, LLC that all personnel follow the written SOPs and the specific requirements of all contracts for which sample analyses are performed.

The Laboratory Director must approve any deviations from contractual requirements and/or SOPs that may be required to complete a specific analysis. Permission to deviate must be obtained from the appropriate contract authorities and documented.

RESPONSIBILITIES

It is the responsibility of the Laboratory Director to communicate contract requirements to personnel, provide extra training if required, and to ensure that all SOPs have been followed.

The Laboratory Director will perform unannounced audits to assure that the requirements of each project are being followed.

If the ability of the analyst to provide impartial data is compromised by a conflict of interest, it is the responsibility of the analyst to inform the Laboratory Director immediately. The Laboratory Director must then take appropriate measures to remove the conflict.

UNETHICAL CONDUCT

Actions defined as unethical include but are not limited to the following:

- Forging another's name or initials.
- Reporting results without actually analyzing the sample.
- Signing for review of data when no review was made.
- Altering data without justification.
- Spiking a "little extra" to improve recoveries.
- Manufacturing or distributing controlled substances on laboratory property.
- Fabrication or falsification of records or data

NOTIFICATION

Laboratory personnel who are aware or suspicious of any unethical practices or misconduct occurring in the lab shall inform the Laboratory Director.

No employee shall be discriminated against or disciplined for making an allegation in good faith or for providing evidence of possible unethical behavior.

The Laboratory Director shall investigate any allegation of wrongdoing in a confidential manner and take disciplinary action where necessary.

DISCIPLINARY ACTION

Any employee who participates in unethical conduct is subject to disciplinary action which may include termination.

REQUIREMENTS

All employees will be required to read this SOP and sign acknowledgement thereof. The signed form will be kept in the employees personnel file.

Data Quality Objectives

Data quality objectives are used to develop data collection designs and to establish specific criteria for the quality of the data to be collected. The process helps planners identify decision-making points for data collection activities, to determine the decisions to be made based on the data collected, and to identify the criteria to be used for making each decision.

The process should consider the following:

- Stating the issue. What is the reason for the analysis? Is it for compliance or regulatory purposes? A clear statement of the reason for the analysis is integral to establishing appropriate data quality objectives.
- Identify possible decisions and actions based upon the data.
- Identifying inputs and study limits.
- Developing a decision rule, i.e. a parameter of interest and action limits.

By having the data objectives defined beforehand, the client can be assured that the resulting analyses will meet the requirements of the study.

Analytical Procedures

The following is a list of analytical procedures for which certification will be attained. SOP's are maintained in a separate manual in the lab. All references to Standard Methods are from the 22nd Edition.

Potable Water

| Analyte | Method |
|------------------------|--------------|
| pH | SM 4500 H-B |
| Aluminum | EPA 200.7 |
| Arsenic | EPA 200.9 |
| Alkalinity | SM 2320B |
| Barium | EPA 200.7 |
| Beryllium | EPA 200.7 |
| Cadmium | EPA 200.7 |
| Chromium | EPA 200.7 |
| Calcium | EPA 200.7 |
| Chloride | EPA 300.0 |
| Copper | EPA 200.7 |
| Fluoride | EPA 300.0 |
| Iron | EPA 200.7 |
| Lead | EPA 200.9 |
| Manganese | EPA 200.7 |
| Nickel | EPA 200.7 |
| Nitrate-N | EPA 300.0 |
| Nitrite-N | EPA 300.0 |
| Silver | EPA 200.7 |
| Sodium | EPA 200.7 |
| Sulfate | EPA 300.0 |
| Thallium | EPA 200.9 |
| Total Dissolved Solids | SM 2540C |
| Turbidity | EPA 180.1 |
| Residual Chlorine | SM 4500 Cl G |
| Total Hardness | SM 2340 B |
| Zinc | EPA 200.7 |

Non-Potable Water

| | |
|----------|-----------|
| Aluminum | EPA 200.7 |
| Antimony | EPA 200.7 |
| Arsenic | EPA 200.7 |
| Cadmium | EPA 200.7 |
| Chromium | EPA 200.7 |
| Copper | EPA 200.7 |

| | |
|------------------------------|---------------|
| Iron | EPA 200.7 |
| Lead | EPA 200.7 |
| Manganese | EPA 200.7 |
| Molybdenum | EPA 200.7 |
| Nickel | EPA 200.7 |
| Selenium | EPA 200.7 |
| Strontium | EPA 200.7 |
| Thallium | EPA 200.7 |
| Zinc | EPA 200.7 |
| pH | SM4500-H-B |
| Non-Filterable Residue (TSS) | SM 2540 D |
| Calcium | EPA 200.7 |
| Magnesium | EPA 200.7 |
| Sodium | EPA 200.7 |
| Potassium | EPA 200.7 |
| Total Hardness | SM 2340 B |
| Biochemical Oxygen Demand | SM 5210 B |
| Chemical Oxygen Demand | HACH 8000 |
| Ammonia-N | SM 4500 NH3 D |
| Nitrate-N | EPA 300.0 |
| Ortho-Phosphate as P | EPA 300.0 |
| Ortho-Phosphate as P | SM 4500 P E |
| Total Phosphorus as P | SM 4500 P E |
| Total Phosphorus as P | EPA 200.7 |
| Residual Chlorine | SM 4500 Cl G |
| Alkalinity | SM 2320 B |
| Chloride | EPA 300.0 |
| Conductivity | SM 2510 B |
| Sulfate | EPA 300.0 |
| Total Dissolved Solids | SM 2540 C |
| Total Kjeldahl Nitrogen | HACH 10242 |

SOP's Available

Metals-ICP-EPA 200.7
Metals-GFAA-EPA200.9
HACH Titralab AT1000
EPA 300.0
Hardness by Calc. SM2340B
Total Diss. Solids SM2540C
Turbidity EPA 180.1
Chlorine(T&F)SM4500-Cl-G
Total Sus.Solids-SM2540
BOD(5)-SM5210B
Ammonia-SM4500-NH3-D
Phosphorus-SM4500-P-E
pH-SM4500 H-B
Alkalinity-SM2320B
Conductivity-SM2510B
COD-HACH 8000
TKN-HACH 10242

Sampling-Sample Receipt and Handling

Sample containers will be provided by the laboratory and will be either glass or plastic depending on the analysis required. Sample bottles are disposable and are single use only. Bottles will be labeled showing the preservative added (if any), sample number, samplers name, date and time of collection and sample location. Reference Table IV-7 from the EPA manual in the appendix at the back of this manual for bottle requirements, preservatives and hold times. Samples must be analyzed within hold times as specified in the methods.

A chain of custody is to be filled out by whoever is performing the sampling. The form should include as a minimum: sample number, sample description, date and time of sample collection, specific location of sample, sample type (grab or composite), bottle type and preservative and analysis to be performed from that bottle, date and time received at laboratory, name and signature of sampler and name and signatures of all persons involved in the custody of the sample at any point clearly indicating the date and time of change of possession. Any sample sent by or received by a courier or delivery service must be accompanied by a completed chain of custody.

When the sample is received at the laboratory the person receiving and logging in the sample must first determine that proper sampling procedures have been followed and that the identification of the samples matches the chain of custody. This includes checking for proper sample size and containers, and preservation used. The chart in the appendix may be referenced for this information. Sample pH should be checked to verify proper preservation using pH paper or pH meter and temperature checked if iced or refrigerated. This information should be recorded on the accompanying chain of custody.

Each sample will be assigned a unique identification number which will be written on the bottle with permanent marker or by use of a pre-glued label. The numbers are sequential and if multiple samples arrive for one location, they are assigned the root identification number followed by -1, -2, -3 etc. For example, waste water samples for raw water and final effluent might be 1102-1 and 1102-2 respectively. Sample information will be logged into the database and a computer-generated form printed for laboratory tracking, recording of data and for billing purposes. The chain of custody will be affixed to this sheet and will become part of the permanent record for that particular sample.

Sample hold times can be tracked in the database which will show date sampled and expiration date by analyte. The database query will be run daily to check sample status. The data sheet accompanying the sample through the lab can also be used to track status and will also be used as an internal chain of custody. The box in the lower left corner of the tracking sheet will show location of the sample, date and time of any change, and analyst. The final entry will be sample disposal.

Chain of custody samples are placed in a secure refrigerator after log-in if not analyzed immediately. The temperature is maintained at 4 deg C +/- 2. Completed samples are kept on storage racks and discarded after thirty days. Samples are not to be stored with

reagents or chemicals which may cause cross contamination nor with any highly contaminated samples which are stored in a separate refrigerator labeled “Quarantine”.

Sampling Procedures

Laboratory personnel will not be performing sampling for compliance purposes but must ensure that proper sampling procedures have been followed. Sample containers, preservatives, and hold times are outlined in Appendix i. Written sampling procedures are outlined in Appendix iii.

Grab Samples:

When possible, the sample should be grabbed with the bottle from which the analysis will be performed. This is especially important with oil & grease, as this analyte will adhere to the walls of the container and may be lost if transferred to another container. Use glass bottles for sampling oil & grease and ensure they are acidified to pH<2 with HCl. Do not split oil & grease samples, take two samples if needed. If samples other than Oil and Grease are to be split, be certain the sample is well mixed before doing so.

For organic analyses, especially volatiles, minimize sample exposure to air and agitation to prevent loss of components. Avoid sampling at turbulent locations, which may drive off components. Avoid sampling at weirs which favor retrieval of lighter than water immiscible components. Bottles should be filled so that there is ½” of headspace to facilitate proper mixing at the lab, unless the analytical method specifically calls for zero head space (i.e volatiles).

Composite Samples:

Composite sampling intervals may be specified by a regulatory agency or may be set as required by the client or type of sample being taken. The sampler should be placed as close to the sample source as possible and be located higher than the sample source with the intake tubing sloping downward from the sampler to the source. The tubing should only be long enough to reach from the sampler to the source and must be free of kinks or loops. Samplers should be capable of being programmed to sample for a given amount of time at a chosen frequency. It is important that the receiving vessel inside the sampler, and the tubing used, be properly cleaned and dried in between samplings to help safeguard against any cross contamination. After sampling is completed, the collected sample must be well mixed before distributing into sample containers. Thoroughly clean all samplers, composite containers and any tubing immediately after each use. Bottles should be filled so that there is ½” of headspace to facilitate proper mixing at the lab, unless the analytical method specifically calls for zero head space (i.e volatiles).

Sample Size:

The chart in “Appendix i” at the back of this manual list analytes, bottle requirements, preservatives, and hold times. Ensure that enough sample is collected to fulfill the

analytical and QC requirements of the methods (dups, spikes etc.) and that enough remains to use as a retained library sample for 30 days after completion of analysis.

Rejection of Samples:

Samples may be rejected for any of the following reasons:

Inappropriate sample container or preservative. Holding time exceeded. Proper sampling procedures or handling not followed. Person sampling not trained properly. Sample collection report form or chain of custody not filled out.

Examples warranting rejection include leaking bottles, head space or bubbles in VOC vials, using the wrong bottle (i.e. plastic bottle for volatiles or oil and grease), no refrigeration or ice used when it may be necessary.

The client must be notified immediately by telephone and in writing that the sample has been rejected and that re-sampling is necessary.

Laboratory Equipment-Instrumentation-Calibration-Maintenance

Calibration

Calibration will be performed by constructing a standard curve for each analyte using a minimum of three standards and a blank each time the instrument or method is used. Unless specified in the method, the correlation coefficient of a linear regression calibration curve should be 0.995 or greater per SM1020B. If less than this value, troubleshoot and recalibrate. Results are only to be reported if they fall between the lowest and highest calibration points or are within established linear dynamic ranges. If above the high calibration point or LDR dilute the sample into the working range. Do not report results below the lowest standard. Specific calibration procedures are outlined in the SOP's for each analyte or method.

Maintenance

Preventive maintenance is performed on each piece of laboratory equipment according to the schedule in "appendix ii". The specific procedures follow.

Balances

The balances will be cleaned and calibrated by an outside firm once per year. The top loader balance will have a deflection test performed monthly by placing a 150gm load on the pan, adding a 100mg load, and recording the results in the log book. This will show that the balance has a sensitivity of 0.1gm at a 150gm load. The analyst should ensure that the balance is cleaned after each use and that spilled media or chemical is not allowed to accumulate on surfaces. The analytical balance is calibrated daily using the internal calibration weight. A calibration check is then performed with three different ASTM Class I weights encompassing the typical working range (100mg, 10gm, 100gm). The readings are recorded in the balance log book. Any failure of the balance will require repair by an outside firm. The ASTM reference weights are to be calibrated every five years by an outside firm.

Thermometers

Thermometer calibration will be checked semiannually for microbiology thermometers and annually for all others against a NIST traceable thermometer which is calibrated every five years. All thermometers will be recorded in the log book showing the test thermometer I.D., the NIST thermometer I.D., the temperature readings of each, and the correction factor, if any, to be applied. A sticker will be placed on each thermometer or the liquid filled container in which it is placed showing the correction factor and date of calibration. The digital readout thermometer of the water bath will also be calibrated and a sticker affixed to the water bath. Thermometers used in incubators and water baths shall be graduated in 0.1 degree increments. The temperatures of ovens, water baths,

refrigerators and incubators are to be recorded for each day in use in a permanently bound log book.

pH Meter

The pH meter is part of the HACH TitrLab and shall be calibrated each day the meter is to be used with the results recorded in the log book. The calibration is performed using a buffer range of 4, 7, and 10 followed by reading a second source 7.0 buffer and recording the result and slope in the log book. The meter is equipped with a temperature compensating probe. Commercial buffers are purchased and are to be labeled with date received and date opened. Fresh aliquots are to be used for each calibration. Sample temperatures are recorded with pH results.

Maintain electrodes according to manufacturers instructions. Do not store in distilled water. Store in buffer 7.0 with saturated KCl. Breather hole should be covered during storage and removed during use. Make sure that the internal filling solution is maintained higher than the reference junction. Inspect the electrodes daily for scratches, cracks, salt crystal build-up, or membrane/junction deposits. Rinse off any salt build up with distilled water and remove any membrane/junction deposits as directed in the following cleaning procedure.

- General Cleaning: Soak in 0.1 M HCl or 0.1 M HNO₃ for half an hour.
- Inorganic: Soak in 0.1 M tetrasodium EDTA solution for 15 minutes.
- Grease and Oil: Rinse with mild detergent or methanol solution.

After cleaning, soak the electrode in storage solution for at least one hour.

Spectrophotometer

In use is a HACH DR3900 Spectrophotometer. The instrument and sample cells should be kept clean at all times and spills should be wiped up promptly. The photocell window can be cleaned with lens tissue or soft lint free cloth that will not leave a residue. Sample cells should be cleaned with detergent, rinsed several times with tap water and finally with deionized water. The wavelength is calibrated annually at 880nm using the calibration device supplied by HACH and following the procedure as outlined in the instrument manual. It may also be checked at 577nm by placing a white object into the light path and observing the color. The color should be yellow. If not, a new bulb and calibration is required.

Ion Chromatograph

The Dionex ICS 1600 Ion Chromatograph is in use for the analysis of anions by EPA 300.0. The instrument is to be maintained per manufacturer's recommendations and as required by the method. The instrument has a conductivity detector and autosampler, and is computer driven with software for sample analysis and processing. Calibration must be verified each working day or whenever a new batch of eluent is introduced, or instrument

performance check standards fall outside of acceptable QC ranges. Calibration and operation specifics are outlined in the SOP.

Graphite Furnace

The Graphite Furnace is a THGA configuration Perkin Elmer 900Z with Zeeman Background correction, coupled with an AS900 autosampler. Instrument is calibrated daily with four standards and a blank. The low standard is 0.001 mg/L which is equal to the MRL. Specific operating conditions and maintenance are outlined in the SOP.

Inductively Coupled Plasma (ICP)

The ICP is a Thermo Fisher 7400 ICP Dual View Instrument allowing for simultaneous analysis of multiple elements in either the axial or radial configuration. The detector is a CID camera coupled to computer software to calculate results. The instrument is calibrated daily with 6 standards and a blank. Operating parameters and standards are outlined in the SOP.

Turbidimeter

Calibrate daily with certified standards according to manufacturers recommendations. A certified QC standard between 0.1 and 1.0 is read each day of use and recorded in the turbidity log book.

Cuvettes should be rinsed with distilled water after use. Shake dry. Leave cap on to keep inside clean.

Test well should be kept covered with light shield to help keep clean. Use a soft cloth or tissue to clean inside well as needed.

Conductivity Meter

The HACH Titralab is used for conductivity and is calibrated each day of use with 0.01M KCl and the cell constant determined, readable in micromhos per centimeter. The probe is a graphite type cell and has a built in temperature compensation device. Sample temperatures are recorded with the conductivity result.

Never scratch the graphite bands with any hard substance or strike the probe against any hard surface. Clean thoroughly by stirring it in a mild detergent bath or isopropyl alcohol. Wipe with soft tissue or cloth and rinse thoroughly with deionized water. Recalibrate the meter after cleaning.

Drying Oven

The drying oven will be maintained at the temperature appropriate for the analysis being performed. The temperature will be monitored by a calibrated thermometer labeled with any correction factor if needed. The temperature will be recorded in the log book for each day in use. The oven should be cleaned immediately in the event of any spills.

Desiccators

There are three desiccators in use, two glass type and one stainless steel type. The desiccant used is Natrasorb and is a dark blue/purple color when dry (acceptable for use) and turns a light amber color when moist indicating that it must be regenerated. Regenerate in the drying oven overnight at 105 deg C. or until the pellets turn dark blue/purple in color.

Water Bath

There is one circulating water bath available for microbiology. It is to be maintained such that the water is clear and there is no build up of scale or deposits.

Clean periodically or whenever water is visibly dirty or there has been a spill. Use only distilled or deionized water in the baths.

Deionized/Reagent Water

Reagent water is produced using a pre-filter, reverse osmosis, mixed bed deionization unit producing Type I water with a resistivity of >10megohm, monitored continuously with an in-line conductivity/resistivity meter manufactured by the Myron L Company, and calibrated annually. Resistivity readings are recorded in the DI Water log book each day of use. HPC is monitored monthly and must be less than 1000cfu/ml. Heavy metals are checked annually, Cd, Cr, Cu, Ni, Pb, and Zn. No individual metal is to exceed 0.05 mg/L and total metals not to exceed 0.1 mg/L. Residual chlorine is tested monthly and recorded in the DI water log book. Annual biosuitability testing is not required on Type I water.

The deionized water system will require maintenance when the recorded daily resistivity readings fall below 10 megohm or the intermediate 1 megohm indicator light fails. Typically this will require replacement of the final DI cartridges and the 0.2um filter cartridge but may also require replacement of the RO cartridge if the system remains out of compliance.

Specific Ion Meter/Ammonia Electrode

Clean the instrument routinely and remove any spilled materials. If there are any operational problems error messages will be displayed. The ammonia electrode filling solution (0.1M NH₄Cl) should be changed weekly. A new membrane may be required

monthly or whenever stability is an issue or if visibly damaged. A new membrane must be conditioned by soaking overnight in 1000 mg/L ammonia standard.

For overnight or weeklong storage, place electrode in 0.1M filling solution or 1000ppm standard without ISA.

D.O. Meter/Probe

Clean the instrument routinely and remove any spilled materials. If there are any operational problems error messages will be displayed. The probe is laser based and requires no maintenance. It has a typical life of approximately one year and the instrument will display “replace sensor” when needed.

Refrigerators

Clean regularly and defrost as needed. Wipe up any spills immediately.

Hot Plate/Stirrers

Clean surfaces with mild detergent as needed. Clean up spills immediately.

Muffle Furnace

Clean as necessary and wipe up any spills. Replace or repair fire bricks if chipped or broken.

Micro-liter Pipettes

Calibrated quarterly to ensure accuracy to manufacturers specifications. Results recorded in log book.

QC Checks and Frequency

Instrument Performance Check

An instrument performance check sample is to be analyzed with each batch of samples. The results of this check standard are to be plotted on a control chart which shows the mean, and upper and lower control limits. If the analysis is out of control, do not proceed with sample analysis until the problem is resolved and a new check standard has passed all QC criteria as defined in the method.

Quality Control Sample

Analyze with each batch a standard prepared externally from those used for calibration. Results are to be plotted on control charts using limits specified in the methods or limits established internally over time. If this standard does not meet QC criteria, do not proceed with analysis until corrective action has occurred.

Reagent Blanks

Laboratory reagent blanks are to be analyzed at least once per batch or 5% of samples analyzed. This is to monitor reagent purity and overall procedural performance.

Matrix Spikes

Lab fortified matrix spikes will be performed on 10% of the samples. This is to verify absence of matrix interference, if any. They should be performed when the analyte of interest is absent more than 90% of the time. The known addition should be between 5 and 50 times the MDL.

Duplicate Analysis

Duplicate analysis will be performed on at least 10% of the samples. The two values will be entered into the database and percent relative difference calculated for use in the control charts for precision and accuracy.

Initial Demonstration of Capability

Initial demonstration of capability is to be performed according to the SOP's for the respective methods and will include analysis of second source Quality Control Samples (standards external to the calibration standards), MDL studies, and linear calibration range studies.

Method Detection Limits

Method detection limits are to be established yearly for each analyte in accordance with 40 CFR 136, using the procedure as outlined in EPA publication titled "Definition and Procedure for the Determination of the Method Detection Limit, Revision 2" published December 2016. Determine the MDL by adding the analyte(s) of interest to reagent water at or near a level 5 times greater than the expected MDL. Ion Chromatography requires 2-3 times the expected MDL and is to be performed every 6 months. Perform the analysis 7 times over a three day period and calculate the standard deviation. From a table of the one-sided t distribution select the value of t for $7-1=6$ degrees of freedom which equals 3.14 at the 99% confidence level. This product times the standard deviation is the method detection limit. The new procedure takes in to consideration spiked samples and method blanks for ongoing annual verification. Refer to the new EPA procedure for specifics.

Low Level Capability

The laboratories minimum reporting limit (MRL) should be reported to the client with the data and must be below the MCL. A lab fortified blank is analyzed each day at the MRL level and sample results should not be reported below the lowest calibration level.

Interlaboratory QC/Proficiency Testing Samples

Proficiency Testing Samples (a.k.a. Performance Evaluation Samples) will be analyzed once per year for each analyte in both the potable and non-potable categories. These samples are provided by an outside vendor meeting criteria established by NIST/NVLAP according to 40 CFR Parts 141 and 143 and 310 CMR 42.19(5). Results of these studies must be released directly to the certifying authority at the same time they are released to the laboratory. Continued certification of the laboratory will depend on successfully passing the proficiency testing.

In the event of a failed PT study used to maintain certification, the source of the problem must be identified, corrected and documented. The problem may be isolated by performing an audit trail on all analyses surrounding the failed PT analyte. This will include all reagent preparations, standardizations, calibrations and maintenance histories. The Massachusetts Laboratory Certification Office must be notified in writing, the action that has been taken. A follow-up PT study must be performed before the end of the year.

Failure to do so, or failing a second time, will result in a down-grade to “Not Certified” for that analyte.

On-site evaluations will be conducted periodically by the Massachusetts DEP Lab Certification Office to evaluate the QA/QC program and to work with the laboratory to solve any problems. These inspections may be on a scheduled basis or unannounced

QC Control Charts

Each analytical method will have control charts established to monitor method performance over time. These charts will plot accuracy and precision data and provide trend analysis. The results of sample duplicate analysis will be used to calculate percent relative difference and matrix spike recoveries will be calculated from spiked samples.

Percent relative difference is calculated using this equation:

$$\frac{(\text{sample result}-\text{duplicate result})}{(\text{sample result}+\text{duplicate result})/2} \times 100$$

Matrix spike recoveries are calculated using this equation:

$$\frac{(\text{Spiked sample result} - \text{unspiked sample result})}{(\text{Known spiked amount})} \times 100$$

Percent relative difference and matrix spike percent recovery will be plotted showing the mean value, and the upper and lower control limits, both of which will be ± 3 standard deviations as calculated by the Microsoft chart program. In situations where a matrix spike is not appropriate a Laboratory Control Standard is analyzed at the beginning of each batch and its value plotted instead of a matrix spike value (i.e. glucose/glutamic acid for BOD).

Trend Analysis/Corrective Action

Corrective action is the responsibility of the analyst under direct supervision of the Laboratory Supervisor. A corrective action sheet must be filled out listing the reason for the failure and the action taken. This sheet is to be signed by the analyst and the supervisor and entered in the corrective action log book.

If any quality control data is deemed “out of control”, this procedure should be followed:

If one measurement exceeds a control limit, repeat the analysis. If the repeat is within the control limit proceed with analysis. If it exceeds the limit discontinue analysis and correct the problem. Document the cause and corrective action.

If two of three successive points exceed a warning limit, analyze another sample and proceed with analysis if within warning limits. If outside warning limits, stop analysis and correct problem. Document the cause and corrective action.

If four out of five points in succession exceed 1 standard deviation or are in increasing or decreasing order, analyze another sample. If the next point is less than 1 std.dev. or changes the order continue analysis, otherwise do not proceed until the problem is corrected and documented.

If six successive samples are above the central line analyze another sample. If result is below central line proceed with analysis. If result is on same side of central line stop analysis until problem is corrected and documented.

QA Reporting Procedures

Quality assurance data is integrated into all log books and is also entered into the database for report generation and creation of control charts. Corrective action forms are also an integral part of QA reporting. It is the responsibility of the Laboratory Director/Supervisor to review all QA reporting procedures at least annually. A report should be prepared detailing any shortcomings of the program, overall laboratory performance, and recommend and implement any changes.

QA/QC data associated with sample analysis is supplied to the client when requested and is kept on file at the laboratory. QA/QC data is included on state, federal or regulatory forms when required or requested.

This manual is to be updated with any changes as they occur.

Data Reduction, Validation, Reporting and Maintenance

Significant Figures

All digits in a reported result are expected to be known definitely, except the last digit which may be in doubt. Report only digits that are justified by the accuracy of the work. Do not report more significant figures than are present in the detection limit.

Rounding Off

Round off by dropping numbers that are not significant. If the digit 6,7,8, or 9 is dropped, increase preceding number by one. If the digit 0,1,2,3 or 4 is dropped do not alter preceding digit. If the digit 5 is dropped round off preceding digit to nearest even number. Rounding off is only performed on the final calculation of a result.

Units

Units are listed on all reports with the corresponding analytes. Water samples are reported in mg/L (milligrams per liter) unless otherwise noted. Soil samples are reported in mg/Kg (milligrams per kilogram) unless otherwise noted.

Raw Data

Raw data is recorded in bound notebooks at the work station where the analysis is performed. Included in the log book will be calculated result and reported result. Data from the Ion Chromatograph is printed as well as kept on computer disk. Specific procedures for data reduction and calculations are contained in the SOP for each analyte.

Data Validation

Data is deemed to be valid after a careful review of all quality control parameters associated with a method of analysis finds that the data generated is under control and defensible. This includes calibration verification, matrix spikes, duplicate analysis, lab fortified blanks, reagent blanks, and any other requirements outlined in the methods. This complete evaluation must take place with each analytical run.

Final Data/Reports

Results are calculated from the raw data using standard equations and factors as defined in the method for that analyte. Final data is entered into the logbooks for that analyte, into the database and recorded on the original customer workorder. When all analyses are completed the final report is either printed directly out of the database or processed manually using Microsoft Word or Excel. Report formats should include analyte name,

result, date of analysis, detection limit, units, method of analysis, and analyst name. Compliance samples are reported on state supplied forms where applicable or as supplied by other agencies. An audit trail is performed on each report to check for accuracy, transcription, calculations, and completeness before being signed by the Laboratory Director.

Every effort is made to report data to all clients as soon as possible, typically 3-5 business days. Routine compliance monitoring data for public water systems must be reported to The Department no later than 10 days after the month in which it was received or no later than 10 days after the end of the reporting period whichever is sooner. This requirement applies to any samples that have been subcontracted to us or that we have subcontracted to other labs. The chain of custody for subcontracting has a check box that is used for compliance samples indicating that MCL rules apply and that we must be notified immediately of any EPA or Department established MCL or MRDL exceedance or indicate the presence of regulated microorganisms in potable water. Public water systems must be notified of these validated exceedances within 24 hours of sample analysis whether or not the laboratory accepting the sample subcontracted the analysis to another laboratory. The date, time and method of notification is recorded on the laboratory work order.

Finished water samples not reported on Department reports are generated on laboratory letterhead. Each analyte is listed with its result and corresponding MCL or MRDL. Any exceedances are flagged with a # sign next to the result. The laboratory certification number M-MA1118 is listed at the bottom of all reports. An additional page is provided with each report showing the certification status of each analyte for which it is certified, is not certified, or if no certification is offered, and whether or not the analyses are conducted according to department certification standards.

Data Corrections

In the event that a reported result is found to be incorrect, it will be the responsibility of the Laboratory Director to notify, in writing, the client and any regulatory agency that may have received the report that an error has occurred. The report may be corrected and resubmitted if it is deemed to be a typographical error, the analysis may be repeated if the sample is still within hold time, or a new sample may be required. In any case, an audit must be performed as to why the error occurred, corrective action taken and documented in the Corrective Action log book.

Record Maintenance

The database is available only to authorized personnel.

Raw Data-Created at the bench top and entered into permanently bound log books or generated by computer as with the Ion Chromatograph.

Chain of Custody records-Created at sample log-in and affixed to the laboratory workorder

Calculations-Performed in and recorded in the logbooks for each analyte.

Quality Control Data- Created on the bench top and entered in the logbook for each analyte.

Reports-Created upon completion of analysis, stored electronically or on paper.

All raw analytical data, chain of custodies, calculations used to generate final results, and quality control data, of any format (paper or electronic) are to be stored for ten years, 12 years for lead and copper results, five years for PT results. Raw data logbooks will be stored, and final reported data will be stored electronically in the database. Off-site storage of the database will also be done on a removable storage device. Appropriate computer software programs must be maintained to assure retrieval or regeneration of all data as new software revisions or programs are put into place.

Glassware

The laboratory utilizes Pyrex or Kimax brand Class A glassware, which includes volumetric pipettes, flasks and miscellaneous glassware.

Glassware cleaning requirements specified in methods must be followed. If there are no specifications the glassware is cleaned using Phosphate Free Liquinox detergent prepared according to manufacturers directions. The glassware is thoroughly cleaned and brushed if needed and first rinsed with tap water followed by three rinses with deionized water.

Separate glassware is maintained for phosphorus analysis and may require periodic cleaning with 1:1 Hydrochloric Acid if there is evidence of contamination.

Chemicals/Reagents

Chemicals and reagents must meet specifications required by the methods. If not specified, then “Analytical Reagent Grade” or “ACS” grade or better shall be used for all analysis.

All chemicals, reagents and prepared standards are logged in to the reagent/chemical database upon receipt or preparation. The following information is entered: chemical name, date received or prepared, expiration date, volume or weight, and final concentration.

Chemicals are stored per manufacturers recommendation, reagents are stored per method instructions. Any outdated standards are disposed of and new ones prepared.

Gases are chosen that meet the requirements of the methods and are maintained in a safe and secure manner.

Safety Plan

Achievement of a safe and healthful workplace is the responsibility of the organization, the laboratory director, supervisor, and all laboratory personnel. All employees must make every effort to protect themselves and others as well as visitors from any hazards associated with the laboratory.

The Laboratory Director will be responsible for implementing and enforcing the laboratory health and safety plan. This will include appropriate training, providing of protective equipment, inspections and maintenance of equipment. The Laboratory Director must inform personnel of hazardous materials or procedures with which they may be involved and the procedures required to protect themselves from those procedures. Material Safety Data Sheets (MSDSs) will be located in a notebook in the office area for reference. Training records shall be maintained in the safety record folder in the office.

Training and review will include at a minimum the following items:

First Aid; Protective clothing; Fume Hoods; Sinks; Eye Wash & Safety Shower; Chemical Storage; Labeling; Waste Disposal; Eye Protection; Fire Extinguishers; Fire Prevention; Fire Fighting; Emergency Exits; Hazardous Chemicals; Compressed Gases; Solvents; Glassware; Spill Cleanup.

1. Safety glasses are to be worn at all times in the laboratory areas. Glasses should be checked routinely for cracks or fogging and replaced as necessary.
2. Avoid unnecessary exposure to chemicals by any route and avoid working alone whenever possible. Safety gloves are available to prevent skin contact of chemicals or reagents.
3. Do not smell or taste chemicals.
4. Do not eat, smoke, or drink in laboratory areas. Wash hands before conducting these activities.
5. Do not store food or drink in laboratory refrigerators associated with analytical operations.
6. Do not use damaged glassware. Inspect glassware with each use and discard any that is chipped or broken.
7. Avoid practical jokes or other behavior which may distract another worker. No horseplay.
8. Do not mouth pipette, use a suction bulb.
9. Do not wear personal protective clothing in non-laboratory areas.
10. Personal clothing must include long pants, no shorts, shoes at all times, no sandals.
11. Keep work areas clean and uncluttered. Keep chemicals properly labeled and stored.
12. Use the fume hood whenever working with toxic or noxious vapors.

13. Spill clean up kits are located on both levels of the laboratory. These include materials for acid spills, caustic spills, and solvent spills. Notify supervisor of any spills and ensure that any used material is replaced.
14. Eye wash stations are on both levels and drench hose/eye wash on the lower level. The eyewash on the upper level is a squeeze bottle type. The top cap must be twisted off before use. The one on the lower level is a drench hose/eye wash and the protective covers will automatically come off when used.
15. Fire Extinguishers are maintained on each level, on the upper level near the sink station and the lower level at the end of the island bench top.
16. In the event of an incident where air contaminant levels cannot be maintained below action levels because of a chemical spill or reaction, evacuate the building. There are exits in the front and in the rear on each level. Call 911 for emergency assistance.
17. A first aid kit is mounted on the wall in microbiology and in chemistry and is stocked with Band-Aids, antiseptic ointments, and assorted supplies.

In case of fire or explosion: Immediately assess the situation and attempt to control a fire if comfortable using a fire extinguisher. If not possible, contain the fire by closing windows and doors and assure that all occupants of the building have evacuated using fire exits being at the front door and the rear door. The door to the warehouse is not an exit. Notify adjoining offices in the building of a fire. Call 911 from a safe location and meet at the company sign by the roadside in front of the building. Make certain that everyone is accounted for.

In the event of a burn, run affected area under cold water. Seek medical attention if more serious help is needed.

Hazardous Samples/Waste Disposal

Samples that are known or considered to be hazardous must be handled with proper safety equipment in place and they should be stored in a secluded area of the lab away from other samples until properly dispositioned.

Samples that have been acidified for preservation purposes are to be neutralized to pH 7 +/- 2 before disposal into the sanitary sewer system. These may be adjusted with sodium hydroxide or hydrochloric acid as necessary and verified with pH paper or pH meter.

Samples from analysis (titrations, colorimetric procedures) are collected in a 2 liter Teflon waste disposal container and neutralized to pH 7 +/- 2 before disposal into the sanitary sewer system. These may be adjusted with sodium hydroxide, sodium bicarb or hydrochloric acid as necessary and verified with pH paper or pH meter.

Any samples that cannot be effectively neutralized or may be deemed hazardous waste as defined in 310CMR part 30 must be stored in a secure area of the lab away from all other samples and disposed of off-site through a licensed waste disposal company.

Pollution Prevention

Every effort should be made to minimize the quantity or toxicity of waste at the point of generation. Numerous opportunities exist in the laboratory for reducing the amount of waste generated. The quantities of chemicals purchased should be consistent with the expected use over its designated shelf life. The volume of reagents prepared should be consistent with their expected use and stability.

Appendix i-Preservation-Hold Times-Bottles

| Parameter number/name | Container ¹ | Preservation ^{2,3} |
|---|----------------------------|--|
| e IA—Bacterial Tests: | | |
| 1–5. Coliform, total, fecal, and <i>E. coli</i> | PA, G | Cool, <10 °C, 0.0008% Na ₂ S ₂ O ₃ ⁵ . |
| 6. Fecal streptococci | PA, G | Cool, <10 °C, 0.0008% Na ₂ S ₂ O ₃ ⁵ . |
| 7. Enterococci | PA, G | Cool, <10 °C, 0.0008% Na ₂ S ₂ O ₃ ⁵ . |
| e IA—Protozoan Tests: | | |
| 8. <i>Cryptosporidium</i> | LDPE; field filtration ... | 0–8 °C |
| 9. <i>Giardia</i> | LDPE; field filtration ... | 0–8 °C |
| e IA—Aquatic Toxicity Tests: | | |
| 10–13. Toxicity, acute and chronic | P, FP, G | Cool, ≤6 °C ¹⁶ .. |
| e IB—Inorganic Tests: | | |
| 1. Acidity | P, FP, G | Cool, ≤6 °C ¹⁸ .. |
| 2. Alkalinity | P, FP, G | Cool, ≤6 °C ¹⁸ .. |
| 4. Ammonia | P, FP, G | Cool, ≤6 °C ¹⁸ , H ₂ SO ₄ to pH<2. |
| 9. Biochemical oxygen demand | P, FP, G | Cool, ≤6 °C ¹⁸ .. |
| 10. Boron | P, FP, or Quartz | HNO ₃ to pH<2 |
| 11. Bromide | P, FP, G | None required |
| 14. Biochemical oxygen demand, carbonaceous | P, FP, G | Cool, ≤6 °C ¹⁸ .. |
| 15. Chemical oxygen demand | P, FP, G | Cool, ≤6 °C ¹⁸ , H ₂ SO ₄ to pH<2. |
| 16. Chloride | P, FP, G | None required |
| 17. Chlorine, total residual | P, G | None required |
| 21. Color | P, FP, G | Cool, ≤6 °C ¹⁸ .. |
| 23–24. Cyanide, total or available (or CATC) | P, FP, G | Cool, ≤6 °C ¹⁸ , NaOH to pH>12 ⁶ , re- ducing agent ⁵ . |
| 25. Fluoride | P | None required |
| 27. Hardness | P, FP, G | HNO ₃ or H ₂ SO ₄ to pH<2. |
| 28. Hydrogen ion (pH) | P, FP, G | None required |
| 31, 43. Kjeldahl and organic N | P, FP, G | Cool, ≤6 °C ¹⁸ , H ₂ SO ₄ to pH<2. |
| le IB—Metals: ⁷ | | |
| 18. Chromium VI | P, FP, G | Cool, ≤6 °C ¹⁸ , pH = 9.3– 9.7 ²⁰ . |
| 35. Mercury (CVAA) | P, FP, G | HNO ₃ to pH<2 |

TABLE II.—REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES—Continued

| Parameter number/name | Container ¹ | Preservation ^{2,3} | Maximum holding time ⁴ |
|--|---|--|---|
| 35. Mercury (CVAFS) | FP, G; and FP-lined cap ¹⁷ . | 5 mL/L 12N HCl or 5 mL/L BrCl ¹⁷ . | 90 days ¹⁷ |
| 3, 5-8, 12, 13, 19, 20, 22, 26, 29, 30, 32-34, 36, 37, 45, 47, 51, 52, 58-60, 62, 63, 70-72, 74, 75. Metals, except boron, chromium VI, and mercury. | P, FP, G | HNO ₃ to pH<2, or at least 24 hours prior to analysis ¹⁹ . | 6 months |
| 38. Nitrate | P, FP, G | Cool, ≤6 °C ¹⁸ .. | 48 hours |
| 39. Nitrate-nitrite | P, FP, G | Cool, ≤6 °C ¹⁸ , H ₂ SO ₄ to pH<2. | 28 days |
| 40. Nitrite | P, FP, G | Cool, ≤6 °C ¹⁸ .. | 48 hours |
| 41. Oil and grease | G | Cool to ≤6 °C ¹⁸ , HCl or H ₂ SO ₄ to pH<2. | 28 days |
| 42. Organic Carbon | P, FP, G | Cool to ≤6 °C ¹⁸ , HCl, H ₂ SO ₄ , or H ₃ PO ₄ to pH<2. | 28 days |
| 44. Orthophosphate | P, FP, G | Cool, ≤6 °C ¹⁸ .. | Filter within 15 minutes; Analyze within 48 hours |
| 46. Oxygen, Dissolved Probe | G, Bottle and top | None required | Analyze within 15 minutes |
| 47. Winkler | G, Bottle and top | Fix on site and store in dark. | 8 hours |
| 48. Phenols | G | Cool, ≤6 °C ¹⁸ , H ₂ SO ₄ to pH<2. | 28 days |
| 49. Phosphorous (elemental) | G | Cool, ≤6 °C ¹⁸ .. | 48 hours |
| 50. Phosphorous, total | P, FP, G | Cool, ≤6 °C ¹⁸ , H ₂ SO ₄ to pH<2. | 28 days |
| 53. Residue, total | P, FP, G | Cool, ≤6 °C ¹⁸ .. | 7 days |
| 54. Residue, Filterable | P, FP, G | Cool, ≤6 °C ¹⁸ .. | 7 days |
| 55. Residue, Nonfilterable (TSS) | P, FP, G | Cool, ≤6 °C ¹⁸ .. | 7 days |
| 56. Residue, Settleable | P, FP, G | Cool, ≤6 °C ¹⁸ .. | 48 hours |
| 57. Residue, Volatile | P, FP, G | Cool, ≤6 °C ¹⁸ .. | 7 days |
| 61. Silica | P or Quartz | Cool, ≤6 °C ¹⁸ .. | 28 days |
| 64. Specific conductance | P, FP, G | Cool, ≤6 °C ¹⁸ .. | 28 days |
| 65. Sulfate | P, FP, G | Cool, ≤6 °C ¹⁸ .. | 28 days |
| 66. Sulfide | P, FP, G | Cool, ≤6 °C ¹⁸ , add zinc acetate plus sodium hydroxide to pH-9. | 7 days |
| 67. Sulfite | P, FP, G | None required | Analyze within 15 minutes |
| 68. Surfactants | P, FP, G | Cool, ≤6 °C ¹⁸ .. | 48 hours |
| 69. Temperature | P, FP, G | None required | Analyze |
| 73. Turbidity | P, FP, G | Cool, ≤6 °C ¹⁸ | 48 hours |
| Table IC—Organic Tests ⁹ | | | |
| 13, 18-20, 22, 24-28, 34-37, 39-43, 45-47, 56, 76, 104, 105, 108-111, 113. Purgeable Halocarbons. | G, FP-lined septum | Cool, ≤6 °C ¹⁸ , 0.008% Na ₂ S ₂ O ₃ ⁵ . | 14 days |
| 6, 57, 106. Purgeable aromatic hydrocarbons | G, FP-lined septum | Cool, ≤6 °C ¹⁸ , 0.008% Na ₂ S ₂ O ₃ ⁵ , HCl to pH 2 ⁹ . | 14 days ⁹ |
| 3, 4. Acrolein and acrylonitrile | G, FP-lined septum | Cool, ≤6 °C ¹⁸ , 0.008% Na ₂ S ₂ O ₃ ⁵ , pH to 4-5 ¹⁰ . | 14 days ¹⁰ |
| 23, 30, 44, 49, 53, 77, 80, 81, 98, 100, 112. Phenols ¹¹ | G, FP-lined cap | Cool, ≤6 °C ¹⁸ , 0.008% Na ₂ S ₂ O ₃ ⁵ . | 7 days until extraction, 40 days after extraction |
| 7, 38. Benzidines ^{11, 12} | G, FP-lined cap | Cool, ≤6 °C ¹⁸ , 0.008% Na ₂ S ₂ O ₃ ⁵ . | 7 days until extraction ¹³ |
| 14, 17, 48, 50-52. Phthalate esters ¹¹ | G, FP-lined cap | Cool, ≤6 °C ¹⁸ .. | 7 days until extraction, 40 days after extraction |
| 82-84. Nitrosamines ^{11, 14} | G, FP-lined cap | Cool, ≤6 °C ¹⁸ , store in dark, 0.008% Na ₂ S ₂ O ₃ ⁵ . | 7 days until extraction, 40 days after extraction |
| 88-94. PCBs ¹¹ | G, FP-lined cap | Cool, ≤6 °C ¹⁸ .. | 1 year until extraction, 1 year after extraction |
| 54, 55, 75, 79. Nitroaromatics and isophorone ¹¹ | G, FP-lined cap | Cool, ≤6 °C ¹⁸ , store in dark, 0.008% Na ₂ S ₂ O ₃ ⁵ . | 7 days until extraction, 40 days after extraction |

TABLE II.—REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES—Continued

| Parameter number/name | Container ¹ | Preservation ^{2,3} | Maximum holding time ⁴ |
|---|------------------------|--|---|
| 1, 2, 5, 8-12, 32, 33, 58, 59, 74, 78, 99, 101. Polynuclear aromatic hydrocarbons ¹¹ | G, FP-lined cap | Cool, ≤6 °C ¹⁸ , store in dark, 0.008% Na ₂ S ₂ O ₅ ⁵ | 7 days until extraction, 40 days after extraction |
| 15, 16, 21, 31, 87. Haloethers ¹¹ | G, FP-lined cap | Cool, ≤6 °C ¹⁸ , 0.008% Na ₂ S ₂ O ₅ ⁵ | 7 days until extraction, 40 days after extraction |
| 29, 35-37, 63-65, 107. Chlorinated hydrocarbons ¹¹ | G, FP-lined cap | Cool, ≤6 °C ¹⁸ | 7 days until extraction, 40 days after extraction |
| 60-62, 66-72, 85, 86, 95-97, 102, 103. CDDs/CDFs ¹¹ | G | Cool, ≤6 °C ¹⁸ , 0.008% Na ₂ S ₂ O ₅ ⁵ , pH<9 | 1 year |
| Aqueous Samples: Field and Lab Preservation | G | Cool, ≤6 °C ¹⁸ | 7 days |
| Solids and Mixed-Phase Samples: Field Preservation | G | Cool, ≤6 °C ¹⁸ | 24 hours |
| Tissue Samples: Field Preservation | G | Freeze, ≤-10 °C | 1 year |
| Solids, Mixed-Phase, and Tissue Samples: Lab Preservation | G | | |
| Table ID—Pesticides Tests: | | | |
| 1-70. Pesticides ¹¹ | G, FP-lined cap | Cool, ≤6 °C ¹⁸ , pH 5-9 ¹⁵ | 7 days until extraction, 40 days after extraction |
| Table IE—Radiological Tests: | | | |
| 1-5. Alpha, beta, and radium | P, FP, G | HNO ₃ to pH<2 | 6 months |

¹"P" is polyethylene; "FP" is fluoropolymer (polytetrafluoroethylene (PTFE, Teflon®), or other fluoropolymer, unless stated otherwise in this Table II; "G" is glass; "PA" is any plastic that is made of a sterilizable material (polypropylene or other autoclavable plastic); "LDPE" is low density polyethylene.

²Except where noted in this Table II and the method for the parameter, preserve each grab sample within 15 minutes of collection. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR Part 403, Appendix E), refrigerate the sample at ≤6 °C during collection unless specified otherwise in this Table II or in the method(s). For a composite sample to be split into separate aliquots for preservation and/or analysis, maintain the sample at ≤6 °C, unless specified otherwise in this Table II or in the method(s), until collection, splitting, and preservation is completed. Add the preservative to the sample container prior to sample collection when the preservative will not compromise the integrity of a grab sample, a composite sample, or an aliquot split from a composite sample; otherwise, preserve the grab sample, composite sample, or aliquot split from a composite sample with in 15 minutes of collection. If a composite measurement is required but a composite sample would compromise sample integrity, individual grab samples must be collected at prescribed time intervals (e.g., 4 samples over the course of a day, at 6-hour intervals). Grab samples must be analyzed separately and the concentrations averaged. Alternatively, grab samples may be collected in the field and composited in the laboratory if the compositing procedure produces results equivalent to results produced by arithmetic averaging of the results of analyses of individual grab samples. For examples of laboratory compositing procedures, see EPA Method 1664A (oil and grease) and the procedures at 40 CFR 141.34(f)(14)(iv) and (v) (volatile organics).

³When any sample is to be shipped by common carrier or sent via the U.S. Postal Service, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO₃) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H₂SO₄) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

⁴Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before the start of analysis and still be considered valid (e.g., samples analyzed for fecal coliforms may be held up to 6 hours prior to commencing analysis). Samples may be held for longer periods only if the permittee or monitoring laboratory has data on file to show that, for the specific types of samples under study, the analytes are stable for the longer time, and has received a variance from the Regional Administrator under §136.3(e). For a grab sample, the holding time begins at the time of collection. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR Part 403, Appendix E), the holding time begins at the time of the end of collection of the composite sample. For a set of grab samples composited in the field or laboratory, the holding time begins at the time of collection of the last grab sample in the set. Some samples may not be stable for the maximum time period given in the table. A permittee or monitoring laboratory is obligated to hold the sample for a shorter time if it knows that a shorter time is necessary to maintain sample stability. See §136.3(e) for details. The date and time of collection of an individual grab sample is the date and time at which the sample is collected. For a set of grab samples to be composited, and that are all collected on the same calendar date, the date of collection is the date on which the samples are collected. For a set of grab samples to be composited, and that are collected across two calendar dates, the date of collection is the dates of the two days; e.g., November 14-15. For a composite sample collected automatically on a given date, the date of collection is the date on which the sample is collected. For a composite sample collected automatically across two calendar dates, the date of collection is the dates of the two days; e.g., November 14-15.

⁵Add a reducing agent only if an oxidant (e.g., chlorine) is present. Reducing agents shown to be effective are sodium thiosulfate (Na₂S₂O₃), ascorbic acid, sodium arsenite (NaAsO₂), or sodium borohydride (NaBH₄). However, some of these agents have been shown to produce a positive or negative cyanide bias, depending on other substances in the sample and the analytical method used. Therefore, do not add an excess of reducing agent. Methods recommending ascorbic acid (e.g., EPA Method 335.4) specify adding ascorbic acid crystals, 0.1-0.6 g, until a drop of sample produces no color on potassium iodide (KI) starch paper, then adding 0.06 g (60 mg) for each liter of sample volume. If NaBH₄ or NaAsO₂ is used, 25 mg/L NaBH₄ or 100 mg/L NaAsO₂ will reduce more than 50 mg/L of chlorine (see method (Kelada-01) and/or Standard Method 4500-CN— for more information). After adding reducing agent, test the sample using KI paper, a test strip (e.g., for chlorine, SenSafe™ Total Chlorine Water Check 480010) moistened with acetate buffer solution (see Standard Method 4500-Cl.C.3e), or a chlorine/oxidant test method (e.g., EPA Method 330.4 or 330.5), to make sure all oxidant is removed. If oxidant remains, add more reducing agent. Whatever agent is used, it should be tested to assure that cyanide results are not affected adversely.

⁶Sample collection and preservation: Collect a volume of sample appropriate to the analytical method in a bottle of the material specified. If the sample can be analyzed within 48 hours and sulfide is not present, adjust the pH to >12 with sodium hydroxide solution (e.g., 5 % w/v), refrigerate as specified, and analyze within 48 hours. Otherwise, to extend the holding time to 14 days and mitigate interferences, treat the sample immediately using any or all of the following techniques, as necessary, followed by adjustment of the sample pH to >12 and refrigeration as specified. There may be interferences that are not mitigated by approved procedures. Any procedure for removal or suppression of an interference may be employed, provided the laboratory demonstrates that it more accurately measures cyanide. Particulate cyanide (e.g., ferric ferrocyanide) or a strong cyanide complex (e.g., cobalt cyanide) are more accurately measured if the laboratory holds the sample at room temperature and pH >12 for a minimum of 4 hours prior to analysis, and performs UV digestion or dissolution under alkaline (pH=12) conditions, if necessary.

(1) Sulfur: To remove elemental sulfur (S₈), filter the sample immediately. If the filtration time will exceed 15 minutes, use a larger filter or a method that requires a smaller sample volume (e.g., EPA Method 335.4 or Lachat Method 01). Adjust the pH of the filtrate to >12 with NaOH, refrigerate the filtrate to >12 with NaOH, refrigerate the filtrate and ship or transport to the laboratory. In the laboratory, extract the filter with 100 mL of 5% NaOH solution for a minimum of 2 hours. Filter the extract and discard the solids. Combine the 5% NaOH-extracted filtrate with the initial filtrate, lower the pH to approximately 12 with concentrated hydrochloric or sulfuric acid, and analyze the combined filtrate. Because the detection limit for cyanide will be increased by dilution by the filtrate from the solids, test the sample with and without the solids procedure if a low detection limit for cyanide is necessary. Do not use the solids procedure if a higher cyanide concentration is obtained without it. Alternatively, analyze the filtrates from the sample and the solids separately, add the amounts determined (in µg or mg), and divide by the original sample volume to obtain the cyanide concentration.

(2) Sulfide: If the sample contains sulfide as determined by lead acetate paper, or if sulfide is known or suspected to be present, immediately conduct one of the volatilization treatments or the precipitation treatment as follows: Volatilization—Headspace expelling: In a fume hood or well-ventilated area, transfer 0.75 liter of sample to a 4-L collapsible container (e.g., Cubtainer™). Acidify with concentrated hydrochloric acid to pH <2. Cap the container and shake vigorously for 30 seconds. Remove the cap and expel the headspace into the fume hood or open area by collapsing the container without expelling the sample. Refill the headspace by expanding the container. Repeat expelling a total of five headspace volumes. Adjust the pH to >12, refrigerate, and ship or transport to the laboratory. Scaling to a smaller or larger sample volume must maintain the air to sample volume ratio. A larger volume of air will result in too great a loss of cyanide (>10%). Dynamic stripping: In a fume hood or well-ventilated area, transfer 0.75 liter of sample to a container of the material specified and acidify with concentrated hydrochloric acid to pH <2. Using a calibrated air sampling pump or flowmeter, purge the acidified sample into the fume hood or open area through a fritted glass aerator at a flow rate of 2.25 L/min for 4 minutes. Adjust the pH to >12, refrigerate, and ship or transport to the laboratory. Scaling to a smaller or larger sample volume must maintain the air to sample volume ratio. A larger volume of air will result in too great a loss of cyanide (>10%). Precipitation: If the sample contains particulate matter that would be removed by filtration, filter the sample prior to treatment to assure that cyanide associated with the particulate matter is included in the measurement. Ship or transport the filter to the laboratory. In the laboratory, extract the filter with 100 mL of 5% NaOH solution for a minimum of 2 hours. Filter the extract and discard the solids. Combine the 5% NaOH-extracted filtrate with the initial filtrate, lower the pH to approximately 12 with concentrated hydrochloric or sulfuric acid, and analyze the combined filtrate. Because the detection limit for cyanide will be increased by dilution by the filtrate from the solids, test the sample with and without the solids procedure if a low detection limit for cyanide is necessary. Do not use the solids procedure if a higher cyanide concentration is obtained without it. Alternatively, analyze the filtrates from the sample and the solids separately, add the amounts determined (in µg or mg), and divide by the original sample volume to obtain the cyanide concentration. If a ligand-exchange method is used (e.g., ASTM D6888), it may be necessary to increase the ligand-exchange reagent to offset any excess of cadmium chloride.

(3) Sulfite, thiosulfate, or thiocyanate: If sulfite, thiosulfate, or thiocyanate is known or suspected to be present, use UV digestion with a glass coil (Method Kelada-01) or ligand exchange (Method OIA-1677) to preclude cyanide loss or positive interference.

- (4) Aldehyde: If formaldehyde, acetaldehyde, or another water-soluble aldehyde is known or suspected to be present, treat the sample with 20 mL of 3.5% ethylenediamine solution per liter of sample.
- (5) Carbonate: Carbonate interference is evidenced by noticeable effervescence upon acidification in the distillation flask, a reduction in the pH of the absorber solution, and incomplete cyanide spike recovery. When significant carbonate is present, adjust the pH to ≥ 12 using calcium hydroxide instead of sodium hydroxide. Allow the precipitate to settle and decant or filter the sample prior to analysis (also see Standard Method 4500-CN.6.3.d).
- (6) Chlorine, hypochlorite, or other oxidant: Treat a sample known or suspected to contain chlorine, hypochlorite, or other oxidant as directed in footnote 5.
- ⁷For dissolved metals, filter grab samples within 15 minutes of collection and before adding preservatives. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR Part 403, Appendix E), filter the sample within 15 minutes after completion of collection and before adding preservatives. If it is known or suspected that dissolved sample integrity will be compromised during collection of a composite sample collected automatically over time (e.g., by interchange of a metal between dissolved and suspended forms), collect and filter grab samples to be composited (footnote 2) in place of a composite sample collected automatically.
- ⁸Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.
- ⁹If the sample is not adjusted to pH 2, then the sample must be analyzed within seven days of sampling.
- ¹⁰The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.
- ¹¹When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity (i.e., use all necessary preservatives and hold for the shortest time listed). When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to ≤ 6 °C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (regarding the requirement for thiosulfate reduction), and footnotes 12, 13 (regarding the analysis of benzidine).
- ¹²If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 ± 0.2 to prevent rearrangement to benzidine.
- ¹³Extracts may be stored up to 30 days at -20 °C.
- ¹⁴For the analysis of diphenylnitrosamine, add 0.008% $\text{Na}_2\text{S}_2\text{O}_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.
- ¹⁵The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of atrin, add 0.008% $\text{Na}_2\text{S}_2\text{O}_3$.
- ¹⁶Sufficient ice should be placed with the samples in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present when the samples arrive, it is necessary to immediately measure the temperature of the samples and confirm that the preservation temperature maximum has not been exceeded. In the isolated cases where it can be documented that this holding temperature cannot be met, the permittee can be given the option of on-site testing or can request a variance. The request for a variance should include supportive data which show that the toxicity of the effluent samples is not reduced because of the increased holding temperature.
- ¹⁷Samples collected for the determination of trace level mercury (<100 ng/L) using EPA Method 1631 must be collected in tightly-capped fluoropolymer or glass bottles and preserved with trace level mercury should be filtered in the laboratory within 24 hours of the time of collection. However, if circumstances preclude overnight shipment, the sample should be filtered in a designated clean area in the field in accordance with procedures given in Method 1669. If sample integrity will not be maintained by shipment to and filtration in the laboratory, the sample must be filtered in a designated clean area in the field within the time period necessary to maintain sample integrity. A sample that has been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.
- ¹⁸Aqueous samples must be preserved at ≤ 6 °C, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of " ≤ 5 °C" is used in place of the " 4 °C" and " ≤ 4 °C" sample temperature requirements listed in some methods. It is not necessary to measure the sample temperature to three significant figures (1/100th of 1 degree); rather, three significant figures are specified so that rounding down to 6 °C may not be used to meet the ≤ 6 °C requirement. The preservation temperature does not apply to samples that are analyzed immediately (less than 15 minutes).
- ¹⁹An aqueous sample may be collected and shipped without acid preservation. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. If the sample must be analyzed within 24 hours of collection, add the acid immediately (see footnote 2). Soil and sediment samples do not need to be preserved with acid. The allowances in this footnote supersede the preservation and holding time requirements in the approved metals methods.
- ²⁰To achieve the 28-day holding time, use the ammonium sulfate buffer solution specified in EPA Method 218.6. The allowance in this footnote supersedes preservation and holding time requirements in the approved hexavalent chromium methods, unless this supersession would compromise the measurement, in which case requirements in the method must be followed.
- ²¹Holding time is calculated from time of sample collection to elution for samples shipped to the laboratory in bulk and calculated from the time of sample filtration to elution for samples filtered in the field.

Appendix ii-Instrument Maintenance Schedule

| <u>Instrument</u> | <u>Maintenance</u> | <u>Frequency</u> |
|--|---|--|
| Mettler Analytical Balance | Verified with 3 ASTM Class I wts. Cleaned and Calibrated by outside firm | Each day of use Annually |
| Mettler Top-loader Balance | Deflection test Cleaned and Calibrated by outside firm | Monthly Annually |
| Fluid Solutions Type 1 Water System filters as needed | Conductivity check Heterotrophic Plate Count Heavy Metals Chlorine | Daily, replace Monthly Annually Monthly |
| Thermometers | Verified against NIST thermometer | Annually or semi Annual for micro |
| pH Meters | Clean probe, check filling solution, inspect for damage | Each day of use |
| Conductivity Meter | Clean probe, check filling solution, inspect for damage | Each day of use |
| Dionex ICS1600 Ion Chromatograph | Replace pump seals Replace columns | Annual Poor response |
| HACH DR3900 Spectrophotometer | Clean Cuvettes Clean Photocell Window Lamp verification (880nm) when lamp is changed | Daily Weekly Annually, or |
| Orion Specific Ion Meter With Ammonia Electrode | Clean surface of meter Change filling solution Change membrane | As needed Weekly Monthly |
| Turbidimeter | Clean cuvettes and test well Calibrate Change bulb | As needed Daily As needed |
| Desiccators | Clean, replace or regenerate desiccant | As needed |
| HACH D.O. Meter & Probe | Clean surface of meter Change laser module | As needed Annually |
| Hot Plates/Magnetic Stirrers | Clean | As needed |
| Precision Water Baths | Clean | As needed |
| Sybron Thermolyne Muffle Furnace | Clean | As needed |
| Refrigerators | Clean; Defrost | As needed |

| | | |
|------------------------|---|---|
| Precision Drying Oven | Clean | As needed. |
| Perkin Elmer 900Z | Change Graphite Tube Check cooling water Check furnace windows Check autosampler capillary Check autosampler pump heads Change contact rings | As required Daily Daily Daily Daily As required or low response |
| Thermo Fisher 7400 ICP | Outlined in SOP EPA200.7 | |

Appendix iii - Water Sampling Instructions

Samples for microbiological analysis will be collected in disposable pre-sterilized plastic bottles.

Chemical analysis will be performed from a clean 500ml glass or plastic container.

When the sample is collected, sufficient air space (2.5cm) should be left in the top of the bottle to facilitate shaking prior to analysis. Keep sample bottles closed until they are to be filled and be careful not to contaminate inside surfaces of containers. Fill containers without rinsing and cap immediately.

1. Remove aeration screen or other attachments from faucet.
2. If tap cleanliness is in doubt it may be cleaned with either an alcohol prep pad or a weak solution of Clorox bleach.
3. Run hot water first (if available) for 2-3 minutes, followed by cold water for 2-3 minutes.
4. Reduce water flow to permit filling bottle without splashing.
5. Fill sample container(s) and recap immediately.
6. Deliver samples to the drop off location or to the laboratory as soon as possible.
Samples must be received at the laboratory within 24 hours of collection.

Do not take bacteria samples from a hose, they will almost certainly fail. Use a faucet that has been cleaned properly.

Special Instructions for Radon or Volatile Organic compounds:

If sampling for radon or volatile organic compounds special vials are required. They must be filled so that there are no air bubbles present after capping the vials. Invert the vial after filling and observe for any rising bubbles. If there are bubbles, just add some more water.

Label bottles with your name, address, date and time sampled.

Please fill in all information and include this sheet with the samples.

Name: _____
Address: _____
Town: _____
State: _____
Zip Code: _____
Phone: _____
Fax: _____
Email: _____
Date and time sampled: _____

Appendix iiiii – Safety Training Check List

Safety Training Check List

| | | | | | | | | |
|---------------------|--|--|--|--|--|--|--|--|
| Employee | | | | | | | | |
| Date | | | | | | | | |
| General | | | | | | | | |
| First Aid | | | | | | | | |
| Protective Clothing | | | | | | | | |
| Fume Hoods | | | | | | | | |
| Sinks | | | | | | | | |
| Drench Hose | | | | | | | | |
| Eye Wash | | | | | | | | |
| Chemical Storage | | | | | | | | |
| Labeling | | | | | | | | |
| Waste Disposal | | | | | | | | |
| Eye Protection | | | | | | | | |
| Fire Extinguisher | | | | | | | | |
| Fire Prevention | | | | | | | | |
| Fire Fighting | | | | | | | | |
| Emergency Exits | | | | | | | | |
| Hazardous Chemicals | | | | | | | | |
| Compressed Gases | | | | | | | | |
| Solvents | | | | | | | | |
| Glassware | | | | | | | | |
| Spill Cleanup | | | | | | | | |

